CONGRESS ON NANOTECHNOLOGIES:

ROUND TABLE
Innovation trends and mechanisms. Commercialization of BNM

THE THIRD INTERNATIONAL SYMPOSIUM
Bulk nanostructured materials: from fundamentals to innovations *BNM*[2011]

THE SECOND RUSSIN-FRENCH-GERMAN WORKSHOP
Atomic transport in BNM and related unique properties

BOOK OF ABSTRACTS

Ufa State Aviation Technical University
&
"NanoMeT" Ltd.,

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Preface

The processing of metals through the application of severe plastic deformation (SPD) is continuing to grow as a research topic in materials science laboratories around the world. Indeed, it is now recognized that the bulk nanostructured materials (BNM) produced by SPD offer many important characteristics and attractive features that are not readily achieved in their conventional coarse-grained counterparts.

This symposium, designated BNM-2011, follows on from the two earlier symposia in the same series. The first symposium, BNM-2007, was held in Ufa on 14-18 August, 2007, and this was followed two years later by BNM-2009 which was also held in Ufa on September 22-26, 2009. All symposia have been concerned with new developments in fundamental and applied research associated with the processing of bulk nanomaterials.

The innovation potential for bulk nanomaterials is now very large and it is becoming increasingly broad. Markets for bulk nanostructured materials exist in every product sector where superior mechanical and physical properties are critical for further development: these exceptional properties include high strength, good strength-to-weight ratio, excellent fatigue life and a potential superplastic forming capability as well as unique magnetic, electric and corrosion behavior. Nanometals are now under review and receiving active consideration for applications in a wide range of industrial sectors including in the broad fields of aerospace, transportation, medical devices, sports products, food and chemical processing, electronics and conventional defense.

The rapid recent developments in the production and reported properties of BNM make this topic especially important for timely discussions and innovative reports. It is hoped that much of the flavor of these new and exciting developments will be presented within the technical program of BNM-2011.

This book comprises the abstracts of reports to be presented, either in oral or poster sessions, at **the BNM-2011 symposium** and the **2nd ATBNM workshop** on August 22-26, 2011, in the Congress on Nanotechnologies in Ufa, Russian Federation. The abstracts are placed in order within appropriate sessions of the symposium and the workshop, and the poster presentations are designated by the code letter P. Following registration, the symposium will open formally on the morning of August 23 with introductory reports by the Co-Chairmen of BNM-2011, Prof. **Ruslan Z. Valiev** of Ufa State Aviation Technical University and Prof. **Terence G. Langdon** of the University of Southampton, U.K. This will be followed by two major keynote addresses: the first by Prof. **Horst Hahn** of the Karlsruhe Institute of Technology, Germany, on the use of nanomaterials in energy applications and the second by Prof. **E.V. Antipov** of Moscow State

University on developments in functional nanomaterials. The afternoon session on August 23 is designed as a Round-table covering the Commercialization of BNM. Incorporated into the Congress on Nanotechnlogies on the morning of August 24 is the Russian-French-German Workshop on “Atomic Transport in BNM and Related Unique Properties”. This workshop will continue also on the morning of August 25 and it will provide an excellent opportunity to hear about new developments in BNM in France and Germany. Special attention is called to the poster session which will be held from 17.30 to 19.00 on August 25. This poster session will be followed by the conference dinner where awards will be presented for the best posters. An important feature of the banquet will be the presentation of the Bulk Nanostructured Materials Honours Award which was first introduced at BNM-2009.

The Organizing committee of the BNM-2011 Symposium and the 2nd ATBNM workshop gratefully **acknowledge** the financial and organization support of the Government of Republic of Bashkortostan (www.tukaeva.ru), the Ministry of Industry and Innotative Policy of Republic of Bashkortostan (www.minpromrb.ru), the Academy of Science of Republic of Bashkortostan (www.anrb.ru), the Russian Foundation for Basic Research (www.rffi.ru), the Russian Academy of Sciences (www.ras.ru) and many others.

We hope you all enjoy your stay in Ufa. Welcome to BNM-2011!

The Organizing Committee

23 August

Plenary reports

August 23, 2011

Introductory report

Severe plastic deformation processing: new trends and approaches to grain refinement

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In the last decade, grain refinement by severe plastic deformation (SPD) has become a widely used processing route to produce bulk nanostructured materials (BNM), in particular metals and alloys. These tendencies of SPD processing development, due to appearance of new SPD techniques and SPD technologies for practical use, are considered in this talk. The adjustment of SPD processing regimes for fabrication of ultrafine-grained structures is another scientific and technical problem. To study it, the elaboration of basic rules for grain refinement including not only parameters of SPD processing (temperature, strain rate and degree, imposed pressure), but also nature and phase composition of a material before processing play an important role. The combination of phase transformations and microstructure refinement during SPD is presented as a new approach for effective nanostructuring of metals and alloys. Examples of its application for fabrication of ultrafine-grained structures in several steels, Al and Ti alloys and achievement of their unique properties are reported in this work.

August 23, 2011

Introductory report

Bulk nanostructured materials:

current status and future opportunities

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The development of a wide rang of bulk nanostructured materials (BNM) over the last decade has had a major impact on the field of materials science. These materials often offer significant advantages over their coarse-grained counterparts including high strength at ambient temperature and a superplastic forming capability at elevated temperatures. This presentation reviews the current status of BNM and examines the potential for improving the processing of these materials using .modified processing techniques.

Key-note report

Functional nanomaterials

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Nanomaterials play an important role in different modern technologies including energetics and metallurgy. The development of these technologies is mainly restricted by chemistry, namely, by physico-chemical properties of the materials used in respective devices or processes. Crystal chemistry approaches based on relationships between chemical composition, atomic structure and properties of compounds plays an important role in creation of the novel materials and improving properties of known ones. In this presentation our results in developing new electrode materials possesing high energy density for applications in Li-batteries – the most effective energy storage devices, high temperature Cu-based superconductors with the highest transition temperature observed up to now which can be used for energy transportation, and ceramic and alloys materials which can be used as low consumable anode materials without emission of CO_2 for aluminum production will be considered.

ROUND TABLE

Innovation trends and mechanisms.

Commercialization of BNM

Invited report

Markets for bulk nanostructured materials

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As the body of knowledge for bulk nanostructured metals has grown, so too have the potential markets for their application. The range of product forms that can be produced by an ever growing number of continuous severe plastic deformation methods is opening new commercial opportunities. In this report, we will review the current status of commercialization and the emerging trends in market development for bulk nanostructured metals. In addition, we will examine specific technological barriers that need to be addressed by the scientific community to improve access to some market areas.

August 23, 2011

Invited report

Biomedical Evaluation on Bulk Nanocrystalline Metals Fabricated by Severe Plastic Deformation

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Abstract: A variety of bulk nanocrystalline metallic biomaterials (including pure iron, 304 stainless steel, pure copper and NiTi alloys) were fabricated by the method of severe plastic deformation (SPD) technique, including high pressure torsion (HPT), equal channel angular pressing (ECAP) or severe rolling. The grain refinement was identified via the calculation from the XRD peak and morphological observation by TEM, with the grain size distribution ranging from dozens of nanometers up to about 200 nm. In vitro corrosion and biocompatibility involved cellular interaction, bioactivity and ion release detection and in vivo animal tests were carried out, as well as haemocompatibility evaluation, in order to explore the biological behavior of these bulk nanocrystalline metals.

In the case of bulk nanocrystalline pure iron[1], a potential candidate as biodegradable stent, a slower corrosion rate and higher tensile strength reduces the risk of early failure. The outcome of a promotion on endothelial cells (ECs) while an inhibition on vascular smooth muscle cells (VSMCs) favors the surface endothelialization and minimizes the thrombophilia under circulatory system.

For bulk nanocrystalline 304ss[2], the polarization resistance trials indicated that nanocrystalline 304ss is more corrosion resistant than the microcrystalline 304ss in oral-like environment with higher corrosion potential, and the amount of toxic ions released into solution after immersion is lower than that of the microcrystalline 304ss and the daily dietary intake level. The cytotoxicity results also elucidated that nanocrystalline 304ss is biologically compatible in vitro, even better than that of microcrystalline 304ss.

Ultra-fine grained (UFG) bulk pure copper has been successfully fabricated by equal-channel angular pressing (ECAP) [3], with the grain size about 380 nm after 8 passes. The potentiodynamic polarization results of the ECAP-copper specimens tested in Hanks solution revealed that the corrosion current of UFG copper is higher than that of the coarse grained copper. The cupric ion release behaviors of UFGcopper immersed inHanks solution for 30 days only displayed a burst release during the first 3 days (in comparison to the 1-2 months for the conventional Cu) from 115 μg/day to 12.5 μg /day, after which the ion release remained constant and slow. During the immersion experiments, Cu_2O was the only corrosion product found on the surface and it took 10 days or so to form a uniformCu_2O layer. Uniform corrosive damage on the surface and fewlocalized corrosion is observed. The above results indicate that UFG copper could have high potential as biomedical materials for contraception.

Bulk nanocrystalline and amorphous Ni50.2Ti49.8 alloy samples were successfully prepared from commercial microcrystalline Ni50.2Ti49.8 alloy discs by high pressure torsion (HPT) technique[4]. Then their corrosion resistance, surface wettability and cytotoxicity were further studied from the viewpoint of biomaterials. In both Hank's solution and artificial saliva, bulk nanocrystalline and amorphous Ni50.2Ti49.8 alloys showed significantly higher pitting corrosion potentials than that of microcrystalline Ni50.2Ti49.8 alloy. Meanwhile, the amount of Ni ion release after immersion in Hank's solution was minor, far below the threatening threshold of daily diet. Murine fibroblast and osteoblast cell lines were indirectly co-cultured with experimental sample extracts, indicating no cytotoxicity. Amongst all samples, the nanocrystalline Ni50.2Ti49.8 shows promising as best biomaterial candidate for its good combination of mechanical property, corrosion resistance and cytocompatibility.

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Invited report

Innovative trends in production of bulk nanostructured products by SPD

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Scaling up is one of the crucial problems of production of nanostructured products by SPD methods. Therefore in order to produce industrial scale products SPD methods are used in combination with tradition metal deformation process, for instance with rolling. Such the combined methods were found to be the most efficient ways for production of the largest scale products. Meanwhile it was shown recently that long size bars of titanium and low alloyed steel can be manufactured by rolling. In the present work recent progress in producing of long size bulk nanostructured products is reported. Some examples of producing products of various types (billets, bars, sheets, foils) of different materials are demonstrated.

Another key problem is producing bulk nanostructured products with homogeneous microstructure and extremely high mechanical properties. Influence of processing parameters on the formation of a homogeneous microstructure is discussed. Change in deformation path in combined processes improves preexisting structure. The effect of structure formed by combined processes on mechanical properties is considered. It is shown that combined SPD methods allow obtaining isotropic nanostructured materials with high strength and satisfactory ductility. Results of the extensive investigation of mechanical properties with respect to potentially attractive for innovative application ultrafine grained two-phase titanium Ti-6Al-4V alloy are submitted.

Invited report

Enhanced irradiation resistance of nanocrystalline alloys

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Nanocrystalline metals and alloys are attractive for advanced structural use under irradiation due to the expectation of their increased irradiation tolerance. In this work we have studied nanocrystalline intermetallic alloys which possess a long-range chemical ordering to examine irradiation effects on the stability or degradation of crystal superlattice in the ordered state. Nanocrystalline TiNi and FeAl alloys were studied as model systems by X-ray diffraction and Mössbauer spectroscopy methods. Bulk ordered nanocrystalline alloys with various grain sizes were processed using severe plastic deformation, namely high pressure torsion technique. Ordered nanocrystalline and coarse-grained samples were subjected to 1.5 MeV Ar ion irradiation at altered temperatures and irradiation rates with damage doses up to 5-6 displacements per atom. It was shown that at the equal damage dose nanocrystalline samples are able to retain a long-range ordering while the coarse-grained counterparts were substantially disordered or amorphised. Comparative analysis of long-range disordering and amorphisation kinetics revealed essentially enhanced irradiation resistance of nanocrystalline intermetallic alloys demonstrating their potential under irradiation conditions.

Oral report

High-strength ultra-fine grained titanium alloys for technical and medical applications

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In resent years bulk ultra-fine grained metal materials (grain size less than 1 μm) obtaining by severe plastic deformation are intensively developed and investigated. Based on the great number of researches it has been established that formation of such structure in metals and alloys leads to essential change in their mechanical and physical properties. Due to the fact, ultra-fine grained materials have high potential for possible practical application.

The results represented in the work received by authors in collaboration with colleagues concern the mechanical and physical properties of the ultra-fine grained titanium alloys produced by severe plastic deformation, as well as some possible aspects of its practical application. The ultra-fine grained structure in the alloys has been formed by methods of multiple forging and hot pressing with reversible hydrogenation.

On an example of Ti-6Al-4V and Ti-4Al-2V alloys it was established that formation of ultra-fine grained structure leads to essential increase in strength of the titanium alloys at room temperature and to displacement of a temperature interval of superplasticity to low temperature range. It was shown, that decrease in temperature of superplasticity of titanium alloys in an ultra-fine grained state can be used, in particular, for essential reduction of rolling temperatures of this material in comparison with traditional industrial technologies. Based on these results the half-finished products in the form of sticks from Ti-6Al-4V alloy with ultra-fine grained structure have been developed for medical implants production.

Studying of the influence of hydrogenation on strength and plastic characteristics of Ti-6Al-4V alloy revealed that formation of ultra-fine grained structure leads to increase more than twice of maximum permissible hydrogen concentration corresponding to transition of this material to brittle fracture. The enhanced resistance of ultra-fine grained state of titanium alloys to brittle fracture caused by hydrogen absorption can be used for production of responsible articles and constructions working in hydrogen containing media.

The formation of ultra-fine grained structure in Ti-4Al-2V alloy by severe plastic deformation leads to essential increase not only in strength but also in its acoustic properties that allows using this material for production of wave guides of ultrasonic systems (fig.1). At that the fracture of stepped wave guides made of ultra-fine grained Ti-4Al-2V alloy occurred at input power of ultrasound in 1.5-2 times higher than for the alloy in coarse-grained state. Moreover, the work life of such wave guides at multicyclic loading considerably increases under conditions of raised power of ultrasonic system (vibration amplitude more than 100 μm).

Thus the obtained results demonstrated the high potential of titanium based alloys with ultra-fine grained structure for possible technical and medical applications.

Fig. 1. Magnetostrictive transformer of ultrasonic system with waveguide thickeners from UFG Ti-4.5Al-2V alloy

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Oral report

Production of LIKO-M system medical items made of nanostructured titanium

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The production technology of LIKO and LIKO-M system medical items made of nanostructured titanium for surgical operations in the sphere of maxillofacial surgery and implantology has been developed and prepared for launching of mass production in the plant in Sarov. The items have been subjected to the full cycle of clinical and mechanical tests. The first operations with the new items have been carried out.

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Oral report

Biomedical application of nanostructured Ti

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The nanostructured titanium Grade 4 under consideration was produced with the help of ECAP-Conform technology in combination with drawing [1]. It has been demonstrated that the produced nanostructured titanium rods with a length of up to 3 meters have grain size of 200 nm and enhanced mechanical and fatigue properties (ultimate tensile strength is 1300 MPa, endurance limit on the basis of 10^7 cycles makes 620 MPa) [2]. It is very important for creation of new medical implants, in particular, it enables to improve dental implant construction – to reduce dimensions retaining service characteristics, and thus to use them for application in thinner bones [3]. In this work a special attention is focused on biomedical investigations of nanostructured titanium and a detailed consideration is given to advantages of nanotitanium application for dental implants. In particular, in case of application of nanotitanium the percentage of surface occupation by fibroblast cells increases, which is indicative of a higher biocompatibility of nanoTi. The growth of nanoTi osseointegration is also characterized by reduction of the period of active mineralization of bone tissue around the set implant [4]. As our investigations have demonstrated, nanostructured titanium possesses enhanced biocompatibility in comparison with coarse-grained titanium, which is probably predetermined by the condition of the oxide film and surface topography. Thus, the combination of enhanced mechanical and fatigue properties and increased biocompatibility makes titanium a more attractive material for fabrication of medical implants.

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Invited report

Multiscale effects of pressure in the formation of bulk nanostructured materials

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The report presents an analysis of research in physics, materials science and mechanics of large plastic deformation under pressure. The multiscale effects identified in this analysis are reflected in the mathematical model of plastic deformation and fracture of structurally-inhomogeneous material. Within this model the deformation and viscous fracture for compact and noncompact materials under pressure was studied. It is shown the adequacy of a developed model to experiment. From the developed theory follows the pressure at the deformation zone is a prerequisite for obtaining nanocrystalline structure by severe plastic deformation. These results became basis for development of technology of a large plastic deformation under pressure.

The results of twist extrusion studying for various materials (Al, Cu, Ti and its alloys, powders of different composition) that were obtained at the Donetsk Institute for Physics & Engineering (DonIPE) of the NAS of Ukraine are presented. By means of optical microscopy showed that the cross section of samples, as a rule, has a characteristic macrostructure with the structural elements, elongated in the direction of vortex centred on the axis of extrusion. In the longitudinal section of samples macrostructure resembles turbulent flow of liquid. The microstructure after multiple twist extrusion characterized by submicron dimensions and large-angle boundaries. Specified structure ensures the billets, treated by twist extrusion, a high strength and ductility. Despite the strain heterogeneity in one pass, multipass twist extrusion leads to a homogeneous structure and properties on the cross section of a sample. This is conditioned by mixing of a metal during twist extrusion, the structure stabilization and saturation of mechanical properties under simple shear.

The results of studies the angular hydroextrusion are presented. It is a severe plastic deformation process, as well as twist extrusion, proposed and developed in the DonIPE of the NAS of Ukraine. This process has certain advantages over the process of equal-channel angular pressing. They are mainly connected with the transition from boundary friction to a fluid friction in the container. The reduction of friction allows in times to increase the length of processed billets.

It is shown that the inclusion of severe plastic deformation methods to a technological chain of metal processing allows forming a structure with a unique combination of physical and mechanical properties. In particular:

- The process of twist extrusion was used for the processing of a commercially pure titanium with the aim of preparing a fine-grained structure. This structure will be inherited in subsequent technological operations. Its ensured the obtaining of a final product - titanium plates and wires with improved mechanical properties by the conventional methods of metal treatment.
- The entering of angular hydroextrusion process to the scheme of a copper CU-FRTP (analog M1) wire manufacturing with diameter of 0.5 mm (hot pressing, cold hydroextrusion and drawing) provided a record for copper and copper alloy combination of strength (σ=6n86 MPa) and electrical conductivity (87% IACS). Such strength of nearly 1.5 times higher the strength of M1 copper, subjected to a monotonic deformation.
- The samples of $Al_{86}Ni_6Co_2Gd_6$ alloy with nanocomposite structure, consisting of Al nanocrystals with the size of 12-14 nm were consolidated by twist extrusion. The obtained material is characterized by strength properties, substantially exceeding the properties of industrial high-strength aluminium alloys, and has a high ductility.
- Its shown the use of combined deformation (warm pressing with following twist extrusion) effectively in the manufacture of billets for the facing shaped charge.

Oral report

Development of nanostructured high-strength aluminum alloys by high-energy milling

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Due to the excellent stiffness-to-density ratio, the corrosion resistance and the susceptibility to different kinds of precipitation strengthening, aluminium alloys are the most important light-weight materials in aerospace and automotive industries. However, the low melting temperature makes aluminium alloys prone to creep damage at temperatures as low as 100°C. Above 200°C, grain growth and coarsening of strengthening phases causes a softening effect and a loss in creep resistance. By using ODS alloys (oxide dispersoid strengthened) the material's microstructure can be stabilized. High-energy milling of Al and oxide powder mixtures results in a mechanically alloyed material strengthened by fine-dispersed oxide particles. Specimens produced by hot isostatic pressing and/or extruding reveal superior mechanical properties, including an improvement in creep resistance and an increase in Young's modulus.

The present paper introduces the production route for two new resp. prototype materials: carbo-nanotube reinforced aluminium Zentallium® and a zink-ferrite-reinforced aluminium alloy, both produced by high-energy milling using a Simoloyer. First results reveal the potential of nanostructured Al alloys for different kinds of applications also at elevated temperatures. It is believed that the fine-dispersed ceramic particles stabilize the microstructure, i.e., they do not grow and they hinder grain coarsening.

Oral report

Nanostructured titanium alloys for innovative applications in mechanical engineering

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High specific strength and enhanced fatigue properties of nanostructured titanium materials, achievable by means of severe plastic deformation (SPD) techniques, have allowed developing new technologies for semiproducts and items made of them in such top industrial sectors as transport and electric-power industry, in particular fabrication of power units for aircrafts and energy transfers. First of all, it is the way for enhancement of their operation reliability, service life, power, coefficient of efficiency, production effectiveness, and other performance properties, which predetermine the commercial demand. High-load parts of electric power plants, gas compressor stations, aircrafts, such as compressor blades and fastener units for threaded joints, are the examples of these items. And emergency failures of such units are often caused by fatigue and erosion damages.

In this regard the subject of this paper was investigation of potential capability of fatigue properties increase for gas turbine engine blades made of the VT6 alloy by means of formation of ultrafine-grained (UFG) structure in the alloy with the help of SPD and subsequent isothermal forging (ISF) of billets at lowered temperatures.

This work represents the research results of mechanical properties and microstructure of VT6 alloy blades, fabricated by isothermal forging of a UFG billet. The dependence of evolution peculiarities of the initial UFG structure on ISF processing conditions in the temperature range of 650…850^{o}C are demonstrated. It has been established that formation of a homogeneous microstructure with the dimensions of grains/subgrains of less than 1 μm with the help of forging allows increasing the endurance limit of blades at room temperature by no less than 30% in comparison with a blade, fabricated by conventional ISF technique.

Oral report

On efficiency of continuous SPD processes for producing of UFG materials

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The report presents the results of studies on continuous SPD processes, such as ECAP-Conform and shear drawing. A number of factors influencing the efficiency of the UFG structure formation, including a scale factor, temperature, specific surface area, are studied. It has been demonstrated that one of the important factors is a specific surface area of the deformation zone and its increment, which enable considering the scale factor during processing and predicting the properties of the processed semi-products and items. Practical use of the results of conducted studies is demonstrated on the example of establishment of a production line for high-strength nanostructured semi-products from CP Ti for medical application.

Oral report

Friction and wear mechanisms of coarse-grained and ultrafine-grained titanium

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High corrosion resistance, biocompatibility and low density have provided application of unalloyed titanium in the chemical industry and in medicine as implants. But low strength properties and low wear resistance at a friction restricts its use as constructional and functional materials. Ultrafine-grained (UFG) structures formation by methods of intensive plastic strain (SPD) has allowed to raise yield stress and ultimate tensile strength of titanium. However there are few examinations tribological behavior and wear mechanisms of the UFG titanium [1, 2].

The present work is devoted comparative examinations of lows and wear mechanisms of the commercial pure titanium VT1-0 with coarse-grained (CG) and ultrafine-grained structure in the conditions of a friction with boundary lubrication. The UFG structure with the different size of grains and a different degree of a non-equilibrium structure has been produced by a method of *abc*-pressing with the subsequent rolling at the room and raised temperatures.

In present work the microstructure of the explored materials is in details certified by methods of the optical microscopy, scanning and transmission electron microscopy. Mechanical properties were examined using tension tests. The method of a quantitative assessment of a degree of a non-equilibrium of UFG materials structure with use of a method of electrons back-scattering diffraction (EBSD) is offered.

For the coarse-grained titanium on the dependence of wear rate vs sliding distance we observed stages with high and very low wear rate, which is caused by periodic formation and fracture on a surface of samples of secondary structures in the form of islets with high wear resistance.

Features and wear mechanisms of the titanium with the ultrafine-grained structure produced under various technological plans are explored. It is shown, that for the titanium with UFG structure a major factor spotting wear resistance, the degree of a non-equilibrium of a microstructure is: the above a non-equilibrium, the below its wear resistance. The average size of grains and strength properties show considerably smaller influence on wear resistance of the titanium.

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24 August

THE SECOND RUSSIN-FRENCH-GERMAN WORKSHOP

Atomic transport in BNM and related unique properties

August 24, 2011

Invited report

Grain boundary allotropy in severely deformed metals and alloys

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Ever since it has been demonstrated that ultrafine grained materials processed by severe plastic deformation (SPD) possess unique properties and property combinations, such as drastically increased strength and high elongation to failure, the physical reasons behind these unusual and highly beneficial property combinations have been discussed controversially. Early on in this ongoing discussion, dislocation-grain boundary interactions have been suggested by A.A. Nazarov and R.Z. Valiev et al. in a series of papers to modify existing or newly formed random high angle grain boundaries into so-called “non-equilibrium” grain boundaries with an increased excess free energy density. These non-equilibrium grain boundaries are expected to exhibit also an increased specific diffusivity as well as an increased microstrain in the boundary-near regions.

The present contribution summarizes recent experimental results based on microstructure analyses (SEM-EBSD, TEM including C_s-corrected HRTEM and local strain analyses by Geometric Phase Analysis) together with detailed grain boundary diffusion analyses on different pure metals and binary alloys using the radiotracer method. Basic issues concerning the existence and evolution of such “non-equilibrium” grain boundaries, their property characteristics and their relation with the performance of SPD-processed materials are addressed.

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Invited report

Effective temperature of severe plastic deformation

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Severe plastic deformation (SPD) can drive the unusual phase transformations in metallic alloys. The theory of driven systems proposed by G. Martin predicts that the external influence can additionally force the diffusion-like atom movements. As a result, the driven systems can be described in terms of the effective temperature T_{eff} which can differ from the treatment temperature. We observed that – together with grain refinement – SPD can lead to the formation of the phases which are stable at the temperature and pressure of SPD treatment. Thus, we observed the extremely quick decomposition of the supersaturated Zn-rich Al alloys. After SPD only the phases which are stable at room temperature (almost pure Al and Zn) remained in the system. After SPD of Fe–C alloys also the metastable phases like retained austenite and Hägg carbide disappeared and only ferrite and cementite (stable above 0.3 GPa) remained. In this case, and also in Co–Cu alloys the effective temperature was around 300°C. In case of Cu–Ni alloys the effective temperature was measured to be around 300°C. SPD leads to the transition from five different binary phases before high pressure torsion to two another crystalline phases and two different amorphous phases in the Ni–Nb–Y alloys. It corresponds to T_{eff} = 1450°C. The analysis of published papers on amorphysation of TiNi and NdFeB permitted to estimate respective T_{eff} = 950-1250°C and T_{eff} = 700°C. The data on nanocrystallisation of amorphous alloys (like) also permited to estimate T_{eff}. The observed dependence of T_{eff} on the bulk diffusion coefficient (extrapolated to the SPD treatment temperature) follows the law predicted by G. Martin.

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August 24, 2011

Invited report

Cu/Nb nanocomposite wires processed by severe plastic deformation: effects of the nanostructure on the resistance to extreme environment (high strain, high stress, high temperature)

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Copper-based high strength and high electrical conductivity nanocomposite wires reinforced by Nb nanotubes are prepared by severe plastic deformation, applied with an Accumulative Drawing and Bundling process (ADB), for the windings of high pulsed magnets. The ADB process leads to a multi-scale Cu matrix containing up to $N=85^4$ ($52.2\ 10^6$) continuous parallel Nb tubes with diameter down to few tens nanometers. After heavy strain, the Nb nanotubes exhibit a homogeneous microstructure with grain size below 100 nm. The Cu matrix presents a multi-scale microstructure with multi-modal grain size distribution from the micrometer to the nanometer range. The use of complementary characterization techniques at the microscopic and macroscopic level (in-situ tensile tests in the TEM, nanoindentation, in-situ tensile tests under high energy synchrotron beam) shed light on the role of the multi-scale nature of the microstructure in the recorded extreme mechanical properties [Acta Mat, 57 (2009), p3157].

The thermal stability of these nanocomposite metals has also been investigated via time-resolved *in situ* annealing under synchrotron high-energy x-rays. The diffraction peak profile analysis demonstrates that internal-stress relaxation begins in the Nb nanotubes at a temperature far below the bulk recrystallization temperature and follows size-specific regimes originating from a proximity effect with the nanostructured Cu matrix: the increased Cu-Nb interface surface disrupts internal-stress relaxation processes, leading to larger thermal resistance [Acta Mat, 58 (2010), p6504].

Oral report

Theoretical strength of nanomaterials and elastic strain engineering

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Elastic strain of crystal lattice results in change of interatomic distances and, as a consequence, in change of its physical and machanical properties. This fundamental physical effect is employed by rapidly developing elastic strain engineering (ESE) [1,2]. Typicaly physical and mechanical properties are proportional to elastic strain. This means that if elastic strain of definite sign gives a negative effect on a considered property, then strain of opposite sign with definitely improve this property. The problem is that conventional materials can sustain rather small external stress (apart form the hydrostatic pressure) at which the elastic component of strain is too small to noticeably affect a desirable property. On the other hand, nano-size objects can demonstrae very high strength approaching the theoretical limit, which was estimated by Frenkel as $\sigma_{theor}=G/2\pi$, where G is the shear modulus. For nanomaterials loaded closely to the theoretical strength limit, one can achieve elastic strain of order of 1% or, in some cases, e.g., for various nanowires, for graphene and carbon nanotubes, of order of 10%. Thus, study of theoretical strength limit is an important problem in frame of ESE.

We report on the results of computer simulation of theoretical strength of intermetallic nanowires and graphene. Mechanisms of defect nucleation at ultra-high external stress are investigated. Effect of large elastic strain on elastic properties, density of phonon states and properties of gap discrete breathers is investigated.

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Oral report

Analysis of the deformation process of NiTi discs using different cross sections of high-pressure torsion discs

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NiTi alloys are of great scientific and technical interest due to their remarkable properties such as the shape memory effect. Nanostructured NiTi attracts particular attention due to enhanced thermomechanical properties in combination with high mechanical strength. Nanostructured NiTi alloys can be processed by methods of severe plastic deformation (SPD) and subsequent annealing. High-pressure torsion (HPT) leads to grain fragmentation and a crystalline to amorphous phase transformation [1]. This is analysed in detail using different cross sections of the HPT discs.

Discs with 8 mm in diameter and a thickness of 0.7 mm were deformed by HPT applying 12 turns at a pressure of 4 GPa. Samples were analyzed using transmission electron microscopy (TEM), X-ray diffraction (XRD) and calorimetric methods. TEM foils were taken at a distance of about 2.8 mm from the centre of the HPT disc (corresponding to a deformation degree $\gamma \sim 400$). Three different TEM sections were prepared. In addition to the planar section (PS) frequently used in previous studies, a radial section (RS) and a transversal section (TS) were analyzed (cf. Figure 1). TEM samples were prepared carefully using spark erosion, grinding and ion milling.

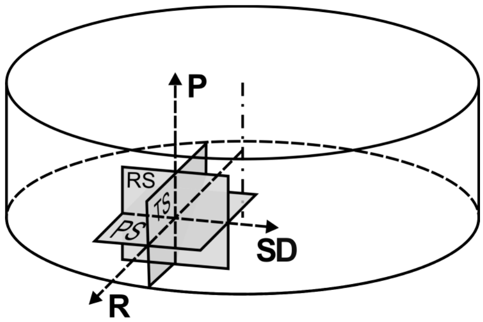

Fig. 1. Sketch of TEM sections: the radial section (RS), tangential section (TS) and planar section (PS) with a foil normal along the radius (R), shear direction (SD) and a parallel line to the rotational axis (P), respectively.

TEM and XRD of HPT deformed NiTi shows a mixture of fragmented grains, nanocrystals and an amorphous phase. In the case of the section PS, TEM samples show a complicated contrast including moiré artefacts due to the overlap of the crystals of different orientations. In the case of RS and TS samples, amorphous bands show sharp and straight interfaces with respect to the nanocrystalline regions. cf. Figure 2).

Diffraction patterns of specimens PS, RS and TS indicate a deformation texture with the texture components P||$\langle 111\rangle$, R||$\langle 1\bar{1}0\rangle$ and SD||$\langle 11\bar{2}\rangle$ (B2 structure is taken as reference). Additional, high-resolution TEM was applied to nanocrystals in RS, showing flat crystalline-amorphous interfaces parallel to SD and fuzzy crystalline-amorphous interfaces inclined to SD. Isochronal and isothermal calorimetry show an exothermic heat flow arising by relaxation and crystallization of the amorphous phase.

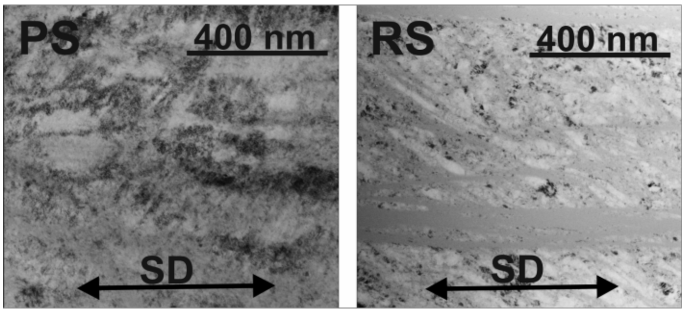

Fig. 2. TEM dark-field images of PS and RS samples of HPT NiTi (γ~400). Complicated contrast arising from overlapping grains occurs in the TEM projection of PS samples, whereas RS samples show frequently elongated grains with comparable clear contrast. The shear deformation SD can be analyzed directly

Based on the TEM analysis it is concluded that strong grain fragmentation occurs concomitant with amorphization starting at the martensitic twin interfaces and leading to the formation of thin amorphous bands inclined to SD. Locally, amorphization also occurs by the formation of shear bands. Frequently nanocrystalline debris is observed; caused by the shear deformation, the nanocrystals get elongated, rotated towards SD, and finally fragmented as they break in parts as deformation proceeds.

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Oral report

Grain boundary diffusion in ultra fine grained copper processed by high pressure torsion straining

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Ultrafine grained (UFG) materials produced by severe plastic deformation exhibit unique properties due to their large defect densities associated with high specific excess energies [1]. According to the existing models of grain refinement by severe plastic deformation (e.g. High Pressure Torsion (HPT) [2]), the abundance of lattice dislocations created during the severe straining serves to modify the structure of high angle grain boundaries such that so-called "non-equilibrium" grain boundaries (GBs) with enhanced excess free energy densities are created. To study the unique properties of UFG materials, and specifically to analyze the grain boundaries created by the severe deformation processing, several copper samples were prepared by HPT. Short-circuit diffusion in these UFG copper samples was investigated by the radiotracer method. In addition to conventional GB diffusion rates, which characterize the relaxed high-angle GBs as present in annealed coarse-grained samples, "ultra fast" diffusivities are observed. The measurements were performed in the so-called C-B and C-C kinetics regime after Divinski's classification [3], which represents a modification of Harrison's classical stadium C kinetics. The penetration profiles and deduced diffusivities (in particular the „ultra fast" diffusivities D_{uf}) are affected by recrystallization/grain growth which accompany the diffusion annealing treatments and results in non-Arrhenius behaviour of the derived data (Fig. 1).

The non-equilibrium GBs contribute largely to the "ultra fast" diffusion paths. The kinetics and structural properties of non-equilibrium GBs in HPT Cu are thoroughly investigated as a function of material purity and hydrostatic pressure applied.

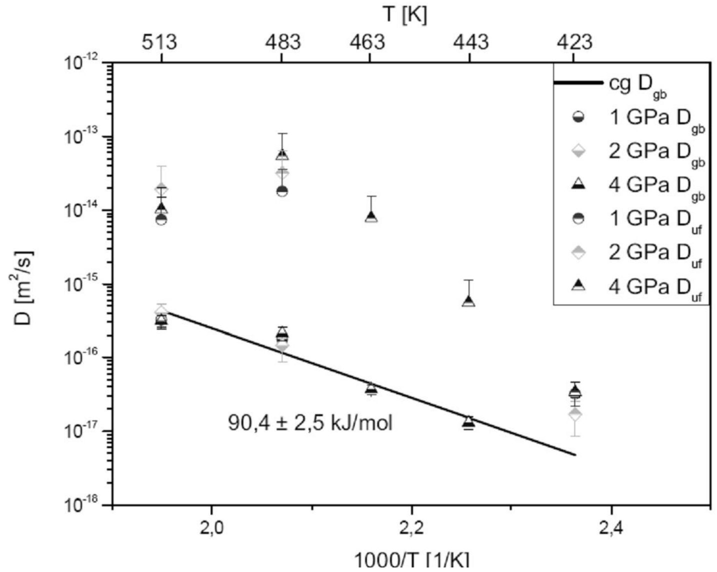

Fig. 1: Arrhenius diagram for Ni diffusion in HPT 4N Cu as a function of the applied hydrostatic pressure. For comparison grain boundary diffusion data of Ni in annealed coarse grained Cu is plotted (solid line). D_{gb} and D_{uf} indicate conventional GB diffusion and "ultra fast" diffusion in HPT Cu.

Furthermore, a basically new defect – a network of percolating porosity – is observed in the HPT strained samples, supposedly introduced during the HPT process or during release of the hydrostatic pressure. The kinetic properties of the percolating porosity pathways in HPT Cu are similar to those observed previously in ECAP (Equal Channel Angular Pressing) Cu [4]. A strong influence of the applied hydrostatic pressure on the effective diffusivity and the volume fraction of the generated percolated porosity is observed.

The support of this work by the Deutsche Forschungsgemeinschaft is gratefully acknowledged.

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Oral report

Self-diffusion in ultrafine-grained Ag and Ti produced by equal channel angular pressing

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Severely deformed materials attain growing technological interest in the last years due to their extraordinary properties and often unique property combinations. In the literature so called "non-equilibrium grain boundaries" are invoked to be responsible for these properties. These specific grain boundaries contain presumably a higher energy, higher free volume and therefore reveal a higher diffusivity. In the present study the self-diffusion in ultrafine-grained Ag and Ti produced by equal channel angular pressing (ECAP) is investigated. The radiotracer method (using ^{110m}Ag and ^{44}Ti isotopes) in combination with parallel sectioning performed by high prescion grinding or microtome was used to measure the diffusion properties of the grain boundaries. The obtained grain boundary diffusion coefficients of ultrafine-grained materials were compared with the grain boundary diffusion coefficients measured in coarse grained counterparts of the same purity.

The Ag samples were deformed by ECAP with a hydrostatic pressure of 2 GPa and 1,2 or 3 passes through a 90°C die (the route A). The resulting microstructure was examined by transmissions electron microscopy (TEM). The grain boundary self-diffusion was studied between 293 K and 373 K depending on the deformation state (1, 2 and 3 passes). Applying the Borisov formalism [1] the grain boundary energies of coarse grained and ultrafine-grained materials were calculated. The energy of non-equilibrium grain boundaries is about 10% to 15% higher than that of relaxed high-angel grain boundaries in the coarse-grained material.

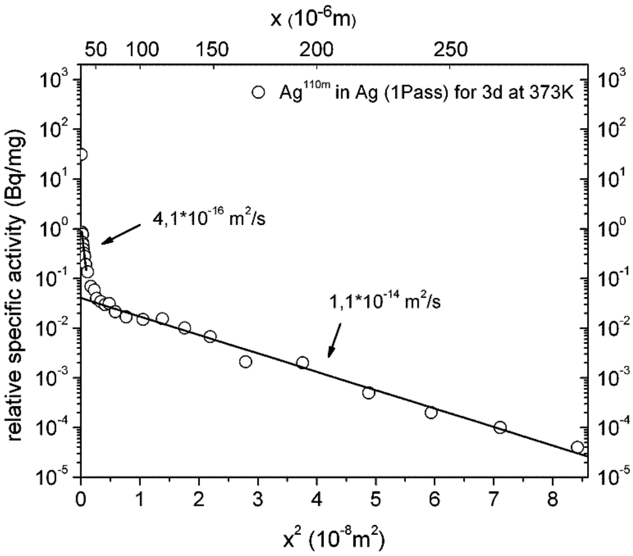

Fig. 1. Diffusion profile of Ag^{110m} in ECAP-Ag. The blue line belongs to normal grain boundaries and the faster part (red line) belongs to non-equilibrium grain boundaries.

Similar, Ti samples were deformed by the ECAP-Conform method and the resulting microstructure was studied by TEM. Self-diffusion and solute diffusion of substitutionally migrating Ag was measured. Additionally the hardness depending on the pre-annealing temperature up to 973 K was measured. A correlation between the structure, hardness and diffusion rates is established.

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Invited report

Structure-phase states and properties of an austenitic stainless steel after severe plastic deformation

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The purpose of this work is to study the regularities of nano - and submicrocrystalline structure formation in the austenitic 0.07%C-17.3%Cr-9.2%Ni-0.7%Ti steel during severe plastic deformation (SPD) via high pressure torsion (HPT) and equal channel angular pressing (ECAP), as well as to enhance the properties of this steel via grain refinement.

Microstructure evolution during HPT at P=6 GPa at room temperature was revealed in this steel. It was shown that a mean grain size at true strain of ~ 6 was 62 nm. The XRD analysis revealed that martensite $\gamma \rightarrow \alpha$ and $\gamma \rightarrow \varepsilon \rightarrow \alpha$ transformations during HPT at room temperature took place. After processing ~ 60 % α - martensite, ~35 % austenite and ~ 5 % ε-martensite were revealed, indicating the appearance of dual-phase austenitic-martensitic state in the steel.

During heating of this nanocrystalline steel after HPT the size of grains doesn't change till the temperature of 400°C, however at 500°C it grows up to 250 nm, and intensive growth begins with 600°C. This leads to changing of a volume fraction of phase components at heating: above 550°C the structure becomes mainly the austenitic one what effects corrosion properties.

Studies of mechanical properties have shown that HPT considerably increases strength characteristics of the steel: YS raises almost in 6 times, and UTS - in 3 times. The YS is maximal after HPT at T=300°C and equals to 1740 MPa. The minimal value of YS=1640 MPa at δ =10 % is fixed at deformation temperature of 500°C.

Influence of a neutron irradiation on behavior of the nanocrystalline steel after HPT was investigated. It is shown that up to fluencies $2x10^{20}$ n/cm^2 strength of a material practically doesn't change and doesn't have irradiation-induced defects. At the same time propensity of a material to corrosion in the chlorine-containing environment has essentially grown at 8-14 times at fluencies of neutrons $2x10^{20}$ n/cm^2.

After ECAP at room temperature with ε = 3.2 the submicrocrystalline structure with grain/subgrain size of 100-250 nm was formed. In this state the quantity of martensite at ε = 3.2 reaches 45%. At heating martensite (ferrite) turns to an austenite and the most intensive transformation goes in the range of temperatures 500 … 600°C. After ECAP the processed steel in austenitic-martensitic state exhibits UTS = 1140 MPa in comparison with the initial state, where UTS = 250 MPa. The enhanced level of fatigue strength has been also revealed after processing.

Thus, it is shown that for obtaining both nano- and submicrocrystalline structures and increased strength and fatigue properties of the 0.07%C-17.3%Cr-9.2%Ni-0.7%Ti steel in mainly austenitic state, it is necessary to perform SPD in the range of temperatures 300-500^{o}C, or at the lowered temperatures with the subsequent heating on 500-550^{o}C.

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Invited report

Atomistic investigation of the grain boundary segregations

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The observations of the abnormal segregations of alloying components in nanocrysalline materials after severe plastic deformation renovated interest to problem of the interactions between solute atoms and grain boundaries (GB). Despite on remarkable efforts our understanding of GB segregation is still based on theory of elasticity and equilibrium thermodynamics arguments. Here we report the results of investigation of GB-solute interactions in framework of the atomistic approach included *ab-initio* calculations and molecular dynamic simulation of the solute atoms in vicinity of GB.

Using the electronic structure total energy calculations we found that strong interaction between solute atoms and special GB take place within few closest to GB atomics layers and variation of the local charge density is dominate in interaction energy (chemical contribution). At the same time for GB with more closed packing of atoms a local distortion give the major contribution to the solute-GB interactions. Using molecular dynamic simulation we investigated the segregation of the solute atoms on different type of GB structure (special GB and non-equilibrium GB).

We propose a generalization of classical Langmuir-MacLean theory of the GB segregation by taking into account the changes of thermodynamic parameters nearby in vicinity of GB. The obtained results allow revealing the condition which determined the segregation capacity of GB. We show that observed strong segregations in nanocrysalline materials connected with the specific state of non-equilibrium GB formed owing to severe plastic deformation.

Oral report

Mesoscopic grain boundary sliding in nanocrystalline metals and their alloys: On the interplay of grain boundary sliding, coupled grain boundary motion and segregating solutes

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As the grain size is decreased into the lower nanometer (nm) range, the contribution of grain boundary (GB) mediated processes to low temperature plastic deformation such as GB sliding drastically increases [1]. In the past, Hahn and Padmanabhan [2] postulated that in nanocrystalline materials neighboring GBs can align themselves, leading to GB sliding on a mesoscopic scale (on the order of a grain diameter or more). Evidence for GB alignment was found in experiment and computer simulations [3, 4].

Depending on the type and misorientation of the boundary, however, it was shown by Cahn et al. [5] that GB motion can couple to shear deformation. Here, the GB moves perpendicular to the shearing direction and therefore out of the shear / sliding plane.

Most interestingly, the driving force for GB alignment as well as the coupling factor for GB motion increase towards a maximum as the GB becomes parallel to the shear direction. This, consequently, leads to a competition between GB alignment where deformation can occur as a cooperative motion and coupled GB motion, forcing GBs out of the potential sliding plane.

This competition between alignment and coupled motion is complicated by the presence of solutes in the GB, which evidently influence GB sliding [6] as well as coupled GB motion [7].

For studying mesoscopic grain boundary sliding in nc metals and the effect of segregating solutes, we utilize molecular dynamics simulations, where nc Cu and CuNb serve as model systems. We start with a special configuration of a nc microstructure (Fig. 1.) where a subset of grain boundaries is already aligned parallel to the direction of the highest shear stress.

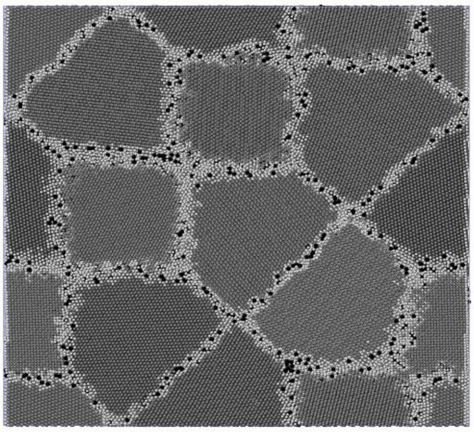

Fig. 1. Slice through a model structure created by the Voronoi tesselation method. A subset of GBs is aligned to form a mesoscopic slide plane.
(Dark grey corresponds to grain interior, bright grey to GBs and black to introduced solutes.)

By testing the structures under both, tensile and compressive load, we find that depending on the type of the GB, coupled motion out of the slide plane is observed. This inevitably leads to the destruction of the alignment and prohibits mesoscopic sliding.

For the case of samples with solute segregation, we show that it is a delicate function of composition, whether a given GB is pinned in place allowing for the development of mesoscopic sliding or can leave the introduced slide plane by coupled motion.

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24 August

BNM-2011 session

Processing of BNM. Deformation mechanisms, transformations and effects

August 24, 2011

Invited report

Principles for friction driven severe plastic deformation

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Keywords: *Tabular High pressure shear; deformation distribution, Finite element analysis; steady state criteria.*

Several existing SPD method: HPT, HPS, continouse ECAP and ECAR are using friction as the driving for severe plastic deformation. Recently new developed method HPS, tube high pressing twist (t-HPT) also use friction to drive the deformation. Tabular high pressure shear t-HPS (a combination of HPT and HPS) is developed and the principles are setup for deformation distribution calculation, steady state criteria, etc. t-HPS, a new SPD technique, has many unique advantages in comparison with previous SPD methods. In this paper, finite element analysis of plastic deformation behavior during HPCS of pure copper tube was conducted using the Johnson-Cook constitutive model. Strain distribution in radial and axial direction is observed. Simulation results indicate the advantage of the t-HPS technique, which promises its excellent application prospect.

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Invited report

Deformation behaviour of copper during the compression stage of high-pressure torsion

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In processing by HPT, the initial samples are in the form of thin disks and are placed in the HPT gap between a stationary upper anvil and a vertically and rotationally moving lower anvil, are subjected to a very high pressure (generally several GPa), and are strained by torsion. HPT processing consists of two stages based on the motion of the lower dies and the workpiece, as shown in Fig. 1: first the compression stage and next the compression-torsion stage. During the compression-torsion stage, the compressive pressure is generally kept constant. Since plasticity is path dependent, unlike elastic deformation, the deformation that occurs at both stage I (compression) and stage II (compression+torsion) is important for the properties and microstructures of HPT processed materials. Although many reports have been published recently on the effect of microstructural evolution and hardness distribution in HPT processed samples, and on torsional behaviour, no studies on the properties of samples after the compression stage haves been done, as far as can be determined. In this paper, we investigate the mechanical properties of copper after the compression stage in HPT by a systematic experimental approach. Microstructure, hardness and the distributions of microstructure and hardness were examined along the radial direction of compressed disks for various applied pressures in order to analyze the deformation and homogeneity in the workpieces during the compression stage, which controls the microstructure, mechanical properties and their homogeneities.

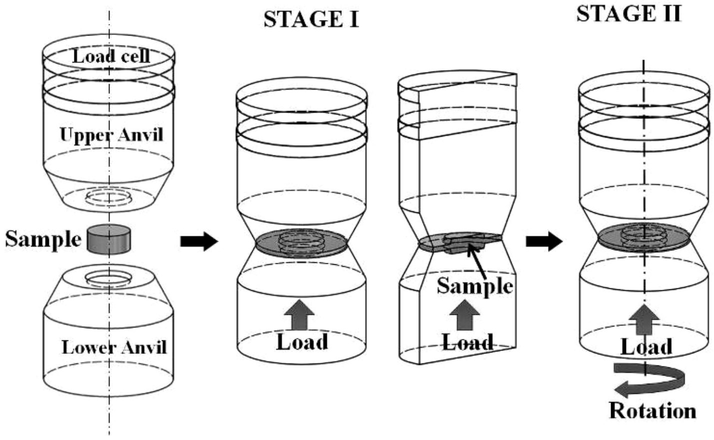

Fig. 1. Schematic of the HPT device showing set-up, compression stage (stage I), and compression-torsion stage (stage II).

This work was supported by a grant (B551179-08-01-00) from the cooperative research project "Development of common manufacturing technology for high functional susceptor" funded by the Ministry of Knowledge Economy, Republic of Korea.

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Oral report

Effect of multi-stage pack rolling on the structure and mechanical properties of pack-rolled ultra-fine-grained IF steel

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The present paper suggests the study of multi-stage pack rolling (MSPR) accumulation method technique on the structure and mechanical properties of ultra-fine grained (UFG) IF steel sheets using the severe plastic deformation (SPD). First, two or three sheets were stacked and joined together by rolling at 600°C. The rolled sheet then was cut into two halves that are stacked together. Thus, a series of rolling and stacking operations were repeated so that ultimately a large strain was accumulated in the sheet. It was produced a piles composed of 4, 8, 16 and 32 bonded by the rolling sheets with true strain e=0.7-5.6. The process has been realized using the laboratory rolling mill with 210 mm roll's diameter. Result in the ultra-fine grain structure (0.5-1 mkm) determined by TEM, was formed and the IF steel was strengthened dramatically (YS increased 3-4 times in compare with initial state).

With increasing of the number of the sheets in the pile from 2 to 8 the rolling stress increased in ~2 times, and with increasing from 8 to 16 – lowered up to 2 sheets pile ones. Increasing the number of the sheets (and accumulative strain correspondingly) up to 32 is neglible.

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Oral report

Impact toughness of ultra-fined grained interstitial-free steel

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Interstitial-Free steel (IF-steel) is a recently developed steel product with very low carbon content leading to excellent deep drawability and high planar anisotropy [1]. However, IF-steel in the coarse-grained (CG) structure possesses very low yield strength along with high ductility [2]. Because, the amount of interstitial carbon and nitrogen atoms inside the ferrite grains decreases by stabilizing the microstructure with micro-alloying elements of Ti and/or Nb [3]. Considering the mono-phase microstructure of IF-steel, there are limited strengthening methods to enhance its mechanical properties. Grain refinement via severe plastic deformation using equal-channel angular extrusion/pressing (ECAE/P) seems to be the most viable method since ultrafine-grained (UFG) structure has potential to improve the mechanical properties of materials [4-5]. Therefore, some efforts have been made to improve the strength and fatigue behavior of IF-steel by grain refinement via severe plastic deformation techniques [6-7]. On the other hand, toughness is an important property for structural materials like steels used under impact loading conditions [8]. In addition, the ductile-brittle transition temperature is an important parameter especially in HCP materials [9]. Although many studies have been published on the impact behavior of CG materials, little data can be found on the toughness of UFG materials [8]. Therefore, the purpose of the current study is to investigate the effect of grain refinement on the impact properties (fracture toughness and ductile-brittle transition temperature) of IF-steel. For this purpose, Ti-stabilized IF-steel billets were processed by ECAE following different routes and strains. The changes in both strength and toughness of UFG IF-steel were evaluated in terms of tension and Charpy impact tests. Charpy V-notch tests were conducted at various temperatures from liquid nitrogen temperature to ambient temperature on the miniaturized samples.

The CG (grain size: ≈30μm) microstructure of IF-steel billets substantially refined down to ultrafine-grained sizes (grains size range: ≈250-500 nm) after different ECAE processing. The UFG microstructure brought about a dramatically increased static strength in the expense of ductility (Fig. 1). This was attributed to the combination the effect of grain boundary and dislocation strengthening.

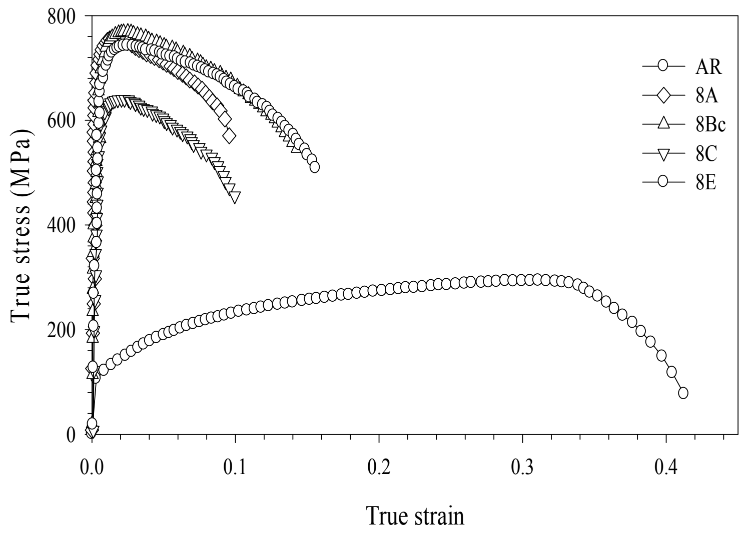

Fig. 1. Stress-strain curves of CG and UFG IF-steels

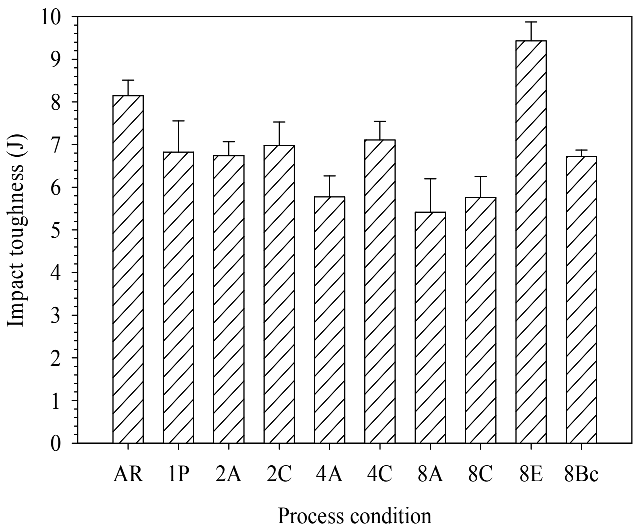

Fig.2. Effect of multi-pass ECAE on the impact toughness of IF-steel at room temperature

The Charpy V-notch impact energies showing the fracture toughness of CG and UFG IF-steel samples are shown in Fig. 2. From this figure, the CG IF-steel has an impact toughness of about 8 J. The effect of ECAE on the toughness depends on the processing routes as in the case of tensile properties. While route-E increases the impact toughness of CG steel, its toughness decreases slightly after processing with other routes. After processing for 8 passes, route-A, route-Bc, Route-C and Route-E have the impact toughness of about 5.4J, 5.8J, 6.8J and 9.4J, respectively. The lowest impact toughness values were obtained after processing via route-A. The differences between impact toughness values obtained in different ECAE routes was explained in terms of differences in the strain path leading to formation of different texture. As close-examined the tensile and impact test results, it can be seen that the impact toughness is strongly related to the ductility rather than strength. In other words, the toughness follows a similar trend with the total elongation, that is, an increase in elongation by the effect of strain path leads to improvement in toughness. The SEM micrographs showing the fractography of impact tested samples (not showing here) support this argument.

The preliminary results showed that the ductile-brittle transition temperature greatly decreased with grain refinement. This is an initial result and temperature-induced toughness of UFG IF-steel is under investigation for obtaining reasonable results about grain size–temperature-toughness relationship.

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Oral report

Shear banding and grain boundary sliding in ultrafine-grained aluminum under tension at room temperature

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It is well known that ultrafine-grained (UFG) metals and alloys processed by severe plastic deformation exhibit advanced mechanical properties in comparison with coarse-grained counterparts, e.g. enhanced strength at ambient temperature. From the other hand, a characteristic feature of plastic flow of UFG metals is limited uniform elongation due to rapid localization of strain in the neck region which is put obstacles in the way of their application. Certainly, the unusual mechanical behavior is due to the change of the ratio of contribution of different deformation mechanisms to the overall deformation. It is demonstrated recently that grain boundary sliding (GBS) and grain rotation occur in a number of UFG metals processed by severe plastic deformation including copper, nickel, aluminum and aluminum alloys even at ambient temperature. However, up to now the role of deformation mechanisms related to grain boundaries in the unusual mechanical properties of UFG metals is poorly understood.

Taking into account the above, the flow behavior and microstructure evolution as well as surface relief were investigated on tensile tested samples of UFG aluminum processed by ECAP using TEM, SEM and EBSD analysis. It was found that UFG aluminum exhibits no noticeable deformation hardening and yield stress practically corresponds to ultimate stress. Relief arising on preliminary polished surface of UFG aluminum during tension included three features corresponding to (i) microscopic dislocation slip inside grains, (ii) individual grain boundary sliding and (iii) shear banding probably due to co-operative grain boundary sliding. Contribution of GBS to the overall deformation was calculated. Structural characteristics were obtained in the near-surface regions where the GBS was visualized. The correlation between the structure and GBS development was discussed.

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Oral report

Creep in Al single crystal processed by equal-channel angular pressing

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In this work the creep behaviour and microstructure changes in Al single crystal during equal-channel angular pressing (ECAP) were investigated in order to better understand the relationships between ECAP microstructure and creep behaviour. It is known that microstructure evolution of single crystals during ECAP strongly depends on their crystallographic orientation [1, 2, 3].

The investigated crystal was oriented so that $\{111\}$ lay parallel to the shear plane and $\langle 011 \rangle$ was parallel to the direction of shear. Single-crystalline sample was subsequently transformed in the ultrafine-grained microstructure by route A (no rotation between subsequent ECAP passes) at room temperature.

Microstructure of samples processed by 1 and 4 ECAP passes was characterized by transmission electron microscope (TEM) and scanning electron microscope (SEM) equipped with the electron backscatter diffraction (EBSD). Tensile creep tests at temperature of 373 K and at applied stress of 50 MPa were performed on the Al single crystal after one and four ECAP passes. It was found that creep behaviour of Al single crystal depends on the number of ECAP passes (Fig. 1) and exhibits similar tendency as creep behaviour in polycrystalline Al processed by ECAP [4,5]. Examination of the surface of ECAP specimens pulled to the fracture revealed the formation of shear bands (Fig. 2) which created step relief on the surface. In particular the shear bands are observed near the fracture and are oriented near the shear plane of the last ECAP pass. The shear bands exceed considerably an average grain size. The creep fracture is probably influenced by inhomogeneity of structure on the mesoscopic level and arrangement of new high-angle grain boundaries created by ECAP.

Acknowledgment: This work was financially supported by the Czech Science Foundation under Grant 108/10/P469 and by the Academy of Sciences of the Czech Republic under the Institutional Research Plan AV0Z20410507 and project KAN300100801.

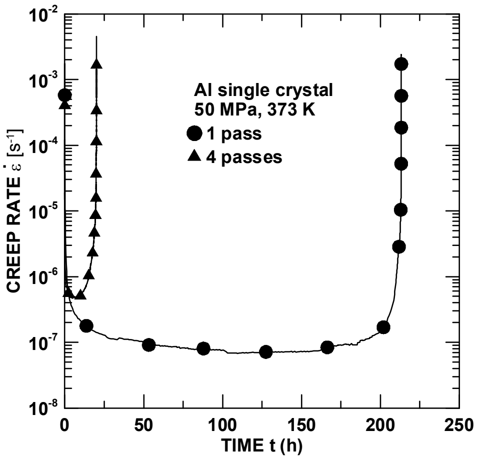

Fig. 1. Creep curves for two specimens processed by 1 and 4 ECAP passes

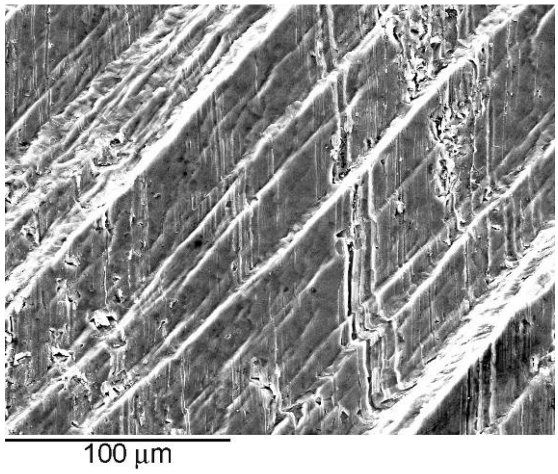

Fig. 2. Appearance of shear bands on the surface of single crystal processed by 4 ECAP passes

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Oral report

Direct and reverse martensitic transformation and nanostructured states formation during severe plastic deformation of metastable austenitic stainless steel

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A defect structure features and phase composition changes of 304 austenitic stainless steel after severe plastic deformation (SPD) by rolling and high pressure torsion (HPT) in Bridgman's anvils has been studied using transmission electron microscopy (TEM), X-ray diffraction and magnetization measurement technique. It is shown that SPD leads to formation of α' – and ε – martensitic phases. At true logarithmic strain $e \approx 1$ the volume fraction of ε – martensite does not exceed 4% and with the further deformation ε → α' martensitic transformation occurred. The volume fraction of α' – martensite increases with the strain and reaches ~ 50 – 70%. At true logarithmic strain $e \approx 4 – 7$ the volume fraction of α' – martensite reduces during SPD by high pressure torsion. These results suggest that the direct γ →α' and reverse α' → γ martensitic transformation occurred during HPT in 304 austenitic stainless steel.

TEM investigations of defect structure features show formation of heterogeneous phase composition consists of submicro- and nanoscales fragments. The regions with primary content of one of phases or two-phases (γ + α') regions were observed. It is shown that the defect structure of γ – phase consists of micro- and nanotwins and strain localization bands (SLB) with interior fragmented structure. SLBs have high angle misorientation with matrix close to ~ 60°<110>, ~ 50°<110> and ~ 35°<110>. Misorientated structures like this can be described due to the mechanism of local direct and reverse (γ → α' → γ) transformations of martensitic type with reverse transformation on alternative systems [1, 2]. Banded structures with high- and low-angle misorientated fragments were formed in martensite. Some of this fragments (including SLBs fragments of α' – phase) have misorientations close to ~ 60°<110> and ~ 35°<110>. The formation of thin nanoscales plates of ε – martensite was found inside individual fragments of α' – phase.

Based on the results obtained, the mechanisms of phase transformation and nanostructured states formation in 304 austenitic stainless steel produced by SPD are discussed.

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Oral report

Hydrofluoric acid treatment effect on surface condition of commercially pure titanium Grade 4

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At the present moment commercially pure titanium Grade 4 (CP Ti Grade 4) is one of the most advanced materials for medical implants due to excellent biocompatibility, high ultimate strength, high yield point and desirable relatively low density and low elasticity modulus. Moreover, the tissue response to a dental implant may involve physical factors such as size, shape, surface topography, and relative interfacial movement, as well as chemical factors associated with the composition and structure [1]. Characterization of implant material for evaluation of tissue reaction necessarily includes analysis of surface composition and impurity [2].

The surface of CP Ti Grade 4 before and after chemical treatment (in 1% HF and 40% HF during 1 min) was investigated with the help of XPS measurements.

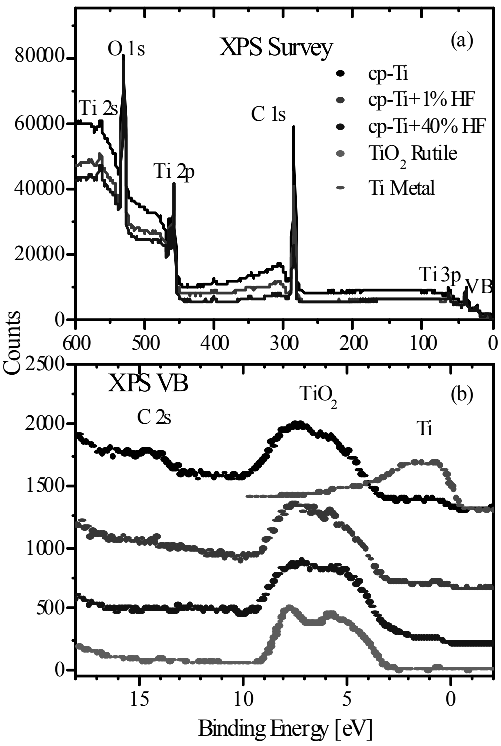

Fig. 1. (a) XPS wide scans of coarse-grained CP Ti Grade 4 before and after chemical treatment in hydrofluoric acid (1 and 40% HF). (b) XPS valence bands of initial and acid treated CP Ti Grade 4.

XPS wide scans of coarse-grained CP Ti Grade 4 before and after chemical treatment in hydrofluoric acid (1 and 40% HF) are presented in Fig. 1 It was found that acid treatment of CP Ti Grade 4 reduces by 1.5-2.6 times the content of hydrocarbons increasing the surface energy and biocompatibility of Ti-implants. XPS Ti 2p-spectra show that the surface of CP Ti Grade 4 is covered by thick TiO2 (Ti4+) oxide layer with rutile structure. It was established that the chemical treatment changes the near- surface oxide layer composition, increasing TiO (Ti2+) content from the surface to bulk. The presence of divalent Ti can provide excellent biocompatibility of chemically treated CP Ti Grade 4 due to Ti^{2+} substitution of Ca^{2+} It is important for dental applications to determine the crystal structure of TiO_2 layer. XPS Ti 2p spectra of anatase and rutile [3] have similar fine structure but the binding energies are found to be slightly different to about 0.3 eV.

Data on hydrofluoric acid treatment effect on surface condition of nanostructured CP Ti Grade 4 are also presented and discussed.

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Invited report

Respond of structural changes on deformation behaviour of ECAP processed aluminium alloy AA7075

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Strength of materials subjected to severe plastic deformation can be significantly improved due to ultrafine grain structure formation. The impact of various strain levels ε_{ef} on structure refinement and proportions different portions of grains with high angle boundaries and subgrains having low angle boundaries is present. Any severe plastic deformation procedure (ECAP, HPT, ARB, and CGP) refines grains to size below 1000 nm and is usually accompanied by strength increase and reduction in ductility.

In order to improve the deformation behaviour of SPD prepared aluminium alloy AA7075 microstructure modification by ageing due to various holds at precipitation temperature prior to ECAP processing and post-deformation treatment were carried out. Over-aging process carried out prior severe deformation modified the characteristics of precipitates (intermetallic phases) present in alloy. The microstructure refined by intensive deformation was then altered by various annealing procedures. Ageing prior to ECAP contributed to a small increase in strength, as hardness results, as shown by results of hardness measurement and observation of microstructure. On the other hand, post-deformation annealing of the ECAP-ed samples at various temperatures markedly modified the deformation response of the alloy as regards ductility, Fig.1a,b. Evaluating the mechanical properties a small increase in strength was achieved at lower temperature of at lower temperatures of annealing (250°C, 300°C), whereas the highest temperature of annealing (350°C) preserved the strength at the same level as in samples without any additional post-deformation annealing. The microstructure analyses (TEM and EBSD) provided evidence about the ultrafine grain structure transformation towards formation of bimodal structure. These preliminary results of purposeful structure modification will be further examined in regard to strength increase.

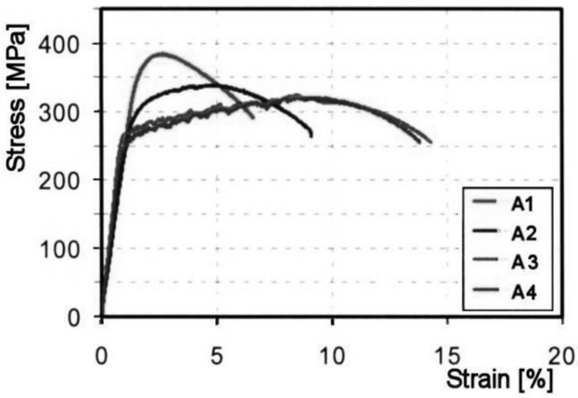

Fig. 1a. Stress-strain curves for AA7075 alloy without post-deformation annealing

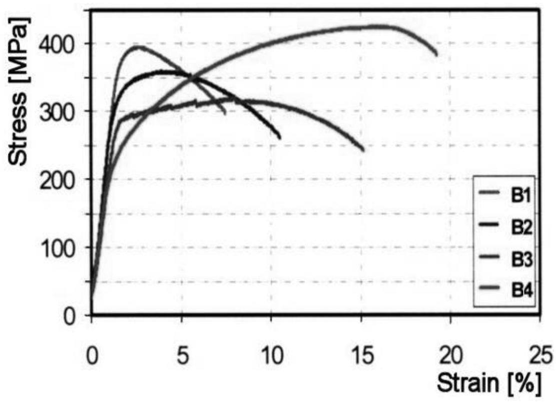

Fig. 1b. Stress-strain curve for AA7075l alloy with post-deformation annealing at 250°C/1h

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August 24, 2011

Invited report

Structural-scaling transitions in polycrystals, thermodynamic and mechanical behavior of bulk submicrocrystalline materials

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Bulk nanostructural materials (BNM) prepared by the methods of intensive plastic deformation are characterized by specific state of ensemble of grain-boundary defect ensemble with long range spatial correlation that provides the unique mechanical behavior of this class of materials [1,2]. Physical properties of BNM are caused by the length and the development of grain boundaries, which for the grain sizes about 10-100 nm consist of 10-50% of atoms of material. As the consequence, the transition to bulk nanocrystlalline state is characterized by pronounced scaling effects and the change of material properties linking with the decreasing of particle size and the increasing role of grain boundary defects. Most important in the study of physics of BNM is the question about the existence of sharp boundary between bulk polycrystalline and nanocrystalline state, i.e. the existence of area below some characteristic grain size, where the properties are characteristic for nanocrystalline solid. There is also thermodynamic statement of problem concerning the analogy of transition from polycrystalline to nanocrystalline state and the first kind of phase transition [3]. This question can be considered as the key point and the unique properties of nanostructural materials can be used in the application under resolving of mentioned fundamental problems.

Statistical theory of collective behavior of mesodefects was proposed in [4] and allowed the establishment of new class of critical phenomena in dislocation substructures (grain boundary defects) – structural-scaling transitions.

Results of statistical theory were used for the formulation of statistically based thermodynamics and the phenomenology of polycrystalline and nanocrystalline states. Phenomenological approach represents the generalization of the Ginzburg-Landau theory for the out-of-equilibrium systems with mesodefects and allowed the definition of characteristic solid states (quasi-brittle, ductile and fine grain states) in terms of collective mesodefect modes [5]. It was shown the links of kinetics of these modes with the mechanisms of structural relaxation under plastic shear, damage localization. Qualitative difference of polycrystalline and bulk nanocrystalline states is the consequence of the scaling transition under the pass of some characteristic grain size. Qualitatively this transition is analogous with the first kind of phase transition and the grain refining leads to the degeneration of collective orientation mode of defects from the auto-solitary shear mode providing the plastic relaxation to the formation under the pass of “scale” critical point the quasi-periodic finite-amplitude sub-lattice of mesodefects. Qualitative change of the types of collective modes leads to the change of global symmetry properties of out-of-equilibrium system “solid with mesodefects” and different mechanisms which responsible for structural relaxation and plastic flow, diffusion properties, damage-failure transition. The scaling regularities at the critical point of transition from polycrystalline to bulk nanocrystalline states allowed the explanation of breaking of the Hall-Petch law for the yield stress under the grain refining and the anomaly of energy absorption under mechanical loading (cyclic and dynamic) was predicted theoretically. Experimental support of the anomaly of energy absorption was realized under the comparative experimental tests coupled with infra-red scanning of temperature field for coarse grain and fine grain titanium specimens subject to cyclic and dynamic loading [5].

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August 24, 2011

Oral report

Processing strip and wire specimens by continuous high-pressure torsion

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Processing bulk metallic materials through the application of high-pressure torsion (HPT) leads to attainment of ultrafine-grained structures with a high strength and reasonable ductility. As a main merit of HPT, since the process is conducted under high hydrostatic pressures, the fracture is significantly suppressed and thus it is applicable to hard and less ductile metals. The HPT process has been used in a form of disc or ring but it is strongly desired to be performed in a form of strip or wire although the sample size is currently extended to 100 mm in diameter for rings [1]. Recently, a new severe plastic deformation method, which we call continuous high-pressure torsion (CHPT), has been developed for processing of metallic strips [2] and wires [3] with HPT in a continuous way which can be a potential process for industrial application.

The facility for the CHPT is schematically illustrated in Figs. 1 and 2, for strip and wire samples, respectively. It consists of a lower anvil, which is rotated during process, and an upper anvil, which is fixed during process. For strip samples, the lower anvil has a flat surface with a roughened ring-shaped area; and the upper anvil has a half ring-shaped groove on the surfaces with 0.5 mm depth, 3 mm width and outer diameter of 20 or 30 mm. For wire samples, the upper anvil has a roughened ring-shaped circular groove with 2 mm cross-sectional diameter, 0.9 mm depth and outer diameter of 40 mm. A U-shaped strip sample with 1mm thickness and 3 mm width or a wire with 2 mm diameter, are used as initial samples. Each sample is placed on the lower anvil and the pressure is applied on the sample. The lower anvil is then rotated with respect to the upper anvil and the material starts to flow in the rotation direction.

In this study, the CHPT was applied to pure Al (99.99%), Cu (99.99%) and Fe (99.96%). Transmission electron microscopy observations showed that CHPT can be used for producing ultrafine grained microstructure in strip and wire samples as the conventional HPT using disc and ring samples. The results of hardness measurements after CHPT were well consistent with those of HPT using disk and ring specimens.

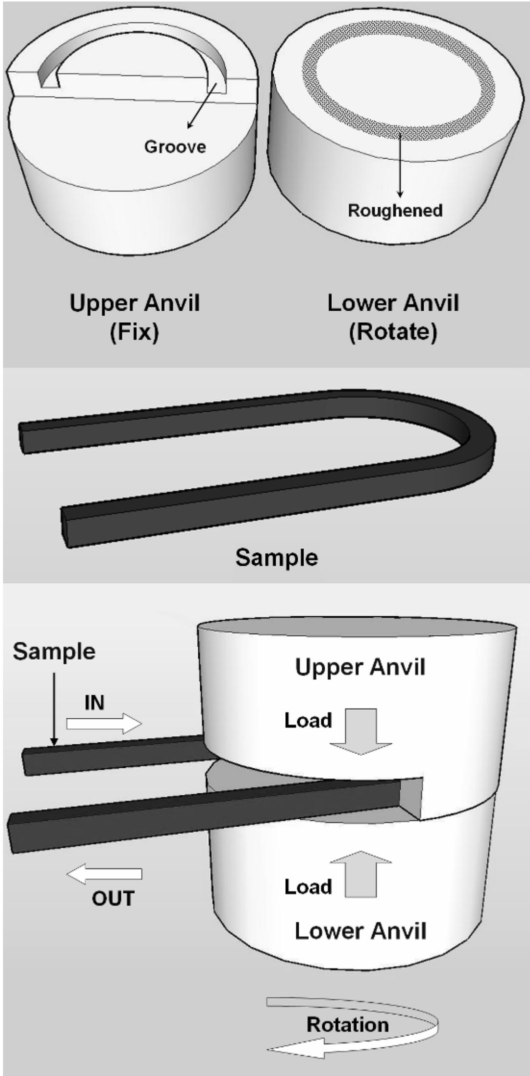

Fig. 1. Schematic illustration of CHPT for Strip samples

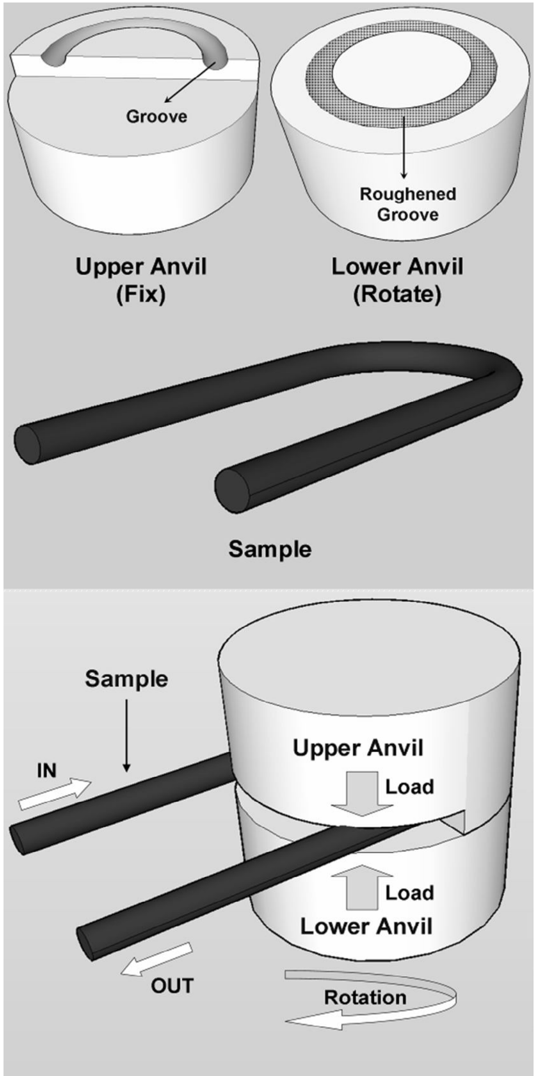

Fig. 2. Schematic illustration of CHPT for wire samples

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Oral report

Degree of strain and grain refinement during plastic deformation

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Severe plastic deformation (SPD) methods are widely used to obtain nanostructured metals and alloys. Nanostructured materials are interesting for various applications due to their high mechanical properties. In the present work it is demonstrated that, depending on the SPD method, the lowest possible grain size can vary by several times and the achievable degree of strain can vary by orders of magnitude. Such a large difference in the estimation of degree of deformation is explained by the fact that different formulae for calculation of strain use different components of the distortion tensor. Compatibility of large plastic strain and the mechanisms of structure transformation are strongly related to the rotational part of the distortion tensor. We offer a method of taking into account the rotational part of the distortion tensor and its use in solving the problem of grain refinement during SPD. Conditions on plastic deformation that lead to minimal grain size are formulated.

August 24, 2011

Oral report

Cycling stability of martensitic transformation in shape memory TiNi alloys subjected to ECAP

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TiNi shape memory alloys (SMAs) have been widely used in the engineering applications due to their excellent functional properties, such as shape memory effect, superelasticity and high damping property. Thermomechanical processing has been proven to be an effective method to improve the mechanical and functional properties. Recently, equal channel angular pressing (ECAP) was used to prepare bulk ultrafine-grained (UFG) TiNi SMAs [1]. To date, several important aspects of TiNi alloys subjected ECAP have been reported, including microstructure, phase transformation, mechanical properties and shape memory effect [2] etc. However, the cycling stability of martensitic transformation in the ECAPed TiNi SMAs remains unknown. In the present work, $Ti_{49.2}Ni_{50.8}$ and $Ti_{50.2}Ni_{49.8}$ alloys after ECAP were prepared and annealed at different temperatures from 300 to 600 °C for 30 min with a temperature interval of 100 °C. For comparative purpose was used coarse grained state obtained solution-treatment at 900°C for 2 h and followed quenching in water. In the present work, structural features of UFG $Ti_{49.2}Ni_{50.8}$ and $Ti_{50.2}Ni_{49.8}$ alloys produced by ECAP and possessing a mean grain size of 260 and 320 nm, respectively, was considered and discussed. The cycling stability of martensitic transformation was studied as a function of annealing temperature. Figure 1 shows DSC curves of $Ti_{49.2}Ni_{50.8}$ and $Ti_{50.2}Ni_{49.8}$ alloys after ECAP. It is seen than the forward martensitic transformation of UFG $Ti_{49.2}Ni_{50.8}$ alloy is characterized by a two-stage manner, which is related with increasing in volume fraction of grain boundaries which makes difficult a direct B2→B19' transformation [3]. After five thermal cycling, the transformation behaviour of the $Ti_{49.2}Ni_{50.8}$ alloy after ECAP does not show any obvious change, however, the transformation temperature of UFG $Ti_{50.2}Ni_{49.8}$ alloy decreases with increasing the cycling number, indicating that the former has a better cycling stability than that of the latter. The nature of these distinctions discussed.

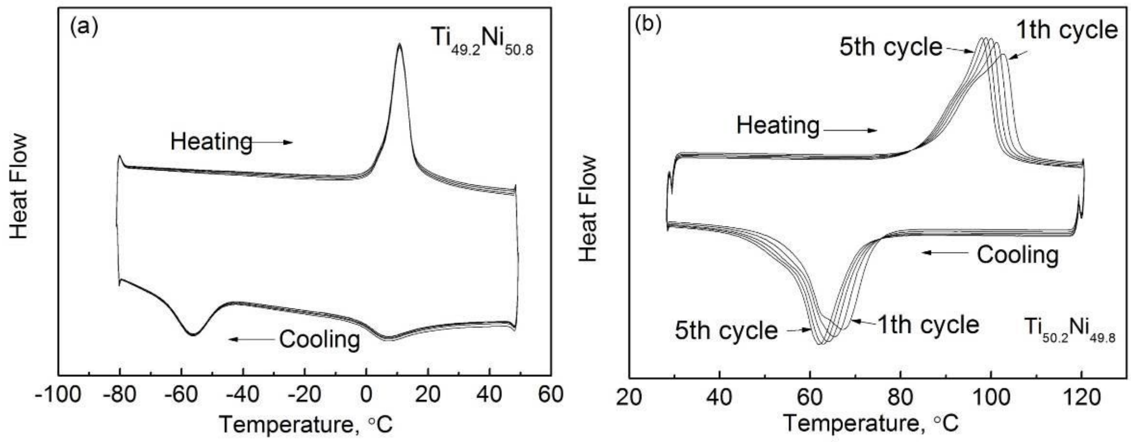

Fig. 1. DSC curves of the $Ti_{49.2}Ni_{50.8}$ (a) and $Ti_{50.2}Ni_{49.8}$ (b) alloys after ECAP

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Oral report

Effect of reversible hydrogen alloying on nanostructure formation in α-titanium alloys

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Combination of severe plastic deformation at $T_d \approx (0.4\text{-}0.5)T_m$ with reversible hydrogen alloying allows to produce bulk nanostructured billets out of two-phase titanium alloys. Grains/fragments, of several tens nanometer in size, form due to simultaneous or successive development of polygonization/recrystallization processes together with hydrogen-induced phase transformations. Their role in the formation of nanostructure is not clear yet because of the difficulties associated with separation of the abovementioned processes in heavily alloyed titanium alloys. It is worth to study the effects of reversible hydrogen alloying in the single α- and β-titanium alloys. The goal of the present work was to study the effect of dissolved hydrogen on α-grains refinement during warm deformation and subsequent hydrogen removal by vacuum annealing.

Commercial pure (CP) titanium VT1-0 (Ti-0.32Al-0.1Fe-0.07Si-0.05C-0.035N-0.1O-0.003H wt.%) and α-titanium alloy VT5-1 (Ti-5.4Al-2.8Sn-0.01Zr-0.03Fe-0.01Si-0.11O-0.03C-0.01N-0.002H wt.%) were studied. Both alloys had initial coarse-grained lamellar structure with the thickness of α-lamella of 10–15 μm. Cylindrical samples were subjected to vacuum annealing and subsequent soaking in a pure hydrogen atmosphere under different gas pressures. After such treatment, samples with different hydrogen content (C_H) ranging from 0.1 to 5.3 at. % (0.002 – 0.12 wt. %) were prepared. The hydrogen levels were determined by weighing the samples before and after hydrogenation to the nearest 5×10^{-5} g. The samples of the base alloys with C_H=0.1 at. % were treated under the same conditions without soaking in the hydrogen atmosphere. The samples were subjected to deformation at temperatures varying from 380 to 750°C. Plates with thickness of 1.4 mm were cut from the centre part of the deformed samples and annealed in vacuum at temperatures 500–650°C. Structure of the samples after different steps of treatment was examined by optical, scanning (SEM) and transmission electron microscopy (TEM) techniques.

It was shown that hydrogenating treatment did not lead to change in the sizes of α-grains, colonies and lamellar thickness. The deformation was accompanied with development of polygonization/recrystallization processes which caused transformation of initial lamellar structure to equiaxed one. The increase in C_H led to decrease in the size of equiaxed α-grains observed by SEM in the etched samples and effected slightly on the size of misoriented fragments observed by

TEM. The effect of hydrogen on the α-grains refinement is more significant in CP titanium. The difference between sizes of the equiaxed α-grains and the fragments decreased with hydrogen addition and at C_H=5.3 at. % these sizes were about the same. The measurements of the angles between zone axes of the adjacent fragments revealed their increasing with an increase in C_H in α-solid solution. It was shown that the volume fraction of the equiaxed structure increases with hydrogen addition. The structure with grain/fragment size about 0.5 μm may be obtained in VT1-0 and VT5-1 alloys with different C_H after deformation at 450 and 650°C, respectively. Microstructure of the alloys was finer in the case of lower deformation temperatures.

Structure development of the deformed samples during vacuum annealing was accompanied with static polygonization/recrystallization processes, hydrides dissolution and removal of soluted hydrogen. During the vacuum annealing at tested temperatures the grain growth was observed in VT1-0 alloy while grain refinement took place in VT5-1 alloy. The higher was C_H in the VT5-1 alloy before vacuum annealing, the smaller was grain size after hydrogen removal.

Thus, hydrogen soluted in the α-phase of titanium alloys promotes development of dynamic polygonization/recrystallization processes and formation of nanostructure in the alloys. Hydrogen removal may lead to additional structure refinement if the temperature of vacuum annealing is lower than deformation one and the α-phase contains α-stabilizing substitution elements.

25 August

THE SECOND RUSSIN-FRENCH-GERMAN WORKSHOP

Atomic transport in BNM and related unique properties

Invited report

Grain boundary diffusion in SPD materials: characteristics, oddity and mechanisms

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Recently we have initiated a systematic investigation of grain boundary diffusion in SPD-processes materials, especially in pure metals like Cu, Ni, Ti and Ag. In this talk, the obtained results are systematized and discussed.

The following main findings can be highlighted:

- Existence of a hierarchy in diffusion properties in severely deformed metals, which implies the formation of different types of interfaces on different scales with significantly different properties.
- A versatility of ultra-fast diffusion paths in SPD materials which include pores/microcracks and so-called "non-equilibrium" grain boundaries.
- An intricate and unusual defect can even appear – the percolating porosity, which develops presumably along critical triple lines separating selected non-equilibrium interfaces.
- A relaxation of the non-equilibrium state and the redistribution of free volume defects are clearly observed as a result of a heat treatment of the deformed materials.
- Both enhancement and retardation of diffusion rates along non-equilibrium grain boundaries can be observed in dependence on the diffusion mechanism of the solute.

These results are correlated with detailed observations of microstructure evolution and DSC and X-ray diffraction data.

A model of solute diffusion in non-equilibrium interfaces is proposed and tested by atomistic molecular dynamic simulations.

Funding by Deutsche Forschungsgemeinschaft is gratefully acknowledged.

Oral report

Role of grain boundary sliding, texture and activation energy in understanding creep flow in HPT-processed ultrafine grained copper

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In pure metals with a processing history which includes severe plastic deformation (SPD), an ultrafine grain size and the presence of defects of high excess energy densities lead to changes in the basic mechanisms that accommodate externally applied mechanical stresses, e.g., grain boundary diffusion and boundary sliding may account for the rate controlling deformation process in this sub-micrometer grain size range [1]. To study the time-dependent deformation (creep) behavior of such materials, copper samples were prepared by high pressure torsion straining [2], using a hydrostatic pressure in the range of 1-4 GPa. The samples were cut to a dog bone shape and polished to facilitate electron backscattering diffraction measurements (EBSD) in the scanning electron microscope (Fig. 1).

For deciding on the rate controlling process, the strain rate sensitivity and the activation energy for rate controlling flow were determined by (tensile) load jump tests at low and medium homologous temperatures over a wide range of strain rates. In addition, with the use of a gas injection system, platinum was deposited by an electron beam to scribe a fine grid on the specimen surface. These markers offer, subject to certain precautions and up to a maximum strain of about 10-20 % [3], the possibility of measuring the surface grain strain and the strain associated with grain boundary sliding [4].

Moreover, microstructure characterization by EBSD allowed the estimation of the changes in grain size distribution, texture and misorientation of grain boundaries. The results are discussed, in combination with the activation energy and stress exponent values measured by the load jump and different strain rate tests and the contribution of grain boundary sliding as revealed by the marker experiments, to derive meaningful information on the rate controlling flow mechanism in this class of (SPD) materials.

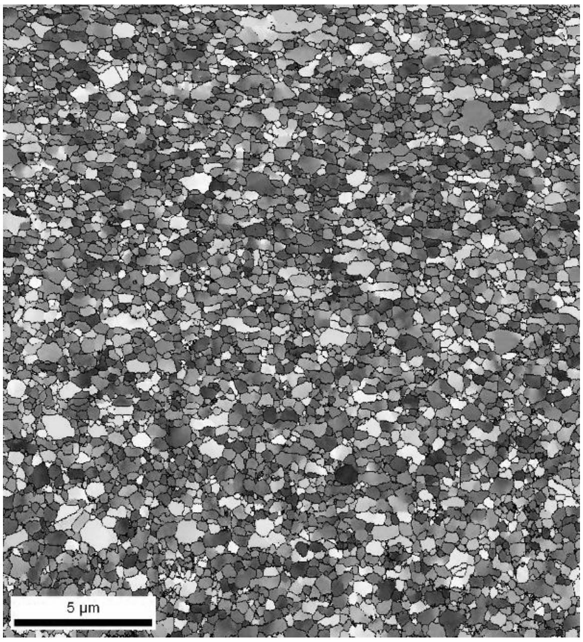

Fig. 1. EBSD measurement of a Cu sample deformed by HPT with a shear strain of γ =21 at 4GPa hydrostatic pressure. The average grain size is ≈400nm

The support of this work by the Deutsche Forschungsgemeinschaft is gratefully acknowledged.

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August 25, 2011

Oral report

Microstructure and texture of IF steel processed by high pressure tube twisting (HPTT)

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High Pressure Tube Twisting (HPTT) is a process of severe plastic deformation which is suitable for deforming a thin wall cylindrical tube to very high strains in one single operation [1]. It was derived from the High Pressure Torsion technique [2] and Cone-Cone Shearing processes [3]. Industrial application of this new SPD technique seems realistic in the near future. Results obtained on IF Steel are presented in this work.

A schematic representation of the experimental set-up is shown in Figure 1. The sample is constrained between the lower and the upper die. High hydrostatic pressure is achieved by axial compression of a cylindrical mandrel placed into the tube. The tube is twisted by an external torque with the help of the friction force generated by the hydrostatic pressure. A new high capacity experimental device is under construction.

Assuming homogeneous deformation, the shear strain can be estimated by $\gamma = r_0\beta / t$, where β is the angle of twist, r_0 is the average radius, and t is the thickness of the tube. For dimensions of r_0=7.5 mm and t =1 mm, one turn corresponds to a theoretical shear strain of $\gamma = 47$.

Experiments were performed successfully on different aluminium alloys, copper and IF steel. Generally a strain gradient was observed through the wall. Finite element simulations were carried out using Abaqus software to investigate the heterogeneity of strain across the thickness of the tube (Figure 2).

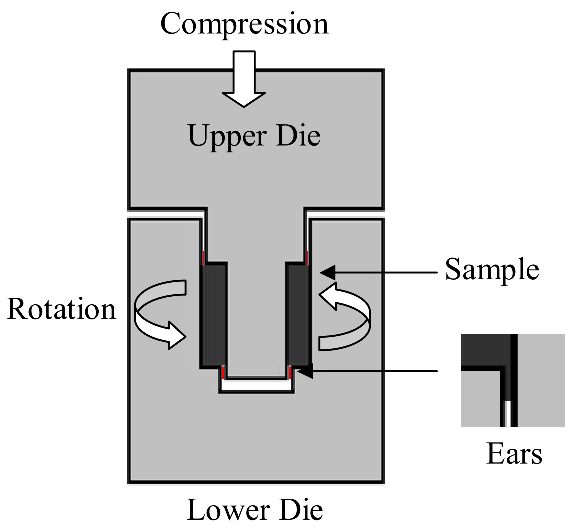

Fig. 1. HPTT experimental setup

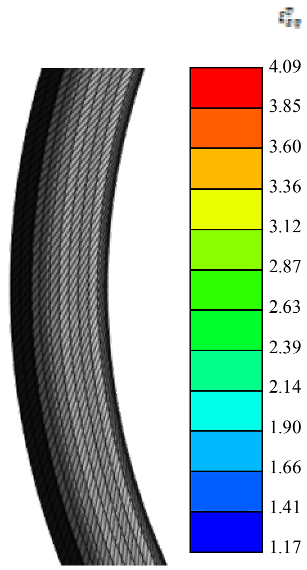

Fig. 2. FE simulated equivalent plastic strain in the tube wall

Texture development, grain refinement and strain hardening were also simulated using the newly developed grain refinement model [4]. The model is based on the lattice curvature that is induced near the grain boundaries due to the rotation of the lattice. The present paper will report about the experimental and simulation results obtained for HPTT deformation of IF steel tubes.

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August 25, 2011

Oral report

Texture evolution in nanocrystalline Ni subjected to high pressure torsion

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Electrodeposited Ni with nanocrystalline grain size was subjected to high pressure torsion to 0.5 and 5 rotations under the pressure of 6GPa. Texture studies were performed using diffractometer with a beam size collimated to 500 μm to study areas of the samples which are characterized by similar applied strain and shear direction. It is shown that at lower strains significant scattering of texture was observed while at larger strains a typical shear texture was formed. The observed features are interpreted in terms of deformation mechanisms operating at different stages of samples' deformation.

August 25, 2011

Oral report

Nanostructures and magnetic properties of FePd alloys processed by severe plastic deformation

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The intermetallic FePd alloy was processed by severe plastic deformation (high pressure torsion) both in the ordered and the disordered state. The resulting materials are nanostructured with a grain size smaller than 100nm. Using x-ray diffraction, transmission electron microscopy and magnetic properties measurements some strain induced disordering was revealed. The ordering kinetics during post deformation aging treatment was also investigated. In the optimum state, nanoscaled ordered domains give rise to a record coercivity of about 1800 Oe.

Oral report

Radiation resistance of ultrafine-grained austenitic stainless steels

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Due to a high number density of grain boundaries acting as point defect sinks, ultrafine-grained materials are expected to be more resistant to irradiation damage. In this context, ultrafine-grained 316 austenitic stainless steel (UFG-316) samples have been fabricated by high pressure torsion. Samples after severe plastic deformation exhibit a grain size of about 40 nm. Ion irradiations at 350°C have been performed on these UFG-316 samples and their microstructural evolution under irradiation has been studied using atom probe tomography. Results are compared with those obtained in an ion irradiated conventional coarse-grained steel.

While a high number density of Ni and Si enriched clusters are observed in the conventional material, the repartition of solute atoms is homogeneous in the UFG-316. The comparison also shows that the effects of irradiation are limited at grain boundaries. Indeed, the radiation-induced segregation intensity is lower in the UFG-316 than in the conventional material. Using cluster dynamic modeling, results are interpreted by a higher annihilation of point defects at grain boundaries in the ultrafine-grained steel.

August 25, 2011

Oral report

Deformation behavior and spall fracture of ultra-fine grain heterophase alloy Al-Mg-Li-Zr irradiated by nanosecond relativistic high-current electron beam

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The investigation of deformation behavior and spall fracture mechanism of heterophase alloy Al-Mg-Li-Zr with ultra-fine grain and coarse grain structures after irradiation by nanosecond relativistic high-current electron beam was realized. Ultra-fine grain structure was formed with intensive plastic deformation by isothermal multiple all-round pressing technique. The irradiation of targets has been carried out on «Sinus – 7» accelerator with electron energy 1,3 MeV, pulse duration 50 ns, power density $7\cdot10^9$ W/cm^2. Computer modeling at above-mentioned parameters of the electron beam had shown that in zone energy liberation the temperature reaches melting temperature and it is forming the monopolar wave, which spreading causes deformation with velocity 10^5 c^{-1}. At the shock wave reflection from rear surface target it is form the sprain wave, which amplitude decreases with increasing of target thickness. Hereupon the thickness of spall layer increases with increasing of irradiated target thickness.

In experimental investigations targets were in the manner of discs with thickness from 3 to 9 mm and with ultra-fine grain and coarse grain structures (the sizes of grain - subgrain structure elements were less 1 μm). The phase composition of all targets was identical: there are large particles of Al_2LiMg phase and nano-size particles of $Al_3Li(Zr)$ phase. It was established that thickness of spall layer increases and plastic deformation degree decreases with increasing of target thickness. The magnitude of spall strength equals approximately 1,4 GPa.

The changes of structured-phase state not far from spall-fracture surface were reveälled by means of electron-microscope investigation. In particular, the reducing of grain - subgrain structures sizes before 100 - 300 nm and increasing of dislocation density in grains were discovered at the both grain structures.

It is shown that fracture mechanism of Al-Mg-Li-Zr alloy under dynamic loading is identical to ones under quasi-static loading. It is ductile trans-crystalline or inter- crystalline fracture at initial ultra-fine grain structure, but it is brittle inter- crystalline fracture at initial coarse grain structure. At the beginning before the fracture there are appearance of microcracks and then appearance mesocracks with the following merging them in macro-crack. At the big thickness of target two macro-cracks are forming on different distances from rear surface, and then the spalling goes through one of these cracks.

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Invited report

Mg segregations along grain boundaries in an aluminium alloy processed by SPD

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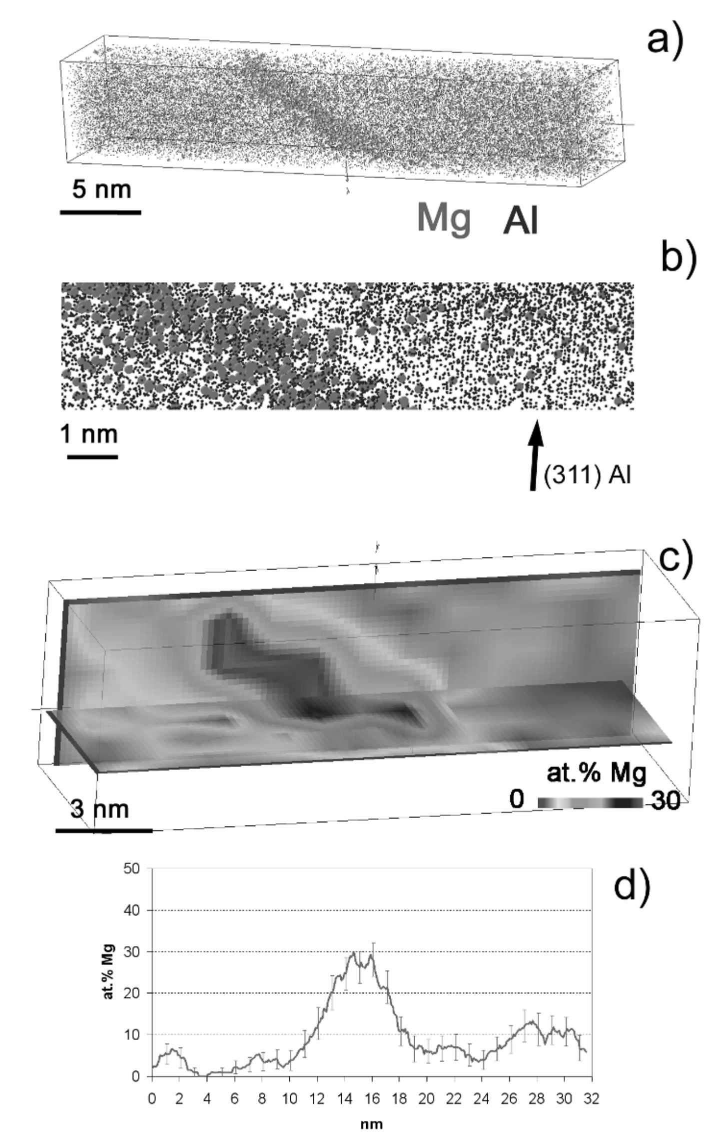
It is well established that impurities and alloying elements in solid solution may have a significant influence on the grain size refinement induced by Severe Plastic Deformation (SPD). However, the exact underlying mechanisms like possible interactions between solute atoms and crystalline defects (vacancies, dislocations or Grain Boundaries (GB)) are still not fully understood. To clarify this point an Al alloy containing 5.7wt. % Mg in solid solution was processed by High Pressure Torsion (HPT) and the 3D distribution of Mg atoms was characterized by Atom Probe Tomography (APT). In the Ultra Fine Grained (UFG) structure resulting from SPD at room temperature, some significant Mg segregations were exhibited. HPT at 200°C also leads to a UFG structure, however segregations seem less pronounced. Comparing to the undeformed state where a Mg depleted GB was successfully analyzed, it is assumed that SPD induced segregations result from the non equilibrium state of the boundaries in the UFG structure. It is also worth noticing that the SPD processed alloy exhibits an extremely high yield stress, up to 900MPa. The influence of the observed Mg segregations along GBs on the strengthening is discussed.



3D reconstruction of an analyzed volume in the UFG 1570 alloy;
(a) full data set showing a planar segregation of Mg
(Al atoms are displayed in dots and Mg atoms in bubbles);
(b) selected part orientated to display (311) Al atomic planes on the right of the planar segregation;
(c) 2D chemical map showing the Mg concentration fluctuations within the volume;
(d) concentration profile computed across the segregation (sampling volume thickness 1 nm) – From [1].

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August 25, 2011

Oral report

Mechanical properties and nanoscale features in a severely deformed steel alloyed with Si and Ti

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Severe Plastic Deformation (SPD) has a great potential to produce bulk nanostructured metals and alloys for use in a wide range of structural applications. In this work, steel alloyed with Silicon (Si) and Titanium (Ti) (Fe-2.5wt%Si-1wt%Ti) was deformed by Equal-Channel Angular Pressing (ECAP) which is one of the major well-established SPD processing techniques, leading ultimately to a ultrafine-grained (UFG) structure, making the Fe-Si-Ti alloy as a potential candidate for automotive applications. In particular, the Fe-Si-Ti alloy system is known to exhibit an interesting hardening potential thanks to its ability to produce a fine and dense precipitation microstructure.

Understanding the atomistic processes which occurred during SPD is of particular relevance to improve mechanical performances like increasing yield stress and strain-hardening. To this end, in the Fe-Si-Ti alloy, we were interested to analyse the solid solution effects on the refinement mechanisms. Moreover, ageing treatments were performed in order to understand the interplay between the ultrafine-grained size structure and the precipitated particles. To interpret and to elucidate the relationship between microstructure evolution and mechanical properties, experimental characterizations were performed in the hardening steel using Atom Probe Tomography and Transmission Electron Microscopy.

Oral report

Mechanical properties and fracture of nanostructured TiNi alloys during tensile tests at various temperatures

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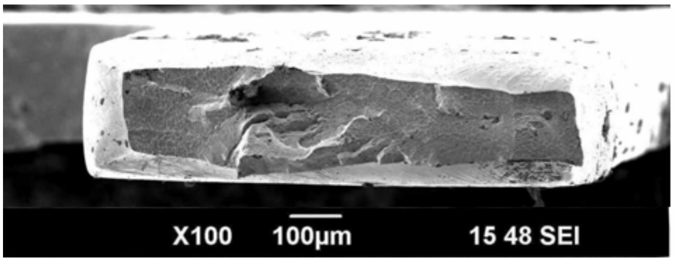
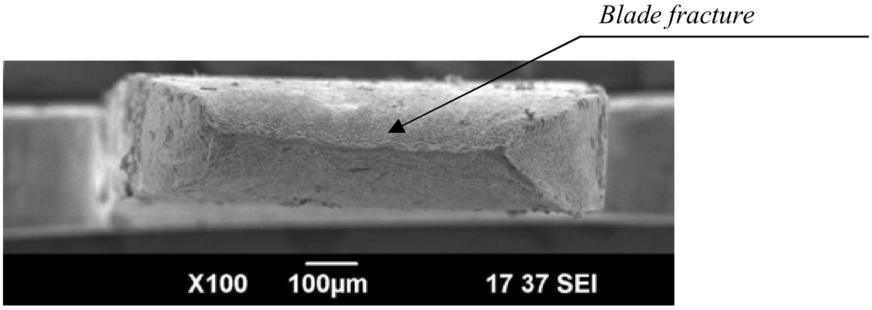
TiNi-based alloys are known as functional materials with the shape memory effect (SME) with superior service characteristics. Strength and characteristics of SME alloys can be significantly enhanced due to formation of a nanocrystalline (NC) structure by severe plastic deformation (SPD). The amorphized state of the TiNi alloy is formed as a result of high pressure torsion (HPT) effect. Subsequent annealings result in formation of nanocrystalline states in TiNi HPT samples [1, 2]. The nanocrystalline TiNi alloys processed by HPT at room temperature have enhanced strength and yield stress in comparison with their coarse-grained counterparts [1, 2]. In the present work the mechanical properties of nanostructured TiNi alloys processed by HPT at increased temperatures have been studied as there is not much information on this subject in earlier reports [2].

The TiNi alloy samples have been processed with the help of unique die-set and HPT technique which allows to fabricate TiNi samples with a diameter of 20 mm and a thickness of up to 1 mm at high strain degrees (e>5) and pressure (to P=6 GPa) [1]. The amorphized TiNi samples have been processed by HPT at room temperature, pressure P = 6 hPa and 5 rotations of anvils (n). The mechanical tensile tests have been performed on a unique die-set which allows testing small samples with the gauge 4x1x0.2 mm, cut out of HPT disks. The mechanical behavior of specimens has been investigated at temperatures from 20 up to 600° C with strain rate from 10^{-1} to 10^{-4} 1/s. The recrystallization of amorphized TiNi alloy was observed in the course of during tensile deformation at T 400- 600 C. As a result, nanocrystalline (NC) and ultrafinegrain (UFG) structure have been formed, parameters of which are determined by temperature and strain rate. As one would expect, enhancement of deformation temperature leads to decrease of material strength and ductility increase. The NC TiNi alloys have not displayed superplastic behavior at all used temperatures and strain rates. Probably this is connected with the grain growth dueirng the process of testing at elevated temperatures. However, another interesting effect has been found. The TiNi samples' fractures have been investigated with the help of scanning electron microscopy. The HPT amorphized TiNi alloys under tension at room temperature fail with formation of brittle fracture (Fig.1a). In case of tension at the temperature of 400 C the fracture becomes ductile. When the HPT TiNi samples are tested at the temperature of 450 ^{0}C, 500 ^{0}C there is a failure with formation of blade fracture, i.e. the fracture surface is transformed into a thin line (Fig.1b). An unusual relief is formed on the sample's surface after tension. After similar static annealing the samples microhardness (Hv) is higher than after elongation at the temperature of 500 ^{0}C because during deformation in the material the dynamical recrystallization is more intensive than the statistic one.



a)



b)

Fig. 1. HPT TiNi sample fracture after tension a) at room temperature b) at the temperature of 450° C. SEM

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Oral report

The influence of material purity and deformation temperature on the microstructure and diffusion in UFG Nickel produced by ECAP

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Ultra-fine grained (UFG) materials produced by severe plastic deformation (SPD) have become an important target of research due to their improved properties and the promise they hold with respect to novel applications. In this relation grain boundaries and (grain boundary) diffusion controlled processes are thought to play an important role.

It has been suggested that so-called non-equilibrium grain boundaries in SPD-processed materials are largely responsible for their unusual properties. These non-equilibrium grain boundaries contain a high density of extrinsic (excess) dislocations and an increased excess free volume. The existence and the properties of non-equilibrium grain boundaries can be studied in great detail by grain boundary diffusion measurements because such non-equilibrium grain boundaries represent ultra-fast diffusion paths in comparison to conventional, relaxed high-angle grain boundaries in coarse grained materials [1].

The existence and properties of these non-equilibrium grain boundaries are investigated in UFG nickel produced by Equal Channel Angular Pressing (ECAP).

The topic of this talk is the influence of materials purity and deformation temperature on the structural, mechanical and diffusion properties of UFG Ni.

Nickel materials with a nominal purity of 99.6 and 99.99wt% were deformed at room temperature, 200°C and 400°C via the B_c4 route.

A systematic pattern is discovered between the variations of the rate of grain boundary self-diffusion, specific contrast at typical grain boundaries as revealed by TEM, HRTEM and microhardness (see Fig.1).

This correlation is examined and discussed in terms of the non-equilibrium states of the grain boundaries in SPD-processed nickel.

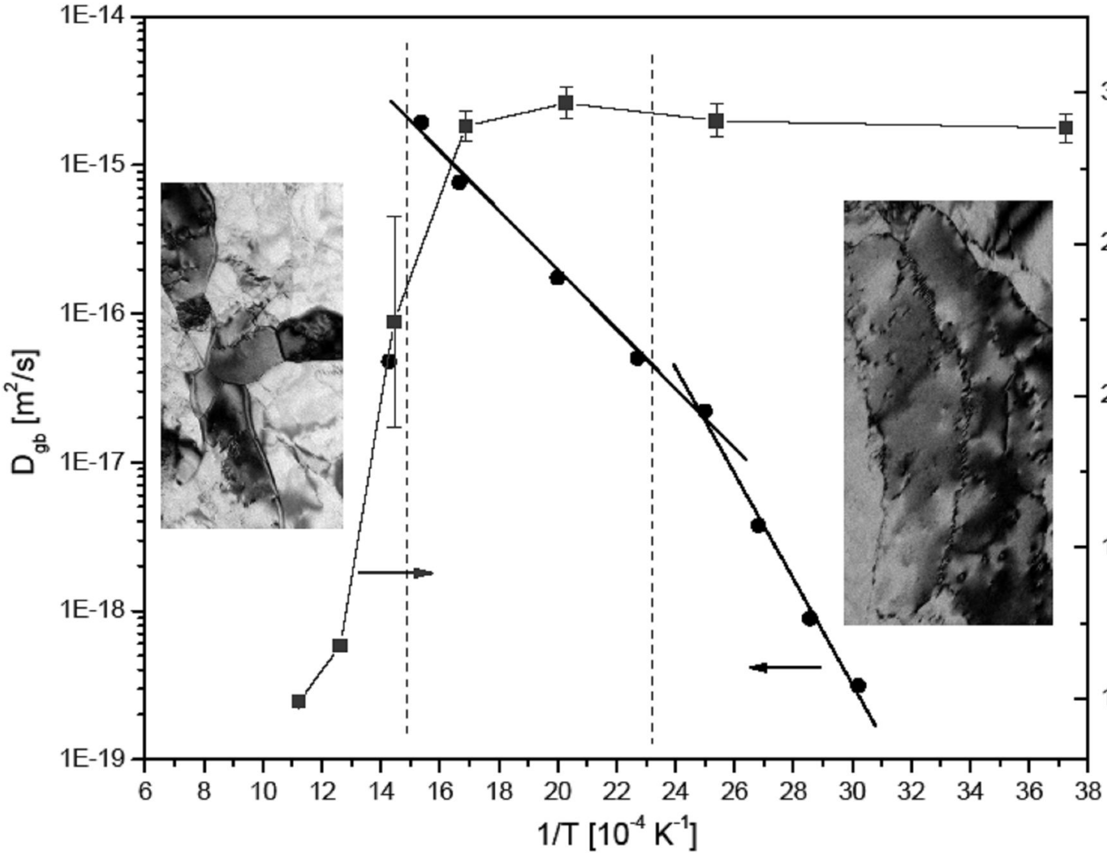

Fig. 1: Correlation between microhardness (HV, right ordinate), microstructure (TEM micrographs) and the rates of self-diffusion (D_{gb}, left ordinate) for UFG Ni of 99.6wt% purity deformed at room temperature

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25 August

BNM-2011 session

Computer simulation. Texture analysis and X-ray studies

August 25, 2011

Invited report

Nanodipoles of partial disclinations as carriers of noncrystallographic shear and crystal lattice reorientation in nanocrystalline metals and alloys

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In Ni alloys and bcc alloys based on V, Ta, and Mo–Re subject to intense plastic deformation on Bridgman anvils, high-defect structure states of the dipole and multipolar types with nanocrystals of characteristic size less than 10 nm, small-angle misorientation boundaries, and unusually high (hundreds of degrees per micrometer) values of the lattice elastic curvature and of the local gradients of internal stresses have been detected inside nanograins 50–100 nm in size. It is supposed that important factors in the formation of these states are, first, the dipole or multipolar character of the nanostructured states, which is responsible for a significant reduction of the internal stresses due to overlapping of the stress fields of opposite signs, and, second, the scale factor: the small size of the high curvature regions, which determines the low values of the internal stresses arising in these regions.

At the initial stages of formation of the above states, several nanometers wide misorientation bands bordered by partial disclination dipoles or by noncrystallographic shear dislocations with effective Burgers vectors several times smaller than the Burgers vectors of the lattice dislocations have been revealed [1].

The suggestion has been made that these nanobands and the mentioned dipole and multipolar substructures form on the nanoscale level due to the motion of partial disclination nanodipoles or noncrystallographic shear dislocations that is controlled by directional flows of nonequilibrium (arising under plastic deformation) point defects in the fields of nanodipole-generated high local gradients of the stress tensor normal components. The principal physical factors governing this mechanism and the conditions for its realization are analyzed.

For the true logarithmic strain $e > 6$, mesoscopic strain bands have been revealed which consist of packs of nanobands with dipole-type misorientations, whose width varies from a few nanometers to several tens of nanometers, which are spread in noncrystallographic directions, forming pronounced rotational structures and numerous nanopores. The supposition has been made that these features are related to the development of collective effects in the disclination substructure that give rise to a group motion of nanodipoles and quadrupoles of partial disclinations.

Realization of the above-described mechanisms should provide additional possibilities for nanostructuring the defect substructure of metallic materials by plastic deformation to produce structural states with nanocrystals several nanometers in size and high local gradients of the lattice orientation.

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Oral report

Influence of texture evolution on nanocrystalline Ni-Fe alloys plasticity

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Electrodeposited nanocrystalline Ni and Ni-Fe alloys containing 5.5, 18.5, 28.5 and 43 wt. % Fe were characterized using a variety of techniques and their deformation behaviour correlated with microstructural evolution especially in regards to texture. It was found that Ni-5.5%Fe had a strong (200) texture while the other compositions had a mixed (111) (200) fibre texture. The lattice parameter of the Ni-Fe composition increased linearly with %Fe, nanocrystalline grain size reaches a maximum for the Ni-18.5%Fe composition and microstrain showed a minimum value for the Ni-18.5%Fe composition. Line scans of the elemental composition using EPMA across the as-deposited samples showed very good uniformity while Fe elemental mapping showed a banding phenomenon. Load-displacement (load-unload) curves were generated by nano-indentation experiments at 3 different loading rates of 1, 10 and 20 mN/sec which gave hardness and elastic modulus of the specimens for all the alloys as a function of %Fe. It was found that the material hardens with increasing loading rate or strain rate. The values of strain rate sensitivity obtained for the Ni-Fe alloys are much less than 0.5. This assumes that dislocation driven mechanism is still operating in the nanocrystalline materials, but the exact role of accompanying factors is not clear. In this connection, possible contribution of disclination defects to the high strain rate deformation behavior of nanocrystalline materials is discussed. It was found that the strain rate sensitivity reaches a minimum and the activation volume maxima for the Ni-18.5%Fe composition which is very close to the composition of Permalloy.

Oral report

The role of grain boundaries in BNM properties' formation

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Grain boundaries in bulk nanostructured metallic materials, produced by the severe plastic deformation (SPD) methods, possess a number of peculiarities (a greater share of grain-boundary phase, mainly a high-angle range of misorientations, high density of the extrinsic dislocations, etc.) [1]. As a result, quite unusual and attractive properties are typical of these materials, both from scientific and application-oriented points of view [1 - 4].

Computer modeling allows making quantitative estimations of the boundary role in development of novel properties and specific behavior of the bulk nanostructured materials, produced by the SPD methods [5, 6].

This report presents the results of the recent research, made by the computer modeling method, in respect of the role of non-equilibrium grain boundaries occupying large area and being sources and sinks of dislocations, atoms and vacancies, stimulating active grain boundary diffusion processes and being responsible for the novel properties of bulk nanostructured materials, including high strength and ductility at quasistatic and dynamic mechanical tests, a reduced temperature of ductile-brittle transition, deviations from the Hall-Petch law, advanced radiation stability.

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Oral report

In-situ X-ray line profile analysis of the tensile deformation of nanocrystalline palladium films

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Materials with grain sizes in the low nanometer range are known to show mechanisms of plastic deformation which are different from those in the ultrafine/coarse grained range. X-ray line profile analysis using synchrotron radiation done in-situ during plastic deformation allows to quantitatively observe the evolution of several microstructural parameters such as the size distribution of the Coherently-Scattering-Domains (CSD), the density and arrangement of dislocations, and the frequency of planar defects. In order to investigate the changes of these microstructural parameters in a pure metal with a grain size around 30nm and below during plastic deformation, samples of nanocrystalline palladium with a thickness of about 1 µm being sputtered on a dumbbell-shaped Kapton foil were subjected to tensile deformation. High resolution X-ray profiles were taken at beamlines of the synchrotrons ANKA, KIT Karlsruhe (Germany), and SLS, Villigen (Switzerland). The samples were deformed in a first cycle to $\varepsilon\sim0.2$ and unloaded, immediately followed by a second loading to $\varepsilon\sim0.4$, and unloading. Several hundred X-ray profiles were recorded during each cycle and subsequently evaluated using the CMWP-fit program [2]. In order to correct any effect of the Kapton foil, the latter without any Pd-layer was measured using the same scheme, and the resulting profiles were incorporated in the evaluation procedure. In this presentation, emphasis is given on the detailed analysis of the X-ray peak profiles. Due to the high number of profiles to be evaluated, and to increase reliability of the evaluated data, a software package was written [3] allowing for automatic evaluation of the profiles with varying fitting parameter sets. The software package also facilitates the analysis of the results obtained from fitting. This made it possible to reliably evaluate the measured data using all the physical models implemented in the CMWP-fit software: The evolution of the median m and variance σ of an assumed log-normal CSD-distribution (also considering grain ellipticity), the density of dislocations ρ and their arrangement M, and the frequency of planar defects β was determined. The scatter of the evaluation results is remarkably low, showing accurate measurements and proper evaluation of the measured data.

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Oral report

FEM modelling of continuous extrusion of high-strength metals using commercial conform(TM) machine

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Continuous extrusion of metals in Conform™-type equipment is a well-known process which has been used in industry for several decades as a technique for extrusion of soft metals (Al, Cu) and their alloys. Traditionally, it has been employed only to achieve the required shape of products. New developments in continuous extrusion of metals include its use for high-strength materials[1] and a utilization of its beneficial side effect: the microstructure refinement. In this case, the extruded material has the same shape as the input stock, making it available for subsequent processing without reconfiguring the machine. Multiple repetitions of these steps lead to refinement of original grains down to the size between tens and hundreds of nanometres (80 – 300 nm). In this fashion, one can obtain nanostructured high-strength materials with unique properties.

Fig.1. Conform™ 315i

The company COMTES FHT a.s. has been systematically engaged in development in this field. At the end of 2010, the company purchased Conform™ 315i[2] machine, which is the standard choice for manufacturing products with required dimensions from rolled or cast stock from copper and aluminium alloys. Long-standing experience with research into the ECAP[3] process and other technologies for preparing ultrafine structured materials inspired modifications to the design and material of the forming die. These changes will allow repeated processing of high-strength alloys with strength above 1,000 MPa.

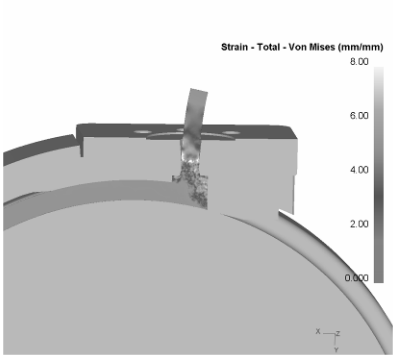

Fig.2. Result of FEM simulation: strain upon first pass

This paper describes modelling of the continuous extrusion process using the finite element method. The purpose of the simulations is to find optimum geometry of the extrusion die and process parameters to achieve large uniform strains and a high-quality surface of the formed product. The simulation of the extrusion process in DEFORM software indicates that the required microstructure refinement can be achieved by running 8 – 10 passes.

The authors are working on optimizing the continuous extrusion process (velocity and temperature) for high-strength materials and optimizing the FEM model to speed up its development.

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Oral report

Analytical modelling of flow stress of metals and alloys processed by severe plastic deformation

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Flow stress increasing by grain refinement in polycrystals is well known fact established by so-called Hall-Petch relationship. One of the most interesting evidence of deviations from this relation revealed by aluminium alloy 1570 is analyzed by means of analytical modeling in the present work. The alloy 1570 (Al–5.7Mg–0.32Sc–0.4Mn, wt.%) processed by High-Pressure Torsion (HPT) at room temperature has showed flow stress much higher than one predicted by the Hall-Petch relationship [1]. Equation variations of developed model allow to investigate other metals and alloys subjected to various techniques of severe plastic deformation (SPD) as well. The model is based on Taylor's relationship between flow stress and dislocation density [2]. Dislocation motion is assumed to be main deformation mechanism in all range of grain sizes [3]. Calculations of the dislocation density evolution have taken into account processes leading to dislocation accumulation and „dynamic recovery" processes. Thus following mechanisms and phenomena are considered: *grain boundaries acting as dislocation barriers limiting the free mean path of the dislocations; dislocation multiplication by double cross slip; annihilation of dislocations by cross slip; absorption of dislocations by grain boundaries and its recovery*. Influence of these factors on the dislocation density in case of aluminium alloys is being estimated in view of the fact that high stacking fault energy is typical for aluminium (it is playing important role in possibility of cross slip) and that content of alloying elements decrease this value [4]. As a result, analytical strain, strain rate, temperature and grain size dependence of dislocation density has been obtained. In addition to strain hardening various hardening mechanisms are being taken into account. For instance, for aluminium alloys processed by SPD they are presented by solid solution strengthening, precipitate hardening and lattice strength. For the alloy 1570 it is concluded that increased solid solution strengthening is playing leading role in high strength because of a high amount of Mg along grain boundaries produced by HPT. Contribution of strain hardening is high as well due to high value of dislocation density after HPT. Deviations from Hall-Petch relation revealed by aluminium alloys processed by SPD are caused by specific strengthening mechanisms, while this relationship has been established to coarse-grained materials which strength is mainly determined by strain hardening due to limitations of the free mean path of the dislocations by grain boundaries.

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August 25, 2011

Oral report

Rate-Strain-Microstructure (RSM) maps for severe plastic deformation (SPD) processes

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Innovation probability for materials with high specific strength is very high [1]. These materials have applications ranging from aerospace, nuclear industry, biomedical equipments to sporting goods. This expectation has spurred much of the interest in Bulk Nanostructured Materials (BNM) that are composed of a nano-scale microstructure, which is characterized by high flow-strengths. Complementing the strength, recent discoveries also point to an array of functional properties in these materials, provided the characteristics of the interfaces encompassing the nanostructures are controlled.

A technologically viable route for creating BNM is using Severe Plastic Deformation (SPD) processes such as Equal Channel Angular Pressing (ECAP), which offer fully-dense materials of scalable cross-sections. In SPD the progression of microstructure refinement as a function of SPD strains is relatively well understood and is exploited in the manufacture of BNM. However, access to a wider-array of useful microstructural characteristics becomes possible by utilizing higher strain-rates and coupled temperatures in addition to the large strains. For example, Dynamic Plastic Deformation in compression has been used at high strain-rates as a tool for controlling the dispersion of nanotwins amongst sub-grain structures [2]. High-Rate Severe Plastic Deformation (HRSPD) involves shear strains >2 and strain-rates $>10^3$/s has been similarly used to generate nanotwinned materials and to control the modality of grain-size distributions, grain-boundary structure and the resulting thermomechanical properties [3-4].

However, under complex combinations of strain, strain-rate and the coupled temperatures, the evolution of microstructures is much more poorly delineated and this is often a barrier preventing their reliable utilization for endowing desired microstructural properties. Here, we utilize Large Strain Machining (LSM) as a "microstructure response test" to elucidate the behaviour of materials across a swathe of deformation strain, strain-rates and temperatures. The idea is that, since LSM involves deformation ahead of a cutting edge and not inside a deformation die, this allows for a direct observation and measurement of the thermomechanics of the process without being occluded. This enables a direct visualization of the plane-strain deformation zone using high speed and Infrared cameras to measure *in situ* the deformation strains, strain-rates and temperatures. When we tried to understand the range of microstructures that emerged from LSM, we noted the absence of any regularized "phase-space" on which the various microstructural characteristics can be projected, one-to-one. Therefore, we hypothesize a prototypical Rate-Strain-Microstructure (RSM) space with the x-axis as the Zener-Hollomon parameter, an Arrhenius-type rate equation and the y-axis is the effective SPD strain. Here, we found that each point (or sub-space) on the RSM space maps to unique microstructural characteristics in the deformed specimen. Using our *in situ* characterization of the thermomechanics of SPD and the elucidation of the subsequent microstructure for a range of conditions, we construct empirically-derived RSM maps of microstructures. These include maps of grain-size, fractions of low-angle boundaries, dislocation densities, grain-boundary energies etc. If accurately constructed and generalizable, the utilization of such maps as a tool for microstructure engineering and control in an array of processes such as Large Strain Extrusion Machining, ECAP, Friction Stir Processing etc. are discussed.

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25 August

Special session

for young scientists and PhD students

Computer simulation. Texture analysis and X-ray studies

Y/Oral report

Dislocation density-based finite element analysis for plastic deformation behavior of high pressure torsion

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Recently, various methods of sever plastic deformation (SPD) have been developed to fabricate bulk nanocrystalline metallic materials [1]. Especially, high pressure torsion (HPT) has become an attractive process among various SPD processes, because it involves larger strain to impose the possibility of producing nano and ultrafine grained materials than the other SPD processes [2]. HPT processing consists of two stages based on the motion of the dies and the workpiece, as shown in Fig. 1: first the compression stage and second the compression-torsion stage. In this paper, the finite element method has been applied for the analysis of plastic deformation behavior of the workpieces during the HPT process. Numerical simulations deal with a phenomenological constitutive model based on the microstructure and dislocation density evolution [3]. The finite element analysis of the plastic deformation behavior of pure copper during the HPT process is conducted. The simulation results of stress distributions and the thickness of deformed workpiece are compared with experimental ones. In particular, couple interactions between geometrical and materials factors are investigated using a three dimensional visual scheme.

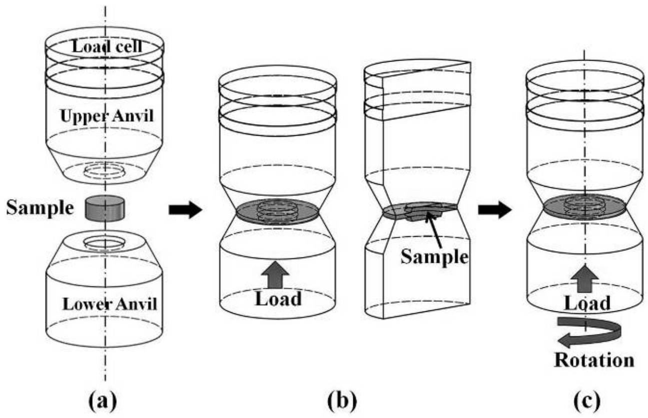

Fig. 1. Schematic of high pressure torsion; (a) set-up, (b) compression stage, and (c) torsion stage.

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Y/Oral report

Textural investigation of 8090 Al-Li alloy fabricated by ECAP

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Equal-channel angular pressing (ECAP) is one of the SPD processing techniques. ECAP is a process in which the ingot is subjected to a very severe plastic strain without any change in the cross-sectional dimensions. ECAP. Numerous reports have confirmed that ECAP is a promising technique for achieving grain refinement in bulk materials. It was demonstrated that the as-processed materials have unusual physical and mechanical properties. Al–Li alloy is a precipitation hardened alloy with shearable precipitates and regarded as a typical planar slip material [1-4]. The research work was conducted using 8090 Al-Li billet contains (in wt %) 2.2Li, 1.3Cu, 1.36Mg, 0.14Zr, 0.19Fe, 0.15Si. The ECAP processing was carried out using a die with an intersecting channel angle, Φ, of 120^o and an outer arc angle,ψ, of 20^o. Both Al-Li billet and die were preheated to 250°C before the start of the ECAP process. At the first the samples experience the A route with 2 passes and annealed at 520 ^oC for 2 hr. After annealing, the samples experience the A and Bc route.

Texture measurements were performed using X-ray diffraction by a Siemens D500 goniometer system (Mo tube) and the standard reflection technique. The ECAP textures were measured on the x-y-z laboratory system of die. Three incomplete pole figures({111}, {200}, and {220}) were measured on the cross-section (x–z plane) of the samples (Fig. 1). Specimens 5 mm thick were cut from both the initial sample and from the extruded bars after passes 1, 2, 3, 4, 6 and 10 with routes A and B_C. The orientation distribution functions (ODF) were calculated from the pole figure data using TextTools software. The textures are presented in the $0^o \leq \varphi1 \leq 360^\circ, \varphi, \varphi2 \leq 90^\circ$ Euler space. Figure 2 demonstrate {111} pole figure of the representative initial texture for annealed sample placed in the ECAP die entry channel. There is a weak texture for the initial sample that can be described as {110}<112> with maximum intensity 3×R (random).

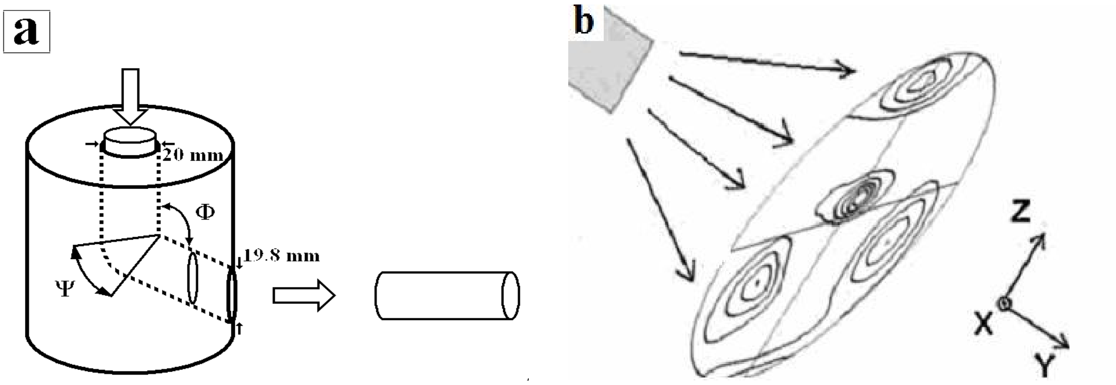

Fig 1. The cross section of the die and texture measurement.

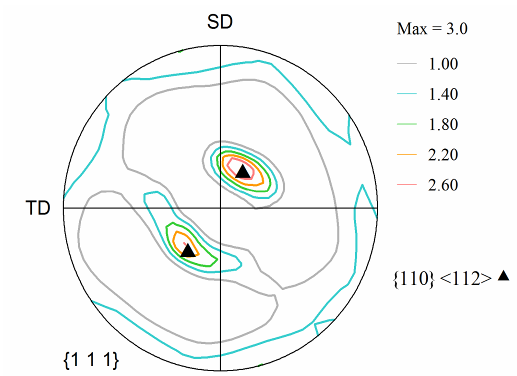

Fig 2. {111} pole figure of the representative initial texture for annealed sample.

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August 25, 2011

Y/Oral report

Integrable maps of a class of two-dimensional and three-dimensional discrete ϕ^4 models

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Discrete nonlinear equations play an increasingly important role in physics in the past few decades finding their applications in the description of properties of topological defects of crystals, in the studies of anharmonic localized modes in nonlinear lattices, mechanisms of DNA denaturation, to name a few. Lately, the attention of researchers was shifted to investigation of lattices of higher dimensions because they often are more realistic. Nonlinear lattice equations are typically non-integrable. A wider class of exceptional discrete models constitute the ones that admit exact moving solutions for selected propagation velocities. At some values of model parameters this specific velocity can vanish and, in this case, the equilibrium static solutions with arbitrary shift along the lattice exist, meaning that such solutions do not experience the Peierls-Nabarro potential. So far such exceptional discrete models were constructed and studied for one-dimensional problems [1]. It was found that the exceptional discrete models have better transport properties since the solitary waves, capable to carry energy, mass, electric charge, etc., are not trapped by the lattice in such models, they can be accelerated by weak external forces, and they interact with each other more elastically.

In the present study we demonstrate that exceptional discrete Klein-Gordon lattices that admit equilibrium static solutions with arbitrary shift along the lattice can also be constructed for two-dimensional and three-dimensional problems, thus extending our work, where similar one-dimensional model was treated [1].

The considered discrete ϕ^4 model in two-dimensional case has the form

$$\ddot{\phi}_{n,m} = \frac{1}{h^2}\left[c_n\left(\phi_{n+1,m}+\phi_{n-1,m}\right)+c_m\left(\phi_{n,m+1}+\phi_{n,m-1}\right)-2\left(c_n+c_m\right)\phi_{n,m+1}\right]+\lambda\phi_{n,m}$$
$$-\frac{A_2}{2}\phi^2{}_{n,m}\left(\phi_{n+1,m}+\phi_{n-1,m}\right)-\frac{A'_2}{2}\phi^2{}_{n,m}\left(\phi_{n,m+1}+\phi_{n,m-1}\right)$$

,where $A_2 + A'_2 = \lambda, \lambda = \pm 1$, c_n, c_m, h some parameters.

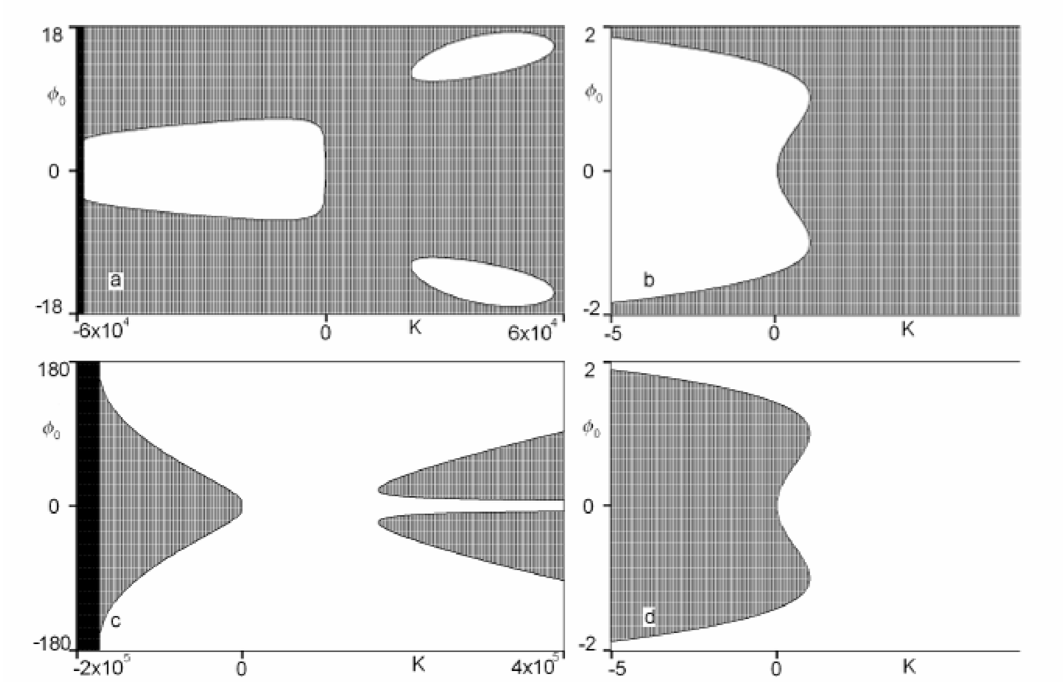

Fig.1. Examples of admissible initial values ϕ_0 as the functions of the integration constant K (shown in grey) for QRT-type map. Black and white regions present inadmissible values of ϕ_0

We found a Quispel-Roberts-Thompson (QRT)-type map [2] that allows one to construct exacts static solutions iteratively starting from an admissible initial value ϕ_0. Sets of admissible initial values were plotted for typical sets of model parameters as the functions of the integration constant K that enters the map (see Fig.1). Numerically we found that the kink solutions to the considered two-dimensional model are generically stable.

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X-Ray analysis of ω – phase of Ti, subjected to high pressure torsion

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The recent investigations have shown that ω-phase can be developed in α-Ti at high pressures and great deformation rates, which affects its strength and ductility. In connection with this, it is important to identify the quantitative regularities of the development of ω-phase during the SPD.
This report presents the research results of a microstructure, a crystallographic texture and deformation mechanisms, which are typical of the ω-phase of Ti, subjected to the HPT with pressure equal to 6 GPa and temperature 298 K. The research has been made with the disk-shaped ingots with the radius 20mm in states after 0.5, 1 and 5 HPT rotations. The research has been made with the help of the X-ray analysis and computer modeling methods. In the result, the regularities of microstructure evolution parameters, such as lattice parameters, the volume fraction of phase, dislocation density, the size of the coherent-scattering regions, the character of preferred orientations, and also the activity of one or another slip and twinning systems in the ω-phase, depending on the SPD degree. The obtained results allow explaining and forecasting deformation behavior of a nanostructured Ti, considering its parameters of microstructure and crystallographic texture.

Y/Oral report

DSC investigations on lattice defects in hydrogenated Pd deformed by high pressure torsion at different temperatures

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Recent investigations on palladium hydride (Pd-$H_{0.78}$, [1]) showed, for the first time, evidence of formation of vacancy-hydrogen (Vac-H) clusters during Severe Plastic Deformation (SPD) by means of High Pressure Torsion (HPT). Vacancy concentrations produced in Pd-H by this method are extraordinarily high, that is of the order of several 10^{-3}.

HPT deformation was performed on disk-shaped Pd (99.95%) samples with a diameter of 6mm and thickness of 0.8mm loaded with hydrogen in a Sieverts-type apparatus up to a hydrogen content of [H]/[Pd]~0.78 and stored in liquid nitrogen. HPT-deformation was carried out at a hydrostatic pressure of 8GPa at temperatures 77K, 195K, 303 K and 363 K.

The desorption of hydrogen was monitored by microhardness and density measurements in order to investigate the influence of hydrogen on mechanical properties. The DSC measurements revealed the vacancies to be thermally stable till about 483 K (by about 110 K higher than in case of undeformed Pd) due to trapping of hydrogen, thereby forming vacancy-hydrogen clusters. However, it turned out that apparently thermal activation is required for the formation of Vac-H clusters during HPT. Results furthermore indicate that hydrogen-assisted thermal stabilization not only occurs with vacancy-type defects but also with dislocations and grain boundaries, an effect which may be of some importance for the stabilization of nanocrystalline materials in general.

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Y/Oral report

On the absence of the role of discrete breathers in thermally activated nucleation of point defects in a 2D crystal

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Intrinsic localized modes or discrete breathers are the spatially localized vibrational modes of large amplitude in nonlinear defect-free lattices whose existence was proved theoretically [1] and experimentally [2]. The role of DBs has being extensively discussed during the past two decades in relation to many physical systems, including solid state physics, nonlinear optics [3], chains of superconducting Josephson junctions [4], DNA models [5], Bose-Einstein condensate [6], materials science [7]. There are several works about DBs at thermal equilibrium [8] and some speculations on possible impact of DBs in formation of point defects. In this study, by means of molecular dynamics simulations, we measure the lifetime of gap DBs in two-dimensional perfect crystal with stoichiometry A_3B at thermal equilibrium. As it was previously demonstrated [9-11], existence of DBs in this crystal is possible for sufficiently large atomic weight ratio M_A/M_B when the gap in the spectrum is sufficiently wide. For comparison, crystal with relatively small M_A/M_B ratio was considered with no gap in the spectrum and thus, supporting no gap DBs. It was found that in the crystal supporting DBs, in contrast to the opposite case, there exist long-lived localized vibrational modes of high amplitude, whose concentration and lifetime grow with temperature. Then we have analyzed various mechanisms of Frenkel pairs' formation to understand the possible role of gap DBs in this process. We have measured the time needed for the appearance of a Frenkel pair in the initially defect-free computational cell of 32x32x4 atoms at various temperatures for the two crystals, with small and large atomic weight ratios. It was demonstrated that, in both crystals, the waiting time for defect nucleation decreases with temperature exponentially.

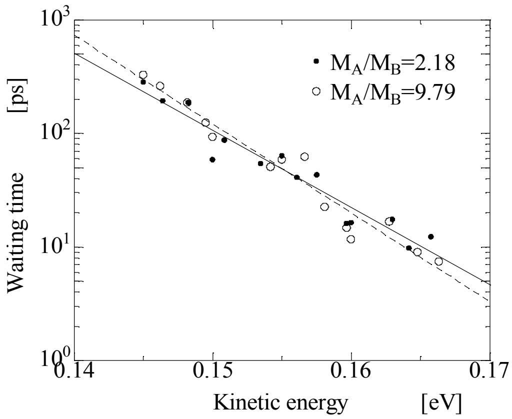

Fig.1. Waiting time of Frenkel pair nucleation vs. temperature for two crystals. Fitting lines are shown by solid (dashed) lines for crystals with small (large) atomic weight ratios.

There was no difference indicated for the waiting time of Frenkel pair nucleation in the crystals supporting and not supporting DBs. We have shown that the mechanism of thermally activated Frenkel pair formation is the cooperative motion of atoms. The cooperative factor of atomic displacements was measured and it was found to be approximately same in the crystals supporting and not supporting DBs.

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26 August

BNM-2011 session

Microstructure evolution and nanostructuring

August 26, 2011

Invited report

Electroplastic effect in nanostructured titanium alloys

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Ti-based alloys possess a remarkable complex of mechanical and functional properties, especially high in nanostructured (NS) state [1]. To obtain NS states the severe plastic deformation technique is used, which realization for long-size products of thin section is the problem from susceptibility to strong strengthening and the limited deformability of Ti-based alloys. The electroplastic rolling (EPR) at room temperature can be considered as alternative technique to formate NS state [2]. The paper considers deformation behavior of the Ti-based alloys, including pure Ti, Ti-64 and shape memory TiNi materials, both at EPR processing and tension with pulse current. It is shown that EPR increases significantly strain to failure and refines microstructure to grain size less 500 nm. Stress jumps up and down was observed on the stress-strain curves at tension with current. Different direction of stress jumps is conditioned by martensitic transformation and electroplastic effect, correspondingly. Both mechanisms are structurally sensitive ones to grain size.

The present work was supported by the Russian Education&Science Ministry, projects P340 and 14.740.11.0825.

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Invited report

The grain refinement, superelasticity and shape memory in TiNi based alloys

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The experimental results about the basic mechanisms of grain microstructure formation and about the inelastic properties (superelasticicity and shape memory effect, SME) in TiNi based alloy under warm multiple pass rolling via chill-pass rolls with square cross-sections of channels are presented. The individual rolling cycle includes the heating (3 min) to the deformation temperature, the one-pass rolling to achieve the reduction of sample area of 2-3% and the secondary pass rolling via the same channel. The series of samples were produced by the isothermal rolling at 723K and 623K with the true deformation, e , from 0,07 to 2,0 and by the warm rolling with successive drop of deformation temperature from 773K to 623K (the summary e about 2). The $Ti_{49.2}Ni_{50.8}$(at.%) alloy are characterized by the B2 structure at 295K and the average grain sizes, d, about 43 μm.

The formation of mesoband structures appeared on different deformation stages and regularities of microframentation due to the achievement of multiple slip of dislocations in volume of grains are analyzed. The nonmonotonous change of *‹d›* versus e was found under rolling at 723K. The sharp *‹d›* increase was observed on the initial rolling stage. The *‹d›* value decreases on the next deformation stages. On the whole, the e dependence of ‹d› is similar to the consolidation of grains under the postdeformation annealing of alloy samples with the critical value of preliminary deformation [1]. However the analysis of results obtained by the optical metallography and scanning electron microscopy confirms that the grain consolidation (GC) realizes “in-situ” under warm rolling of samples. The developed systems of slip lines and microbands of localized deformation appear in the volumes of new coarse grains under the subsequent rolling passes as it was observed in grains with initial sizes. It is shown that the finish alloy microstructure are appeared as the result of a competition of CR and dynamic recrystallization “in-situ” under deformation and metadynamic recrystallization (MDR) at cooling of deformed samples. The effect of GC and MDR make weaker with the e increase. But these factors impede the formation of the ultra-fine grained structures and cause the appearance of heterogeneous graininess during all deformation stages. In particular, nanograins,

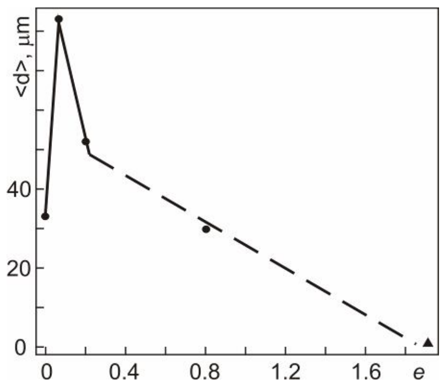

Fig. 1. The ‹d› dependence vs. deformation, e.

submicrocrystalline grains and microscale grains (1-2.5 μm) are found in deformed sample with e=2.

The changes of (i) superelasticity appeared during isothermal, (ii) SME observed during isothermal (295K) the subsequent heating of unloaded samples and (iii) accumulation of plastic deformation after the finish of shape recovery under heating, in depend on the maximum torsion deformation, γ_{max}, produced under the isothermal (295K) loading of samples, are studied. The most bright differences of inelastic effects for alloys with CG and UFG structures are observed under $\gamma_{max}<$ 12%.

The superelasticity dominates over SME for samples of alloy with CG structures, but SME predominates over superelasticity for samples with UFG structure. The predominance of SME with respect to superelasticity are observed under $\gamma_{max}>12\%$ independently of sample microstructures. In this case, the summary inelastic, deformation determined as the sum of SME and superelasticity, enlarges with γ_{max} increased and reaches for samples of alloy 19,7% under γ_{max} about 28-30% independently of sample microstructures. The factors caused differences of inelastic effect for alloys with different microstructures and high levels of summary inelastic deformations are considered.

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August 26, 2011

Invited report

Microstructure and microtexture evolution in pure metals after ultra-high straining

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Ultrafine-grained and even nanostructured materials can be manufactureed using ultra-high straining by ECAP, HPT, machining and their combinations, such as machining of ECAP specimens, HPT of ECAP and HPT of machining chips The report will present recent results of investigation the microstructure and microtexture of pure copper, nickel and aluminum subjected to different deformation processes to ultimately high imposed strain. Comparison of microstructure, dislocation density and microhardness developed during combination of different strain paths have been performed. All characteristics gave been analyzed by x-ray, transmission and scanning electron microscopy, OIM. Influence of different processing routes is discussed in terms of accumulated strain and microstructure refinement. Saturation in grain refinement has been discussed in terms of recovery taking place during ultra-high strain deformation.

August 26, 2011

Oral report

Formation of fine grain structure in high-softening-temperature-materials during friction-stir processing

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Friction-stir processing (FSP) can refine the grain structure and homogenize the heterogeneous microstructure effectively. It had been limited for low-melting-temperature materials in the 20th century, but recent development of the FSP tools enables us to apply friction stirring to high-softening-temperature-materials (HSTMs), such as steels and titanium alloys. In this presentation, recent status of FSP of HSTMs and some examples of the grain refinement in steels and titanium alloys by FSP will be briefly shown.

The tool material is critical in FSP of HSTMs. The tool should meet the significant requirements, i.e., it must maintain sufficient strength to constrain the material to be stirred at the softening temperatures, and also be resistant to fatigue, fracture, mechanical wear, and chemical reactions with both the atmosphere and the stirred material [1]. Recently, many tools made of polycrystalline cubic boron nitride (PCBN), W-Re, WC-Co and Co-based alloy have been developed, and then fundamental studies on FSP of HSTMs have been conducted. FSP of carbon steels, high-strength steels, stainless steels, and titanium alloys was successfully done. FSP results in the grain refinement in steels and titanium alloys without phase transformation during friction stirring. The grain size significantly depends on the FSP parameters, i.e., higher rotational speed and lower travel speed of the tool result in larger grain size, due to higher heat-input. Generally, grain size of the stir zone lies between 1 and 20 μm in steels and titanium alloys, but some steels exhibit very fine grain structure with smaller grain size than 1 μm after the low heat-input FSP. For example, electron backscatter diffraction (EBSD) image of the stir zone of 1.3wt%N containing steel is shown in Fig. 1 [2]. The equiaxed grain structure with average

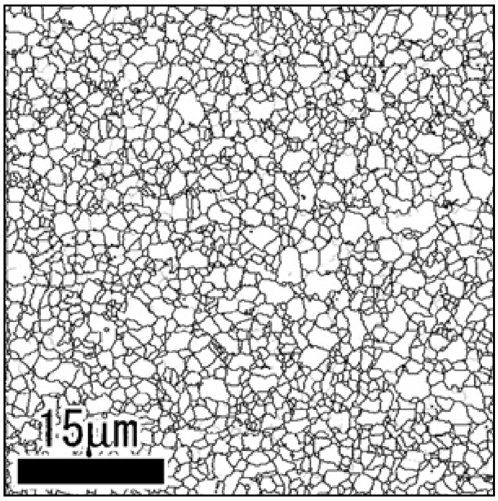

Fig. 1. EBSD map obtained from stir zone of 1.3%N containing steel

grain size of 0.9 μm is produced at the low heat-input parameters. In the materials consisting of duplex microstructure, such as Ti-6Al-4V alloy and duplex stainless steel, FSP can lead to the duplex microstructure with fine distribution of equiaxed phases [3, 4]. The low heat-input FSP can produce an equiaxed ($\alpha + \beta$) structure with average grain size of about 0.5 μm in the stir zone of Ti-6Al-4V alloy, which leads to higher mechanical properties than the base material. These results suggest that FSP is available as an effective method to enhance the mechanical properties of steels and titanium alloys.

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Oral report

Effect of temperature and strain path on the microstructure formation of commercial pure titanium

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Evolution of the microstructure of commercially pure titanium during large warm and cold deformation is known to be associated with twinning and subsequent formation of high angle deformation-induced boundaries. There are a number of works showing considerable microstructure refinement (down to submicro- or nanointerval) of titanium due to large or severe deformation carried out by equal-channel angular pressing (ECAP), high pressure torsion, accumulated roll bonding, multiaxial forging and some other. However in spite of an abundant experimental data, the mechanisms of deformation responsible for the formation of an ultrafine grained microstrustructure in titanium are still unclear.

Effect of temperature (in the interval -196…450°C) on the mechanisms of deformation and the kinetics of the ultrafine grained microstrustructure formation in commercial pure titanium under various deformation paths (sheet rolling, hydrostatic extrusion, ECAP, multiaxial forging) was studied in the present work.

Three distinct stages, each of which was associated with a different mechanism of microstructure formation were revealed in titanium during one-directional strain: (i) twinning, (ii) an increase in dislocation density and the formation of substructure, and (iii) the formation of deformation-induced high-angle boundaries.

A decrease in deformation temperature intensifies twinning and thereby increases the contribution of twinning into microstructure refinement. At 350…450°C the main mechanism of the microstructure refinement was found to be dynamic recovery and dynamic recrystallization. The average grain size after deformation at cryogenic temperature was ~ 40 nm; at higher temperature T=350…450°C the average grain size increased to 200…400 nm.

During one-directional strain both boundaries of twins and deformation-induced high angle boundaries, which arised at different stages of deformation, formed a lamellar type microstructure with lamellae aligned mainly along the metal flow direction. The same processes were observed during multiaxial deformation however changes in the strain path activated new slip/twinning systems. This resulted in the interactions (intersections) of twins and/or deformation induced boundaries thereby increasing both uniformity of plastic flow and homogeneity of the microstructure after large deformation.

The lamellar microstructure formed at moderate deformation can be transformed into globular one during further strain. Change in strain path (for example in case of ECAP or multiaxial forging) considerably decreased the strain required for attaining homogeneous globular microstructure.

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Oral report

Grain refinement in light metals by friction-stir processing

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There is currently significant commercial interest in the development of materials with ultra-fine grain structures for structural applications. Techniques for the production of the fine-grained alloys are thus of considerable scientific and commercial interest. Of particular importance in this context are cost-effective methods that can be used to produce large quantities of such materials. Friction stir processing (FSP) would be one of the possible candidates for achieving this goal.

FSP has been developed by Mishra et al. [1] and this method is based on the fundamental principles of friction stir welding (FSW) technology invented in 1991 [2]. Basically, FSP technique involves plunging a specially designed tool rotating at high speeds into a sheet workpiece and then translating the tool in a direction of interest. The rotating tool produces frictional heat which softens the material so it can be readily extruded around the tool. The material that flows around the tool undergoes extreme levels of plastic deformation and thermal exposure and this normally leads to a significant grain refinement in the centre of processed zone (so-called “stir zone”).

In the present study, we consider refining efficiency of FSP in the case of some light metals. To this end, FSP was applied to pure aluminium, and ZK60 magnesium alloy.

FSP was performed on an FSW machine fitted with automated control system at various combinations of tool travel and tool rotation speeds. Following FSP, samples were sectioned perpendicular to tool travel direction and studied mainly by electron back-scatter diffraction (EBSD) technique.

In pure aluminum, base material (BM) consisted of coarse pancake-shaped grains, ~100 μm long and ~ 10 μm thick, containing dense substructure (fraction of high-angle boundaries did not exceed 25%). Typical microstructure observed in the stir zone (SZ) was dominated by low-aspect ratio grains tending to align in a common direction. Average grain-boundary spacing dramatically reduces dawn to ~ 1 μm. Bearing in mind that the pure aluminum is commonly considered to be a very difficult material for grain refinement, this result demonstrates exceptional refining efficiency of FSP. Average fraction of high-angle boundaries achieves ~ 77% thus evidencing that the obtained material should have high thermal stability. Material in the SZ has a weak (~2.5 x random)

"A" simple shear texture component thus indicating that predominant deformation mode during FSP was a simple shear.

Base material in ZK60 magnesium alloy had a partially recrystallized grain structure with grain sizes ranging from ~10 μm to ~1 mm. After FSP, the developed microstructure was dominated by fine (~5 μm) equiaxed grains containing high fraction of high-angle boundaries (~65%). The SZ had a strong {0002}//shear plane fiber texture thus indicating that the predominant deformation mode was also close to the simple shear.

In conclusion, FSP was shown to be an effective method for grain refinement in light metals. Single FSP pass may dramatically refine microstructure dawn to ultra-fine grain range. Simplicity and high productivity make FSP very attractive for microstructural refinement in sheet materials.

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Oral report

Mechanisms of structure fragmentation in certain metals under pulsed pressure and shear

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The dynamic channel-angular pressing method was reported to be used for producing the bulk nanostructural materials (copper, titanium, and aluminum alloys) [1]. In this work, transmission electron microscopy and X-ray diffraction analysis were used to study how structure fragmentation mechanisms for these metals depend on the initial conditions, i.e. channels geometry, sample velocity, number of passages through the channels, and the initial temperature. The results of this work are provided.

In copper, during dynamic channel-angular pressing, the already formed thin fibro-band structure undergoes fragmentation induced by transversal small-angle boundaries, then the rotation modes of deformation begin to work and cause certain portions to rotate relative to the others and, thus, large-angle boundaries arise. The structure gets fragmented down to the sizes of several dozens of nanometers against the active processes of strain hardening and continuous dynamic micro recrystallization. High strain-rate deformation increases hardness of copper 2.2 times while its strength goes up 1.4 times. Electrical resistivity measured at the liquid helium temperature of 4.2 K was found to be 5 times higher than that of copper in the completely annealed coarse-grained state. This effect is conditioned by the increased concentration of defects and by the size of sub-grains.

In titanium (VT1-0) deformed at 500^0C, stress relaxation under shear conditions is the basic mechanism of the structure fragmentation process. The localized deformation bands are formed throughout the sample and along grains boundaries. Bands are traces of the localized shear deformation, in which temperature buildup was followed by recrystallization. In the deformed portions fringing the large-size grains, the recrystallized grains occur both due to rotation, and local temperature increase. Structure disintegration proceeds with the further deformation of samples and, herewith, strength characteristics improve. Provided are the results on deformation of titanium encased in a copper sheath. In the sample having this structure, diversified twinning structures with high package density are noted to occur.

For aluminum alloys, mechanisms of ultrafine-crystalline state formation are found to depend on the composition, number of sample passages through the channels, and sample velocity. Results of analyzing the high-velocity deformation of samples of Al-3003 and Al-7075 aluminum alloys in crossing channels are provided. The role of both rotation instability of the flow and dynamical recrystallization in the process of materials structure fragmentation is noted.

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Oral report

Ductile to brittle transition of ultrafine-grained iron

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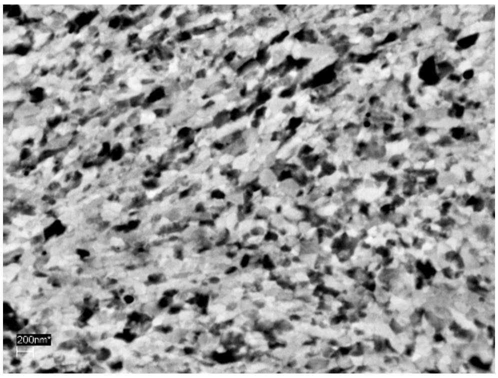
Body centred cubic (BCC) metals are known to show a strong temperature dependence of their mechanical properties, e.g. toughness properties, where grain refinement usually leads to an improvement at lower temperatures. With high pressure torsion (HPT) – a technique belonging to the family of severe plastic deformation – it is possible to obtain bulk materials with a very fine microstructure far below the micron range. This advantage can be used to study the fracture behavior of ultrafine-grained (UFG) bulk materials. In this work, the fracture toughness of iron, i.e. ferrite as the main constituent of most steels, in the UFG condition was determined as a function of temperature. In addition different heat treatments following the HPT-deformation were done to produce microstructures with a mean grain size in the range between some hundred nanometers and some microns, as shown in Fig.1, in order to incorporate the grain size dependence. During the shear deformation of the HPT process a characteristic deformation structure arises which makes the mechanical properties anisotropic. This effect was also taken into account by testing different crack propagation directions of the deformed as well as the deformed and heat treated samples with respect to the direction of deformation. It could be shown that especially for the deformed samples the fracture toughness is strongly anisotropic whereas for the additionally heat treated samples this anisotropy is less pronounced. After testing the fracture surfaces were studied in an SEM to get more information about the fracture processes and the mode of fracture, i.e. transcrystalline and intercrystalline fracture respectively. In summary it was found that there is a change from intercrystalline fracture for the samples with very small grain size to cleavage for samples with larger grain sizes, as shown in Fig.2.



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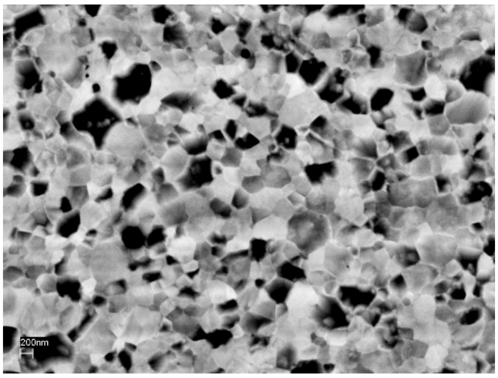

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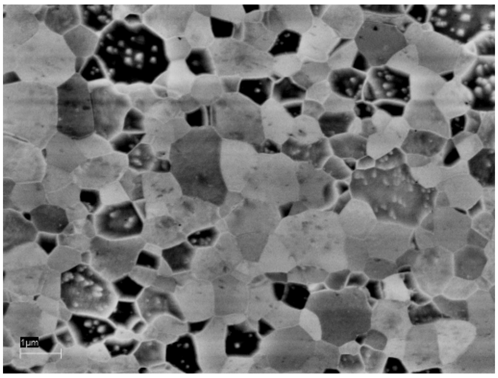

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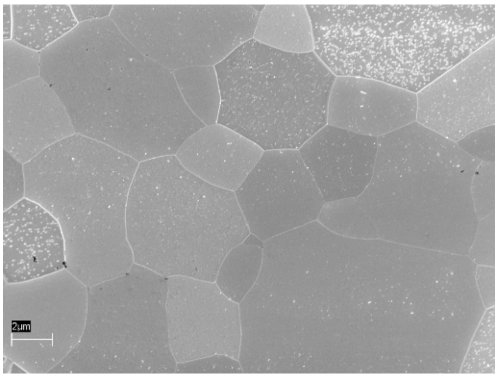
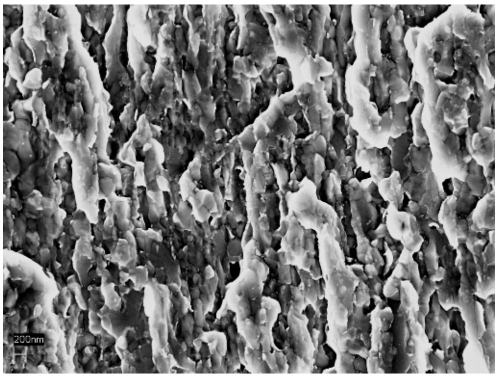
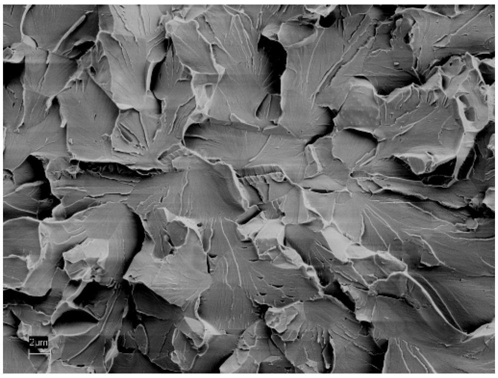


(d)

Fig.1. SEM micrographs of: (a) SPD-deformed iron; (b) to (d) different heat treatments following the SPD-deformation (please not the difference in magnification)



(a)



(b)

Fig.2. SEM fractographs of: (a) Intercrystalline fracture surface of SPD-deformed iron tested ad -196°C ; (b) Cleavage surface of SPD-deformed and heat treated iron with a mean grain size of 5µm tested at -196°C (please note the difference in magnification)

26 August

Special session for young scientists and PhD students

Microstructure evolution and nanostructuring

Y/Oral report

Effect of post ECAP aging on the thermal stability and subgrain structure of 2024 Al-alloy

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Equal channel angular pressing (ECAP) has been one of the most advantageous methods to obtain superior mechanical properties via grain refinement with minimum variation in the dimensions among the severe plastic deformation methods [1]. Researches have shown that there is significant improvement in the mechanical properties of Al alloy after ECAP [1,2] but the thermal stability of these properties and hence the fine grain structure has been an important issue considering the different conditions of use of these alloys. On the other hand post ECAP aging of these alloys shows significant difference in the age hardening kinetics. Under the considerations of these two important aspects, effect of aging on the microstructural stability of ECAPed aluminium comes to be an important issue that should be studied.

The aim of this study is to investigate the thermal stability of microstructure and mechanical properties in one pass ECAPed and post ECAP aged Al 2024 alloy samples. Al 2024 alloys of 2.5 cm diameter were one pass ECAPed with 120° die and some of them were aged in oil bath of 190°C. Both ECAPed and post ECAP aged samples were subjected to different temperatures. Samples were sectioned in all three dimensions to observe isotropic behaviour or texture formation. The differences were examined through X-Ray diffractogrammes, microhardness profiles, HREM and STEM.

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Characterization of severe plastically deformed CuCr alloy in the deformed and annealed state

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Interest in severe plastic deformation (SPD) has recently increased as these methods are serious candidates for producing ultrafine grained or even nanocrystalline bulk materials. SPD materials are of particular interest due to their interesting mechanical and physical properties. One major drawback of these ultrafine or nanocrystalline materials is the low stability of the microstructures. Different options, for example the addition of stable precipitates, a special powder compaction technique or stable second phases are used to generate more stable microstructures by SPD [1].

In this study, a commercial CuCr alloy containing 57wt% Cu and 43wt% Cr was investigated. Cu and Cr can be considered as immiscible [2]. Discs with a diameter of 8 mm and a height of 0.8 mm were SPD deformed using a High Pressure Torsion (HPT) tool. The samples were deformed to different strain levels (1, 5, 25 and 100 rotations) to investigate the influence of the applied strain on the final microstructure. All deformations were performed at room temperature with an applied pressure of 6.25 GPa and a constant rotation speed of 0.2 rounds/minute. The microstructure of the deformed samples was investigated by Vickers microhardness tests using a load of 500 g. In Fig. 1, the hardness of all deformed CuCr samples measured on the surface of the discs with a distance between the indents of 250 μm is plotted. In the beginning, the hardness increases with increasing applied strain (i.e. increasing radius.). Once the so called saturation microstructure is reached, even further deformation does not change the microstructure anymore. This is reflected in constant hardness values at a certain distance from the center of the samples. For example, the sample deformed for 100 rotations reaches this saturation region at a radius of 2 mm. The average grain size after 25 rotations at a radius of 3 mm is in the range of 10 – 20nm [3]. To investigate the thermal stability, the sample deformed for 25 rotations was annealed at 400°C for 30 minutes. Surprisingly, the annealed samples reached higher hardness values than all other HPT-deformed samples without an annealing treatment (see Fig. 1) even if they were deformed to higher strains. To find an explanation for this unusual behavior, synchrotron measurements over the disc diagonal with a distance between the individual measurements of 250 μm were carried out.

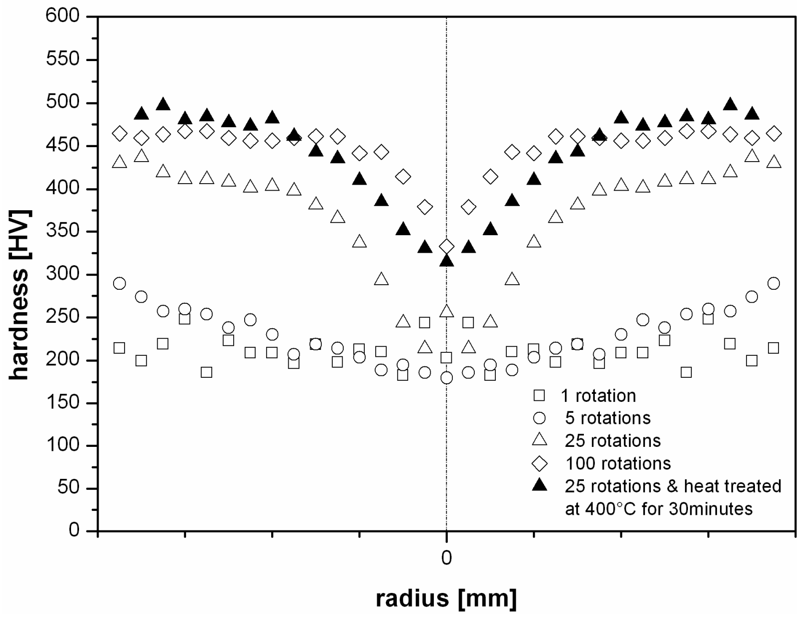

Fig. 1. Hardness of the CuCr HPT-deformed samples and the CuCr HPT-deformed and annealed samples

It is well known from literature that part of Cu can be dissolved in the Cr matrix but no Cr gets dissolved in the Cu matrix [3,4]. In the case of the 25rotation HPT deformed sample this is in the range of 10-20 at% and after heat treatment the Cr grains are free of Cu [3].

Furthermore, a detailed discussion about the mechanical properties and the crystallographic microstructure as a function of the deformation strain will be presented.

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The effect of loading conditions on the structure formation in A3003 alloy upon dynamic channel angular pressing

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Aluminium alloys have high potential as light structural materials. In these work deformation microstructures in industry Al-based alloys were studied. Severe plastic deformation was carried out by the dynamic channel angular pressing (DCAP). As a basis for experimental technique an ECAP like scheme were used, but instead of a press for forcing a sample though the channels the combustion products of powder gases were used. This method allowed reaching the deformation rates of materials equal 10^3 -10^5c-^{1}. Detailed investigations of severe plastic deformation in the Al-3003 alloy have shown an efficiency of the DCAP for the production of ultra fine- grained (UFG) bulk samples with a diameter of 14 mm [1,2].

Data of these studies indicated that there are two mechanisms of the structure formation – a fragmentation and dynamic recrystallization. Such a cyclical structure formation with increasing degree of strain demonstrats the realisation of two channels of the dissipation of elastic energy – plastic deformation and dynamic recrystallization. This work provides results of the studies of UFG structure formation in bulk samples with a diameter of 30 mm and a length of 150-200 mm. The effect of the size of the billets on deformation conditions was estimated. Specimens with diameter of 14 mm and 125 mm produced by DCAP were compared. Kinetic parameters and the effect of channel geometry on the character of stress-deformed state of the samples were determined. It was shown that velocity modes of these specimens loading are similar. Similarity of the deformation processes of specimens having different sizes was also observed in the distribution of shear stresses. Light and electron microscopy and X- ray diffraction were used to study the structure and phase composition of bulk samples. The average size of the structure fragments being calculated from the dark field patterns and histogram of distribution of structural fragment size were established. The values of a microhardness and of the Brinell hardness of bulk specimens were measured. Mechanical properties of DCAP specimens having different sizes were estimated.

This work was supported by the Program of the Presidium of the Russian Academy of Sciences "Heat physics and mechanics of extreme energy effects and physics of high compressed substance" (project № 09-P-2-1017).

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August 26, 2011

Y/Oral report

Grain refinement in copper via cryogenic deformation

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There is currently significant interest in the development of materials with nanocrystalline grain structure for structural applications. Severe-plastic-deformation (SPD) processes have been found to be among the most effective methods for fabricating such materials [1-4]. It is believed that the microstructural refinement down to nanocrystalline (NC) range imparts a considerable increase in their strength. An important limitation of such techniques, however, is the development of an equilibrium (minimum) grain size ~ 100 nm at high levels of strain [5,6]. One of the possible means for further grain refinement is known as "cryogenic deformation".

In present work we compare the structure formed after SPD by high-pressure torsion at cryogenic temperature in commercial-purity copper with the structure of the same material after long-term storage at room temperature. The imposed deformation comprised 20 consecutive, fully reversed 45^{o} rotations in the clockwise and counter-clockwise directions under an applied pressure of 4.5 GPa. To provide cryogenic deformation conditions, each test sample and the tooling anvils were soaked in liquid nitrogen and held for 20 min prior to testing. Microstructural changes during static storage at room temperature were quantified for periods ranging from 2 weeks to 11 months. Microstructures were determined primarily via electron backscatter diffraction (EBSD) but were complemented by transmission electron microscopy (TEM).

The comparison of orientation maps and TEM micrographs revealed the following features:

- Severe cryogenic deformation of copper has been found to lead to very poor microstructural stability. After 11 months at room temperature, the principal feature of the microstructure was the appearance of a number of abnormal, coarse (~10 μm) grains within a matrix of fine grains (~0.4 μm), i.e. the microstructure had become essentially bimodal. The very large difference between the grain sizes suggests that the material had undergone abnormal grain growth.
- The misorientation-angle distribution measurements revealed the reduction of HAB area and formation of annealing twins during storage the sample at room temperature.

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Structure formation of dispersion hardening ceramic SHS- materials in system Ti-Zr-C with binder

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Composite ceramic materials are widely used in many areas in industry. So, for example, materials based on Ti-Zr-C system are used as cathode targets by ion-plasma spraying, electrode materials for electro-spark hardening stamps for hot and cold pressing. Possessing high hardness up to 20 GPa, the given materials are characterized by the increased fragility, high porosity, insufficient heat resistance and low spark-erosion capability. Therefore, to increase strength and erosive capability, as well as functional properties of the deposited coatings, the composition of the carbide ceramics is introduced metal binder based on Ni.

Composite ceramic materials based on Ti-Zr-C system with binder were produced by combined force SHS-pressing technology.

It was established that SHS-products consist of the follow main phases: $(Ti,Zr)_{1-x}C_x$ and NiTi.

Structure of these composite materials presented by carbide phase with a grain size varying 0.6-2.4 μm and intergranular thin layer of NiTi with width less than 1.5 μm surrounded carbide grains (Fig 1.).

The presence of binder has allowed to essentially lower the porosity of samples to 1% and by that to increase hardness 2 times compared to materials without the binder, with improving fragility characteristics.

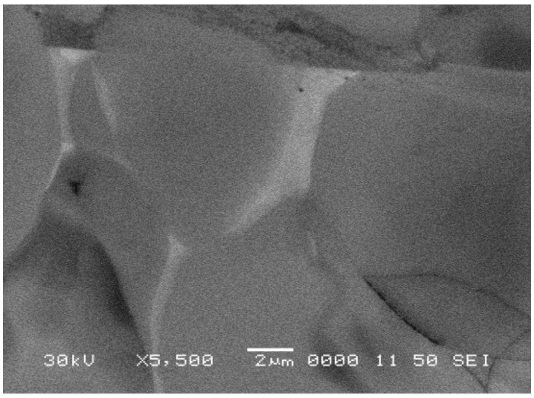

Fig. 1. Typical microstructure of SHS-products on example Ti-Zr-C system: $(Ti, Zr)_{1-[}C_x$ +NiTi

Y/Oral report

Explosively welded materials bond zone: morphology and crystallography

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Mechanical and performance properties of explosively welded metal joints are determined by phase composition, morphologic and crystallographic characteristics of thin nanostructured layers formed in immediate proximity to interface. It is need to understand physical nature of creation thin layers, to study the features of plastic deformation processes behavior at micro-, meso- and nanolevels, to investigate the character and evolution of imperfect structure forming for development explosive welding technology and obtaining joints having a good mechanical properties. Unfortunately, the information of this type is extremely limited. At present it was published only a few papers on this subject related to explosively welded joints such as titanium – titanium, titanium – orthorhombic titanium aluminide. It was shown that complex ensemble of dislocation-disclination defects led to forming fragmentation structure (indused by severe plastic deformation) and structure typical for dynamical recrystallization is formed close by interface zones.

In this paper full structural analysis of explosively welded materials Cu – Cu, Cu – nanostructured Cu foil – 0,09C-2Mn-Si steel (ferritic-pearlitic steel) was carried out. The investigations were conducted by methods having a different degree of resolution (optical microscopy, transmission electron microscopy (TEM), scanning electron microscopy (SEM) method of electron back scattered diffraction (EBSD)). The capabilities of heterogeneous materials mixing during microplastic deformation were studied additionally (using X-Ray spectral analysis SEM method).

The researches by using single reflection TEM method and EBSD-analysis shown, that nanostructured state is remained in foil (Cu) and formed in thin close by interface zones and melt regions in explosively welded joints Cu – Cu, Cu – nanostructured Cu foil – 0,09C-2Mn-Si steel. The investigations of local composition in bond zone neighbourhood using X-Ray spectral analysis along bond zone allowed to find regions without generation zones with components mixing, zones with components mixing, generated due to penetration particles of one alloy to another and alloy components co-dissolution in Cu – nanostructured Cu foil – 0,09C-2Mn-Si steel joint.

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26 August

BNM-2011 session

Physical and mechanical properties of BNM

August 26, 2011

Invited report

Consolidated nanopowder materials

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Nanocrystalline materials for the structural application are produced by the powder metallurgy methods, crystallization from amorphous state and sever plastic deformation. Structure features (grain size, considerable proportion of grain boundaries and their state) depend on the production methods and the structure of nanomaterials has grate influence for their properties. Physical-mechanical properties of the nanocrystalline materials differ from the properties of coarse grain and amorphous analogues. Strength is growth with the retention of sufficient plasticity of materials with grain size refinement. Research and development of the nanocrystalline materials, coatings and strengthening layers with enhanced operating ability have the practical importance for the structural optimization, increase their reliability, energy saving, improvement strength, frictional and antiwear properties of production.

Nanopowder consolidation is one of the basic methods of obtaining bulk nanomaterials. The powder technology is advantageous due to the possibility to obtain billets and articles of different shapes and sizes from virtually all types of materials: metallic, ceramic, polymeric, and composite.

At present, several ten methods are developed for the synthesis of metallic, ceramic, cermet, and other nanopowders. The first papers on the consolidation of ceramic and metallic nanopowders were aimed at obtaining bulk nanocrystalline materials, since it was assumed that they will exhibit the same unique strength properties as those of nanocrystalline metallic whiskers and the phenomenon of superplasticity.

Generally, nanopowders are pressed at room temperature by uniaxial static pressing, pressing *in situ* in a powder synthesis chamber, dynamic pulse-magnetic pressing, isostatic pressing, ultrasonic compacting, severe plastic deformation, and rolling of ribbons. In addition, other nanopowder consolidation methods such as centrifugation, osmotic consolidation, filtration under pressure, and electrophoresis have been developed.

The proposed method of nanopowder consolidation for the preparation of bulk billets by gas extrusion includes the sintering of a pressed piece in reducing atmosphere for the removal of oxide film from the surface of metal particles and hot pressing. The sintering in the reducing atmosphere is performed up to the attainment of closed porosity in the billet, and hot pressing is performed by extrusion in inert-gas medium by local heating of the deformation region to a temperature below the recrystallization temperature. The technical result is the development of the method for the manufacture of long metallic articles with nanocrystalline structure.

The grain structure of a compact extruded from nanopowder of nickel was investigated by scanning electron microscopy, electron backscatter diffraction and transmission electron microscopy. It was found that the specimen has a fine grain textured structure with preferred longitudinal orientations $\langle 001 \rangle$ and $\langle 111 \rangle$, which is in agreement with Taylor theory of texture formation. The grain size is of few microns, nearly every grain contains twins. During tensile testing such an extruded material shows tough fracture characterized by fine dimply topography of the fracture surface. The typical dimple size is of about a few micron. Inside the dimples a substructure with a characteristic size smaller than 100 nm is observed.

The technology of titanium carbide nanopowder synthesis was developed. This powder has a narrow fractional structure with a small maintenance of impurity. Regimes for titanium carbide powder pressing and sintering were determined. The porous material based on titanium carbide powder with open porosity nearly 50 % was synthesized. The strength of materials is in the range from 66 to 95 MPa and tends to reduction with growth of sintering temperature within 1250−1550°C.

Oral report

Comparison of the microstructures formation in Cu-Nb, Cu-V and Cu-Fe nanostructured microcomposites produced by arc-melting and heavy plastic deformation

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The possibility to attain the extremely high mechanical strength in copper matrix composites that contain the nanodimentional filaments of BCC metals is well known and associated with the large volume fraction of the interphase internal boundaries [1]. The specific nature of the interphase boundaries microstructures has been analysed for three different Cu matrix microcomposite materials with Nb, V and Fe as a strengthening filaments. The process of the formation of initial *in situ* composites by vacuum melting has been presented with the reference to the features of appropriate Cu-Nb, Cu-V and Cu-Fe diagrams of the state. The ways of attaining of the uniform distribution of the initial dendrite types of BCC metal inclusions in Cu matrix are discussed. The possibility to obtain the large scale billets with the diameters up to 130 mm with uniform distribution of the inclusions of BCC metal that are less than 10 μm has been shown for all three abovementioned systems. The process of the heavy deformation of the initial *in situ* composite ingots by extrusion and following drawing up to logarithmic deformation degree (lnAo/A) equal to 12 is described. The transformation of the microstructure during the deformation is analysed and illustrated by optical, SEM and TEM analysis. The strong FCC (111) / BCC (110) texture formation in microcomposites was confirmed for all investigated Cu-Nb, Cu-V and Cu-Fe wires. The peculiar features of the microstructures were investigated and some specific differences in the interphase boundaries nature had been revealed [2]. The mechanical strength of the microcomposites has been investigated. It was shown that the dependences of the ultimate tensile strength values on the true deformation degree did not show the tendency for the saturation with the increase of deformation that is illustrated in the fig.1, where the data are presented for all investigated wire microcomposites made of the abovementioned systems.

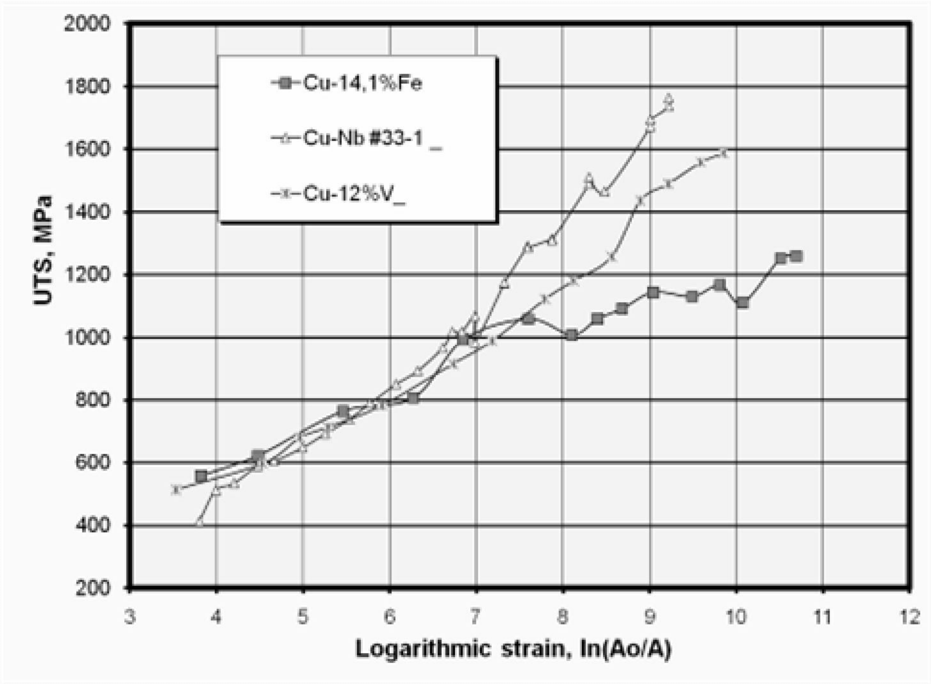

Fig.1 The UTS values of Cu-Nb, Cu-V and Cu-Fe microcomposites vs cold deformation degree

The difference in the rates of UTS increase with deformation that was revealed after attaining of the deformation value of 7 has been discussed and referred to the crystallographic parameters of the components in the each of investigated materials.

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Oral report

Effect of HPT processing temperature on strength of a Mg-Al- Zn alloy

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Abstract: Severe plastic deformation has been widely used to refine the grain structure and enhance the strength of magnesium alloys. However, the processing of these materials by Equal-Channel Angular Pressing is usually carried out at temperatures in the range of 473 K due to the limited ductility at low temperatures. The increased processing temperature leads to the occurrence of recovery and recrystallization in these materials. High-Pressure Torsion enables processing at a low temperature due to the imposition of a high hydrostatic pressure and therefore it permits an evaluation of strength as a function of deformation for different processing temperatures. The present study was designed to determine the evolution of hardness in an AZ31 alloy processed by HPT at temperatures of 296 K, 373 K and 473 K.

Oral report

On the influence of isochronal annealing on defect structure parameters, physical and mechanical properties of high purity submicrocrystalline Ni deformed by high pressure torsion

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The work deals with submicrocrystalline Ni as investigated by special methods of electron microscopy analysis [1]. After two rotations of the anvil a complicated microstructure with high density of defects, high values of lattice curvature and local internal stresses is formed. The following series of isochronal annealings leads to gradual defect relaxation processes. Characteristic parameters of grain and defect structure were defined together with estimation of local internal strain values and corresponding changes of physical properties – electrical resistivity and heat capacity on the material after different steps of the annealing.

The role of point defects and their complexes in the process of high-defect structure states formation and relaxation is discussed.

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Investigations were performed on the equipment of Collective Use Tomsk Material Science Center of Tomsk State University.

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Oral report

Effect of severe plastic deformation on the mechanical and tribological properties of Cu-based alloys

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Among the copper-based alloys, bronze is widely used as a tribo-material because of its moderate wear resistance and good corrosion properties [1-3]. However, relatively low hardness and strength of this alloy is the main reason to restrict its usage in some sophisticated tribo-applications. Therefore, the current wear performance of the Cu-Sn alloys has to be improved to extent its broader applications in such areas. The wear resistance of tribo-materials is thought to be able to increase by increasing their hardness and strength. For this purpose, heat-treatment and strain hardening can be applied. However, heat treatment is not feasible for the Cu-Sn alloy so that the precipitation of δ phase having a high hardness is quite slow and takes a long time [1]. Also, the hardness and strength obtained by heat treatment may not be high enough for such purposes. Recently, a new method (equal-channel angular extrusion/pressing (ECAE/P)) including severe plastic deformation has been developed for obtaining extraordinary increase in hardness and strength by grain refinement and strain hardening [4-6]. To the best our knowledge, no study has been performed on the effect of strengthening on friction and wear performance of Cu-Sn alloys. In view of the above, the main purpose of the current study is to investigate the effect of strengthening by applying severe plastic deformation on the friction and wear properties of Cu-11Sn alloy. For this purpose, the billets with the dimensions of 13x13x120 mm^3 were machined from homogenized alloy using wire-EDM and then processed by one-pass ECAE at 300°C. After processing, the microstructure, mechanical properties and friction and wear behaviour were examined.

The stress-strain curves of both as-received and ECAE-processed alloy is shown in Fig.1. This figure shows that the ECAE significantly increased the strength of the alloy but dramatically decreased its ductility. The yield and ultimate tensile strength increased from 176 MPa and 390 MPa to 742 MPa and 753 MPa, respectively. It was obtained more than two times increase in the hardness (from 102±2 to 274±4. of the as-received alloy by the effect of ECAE. Such improvement in mechanical properties brought about a considerable increase in wear resistance and a decrease in the friction coefficient for the Cu-11Sn alloy (Figs.2 and 3).

As seen in Fig. 2, the mass loss of the alloy sample increased almost linearly for the alloy in both conditions with increasing sliding distance. The friction coefficient of the alloy increased at the beginning of the test run and reached almost steady-state level after 50 m. The mean values friction coefficient taken from the state-state level are 0.59±0.05 for as-received and 0.45±0.12 for ECAE-processed alloy. The increase in wear resistance and the decrease in friction coefficient after ECAE processing were attributed to the considerable increase in both strength and hardness of the alloy. The increase in hardness and strength can lead to an improvement especially in the abrasive wear resistance according to Archard wear equation [7]. As well known, the main wear mechanism in bronze is the abrasion [8]. The increase in resistance against scratch with increasing hardness, on the other hand, decreased the friction coefficient (Fig.3)

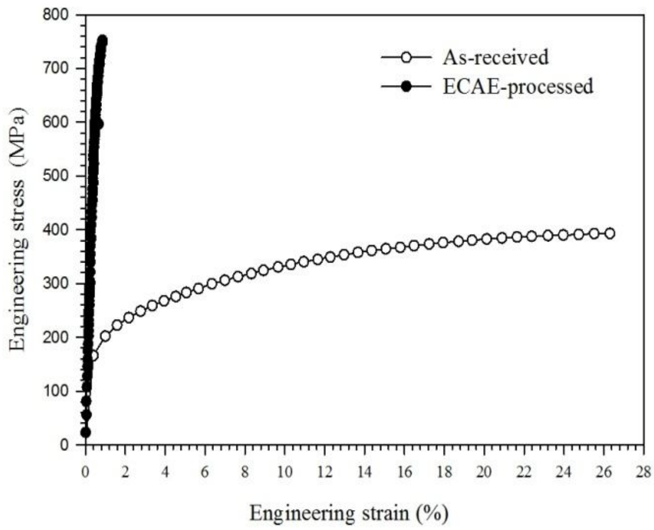

Fig.1. Engineering stress-engineering strain curves for the as-received and ECAE-processed Cu-11Sn alloy

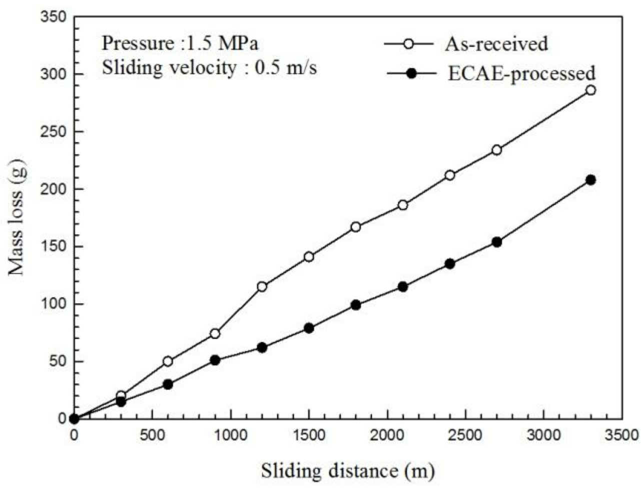

Fig. 2. The mass loss due to wear obtained from as-received and processed alloy versus with sliding distance

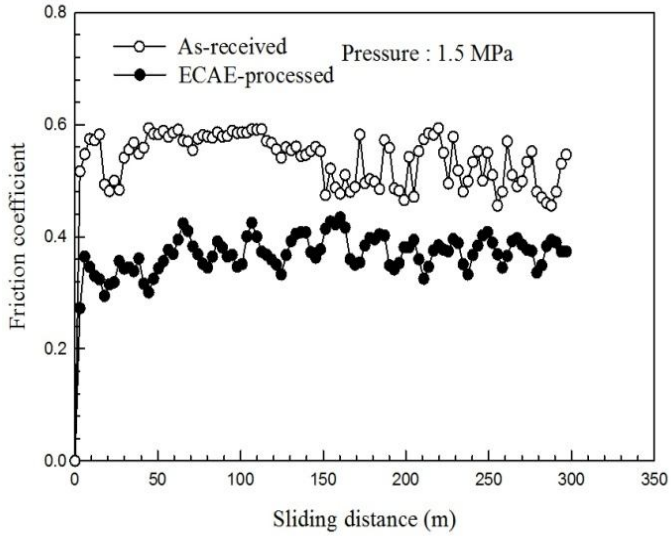

Fig.3. The change of friction coefficient as a function of sliding distance for the alloy

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August 26, 2011

Oral report

The effect of high-pressure torsion on structure and mechanical properties of single-crystalline high-carbon Fe-Mn-Al-C austenitic steels

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High-carbon austenitic steels (Fe-13Mn-1.3C (I), Fe-13Mn-2, 7Al-1.3C (II), Fe-28Mn-2, 7Al-1.3C (III), wt. %) was chosen for deformation by high pressure torsion (HPT). Steels have a stable austenitic structure and different stacking fault energies: ~30 mJ/mm^2 for steel (I), ~45 mJ/mm^2 for steel (II) and ~60 mJ/mm^2 for steel (III). The single crystalline austenitic steels were chosen as a model material without any inner boundaries which can effect the fragmentation process and evaluate the influence of special (twin) boundaries on mechanical properties of nanostructured steel. Specimens were subjected to HPT at temperatures of 20 and 400ºC and P=5-6GPa for 1, 3, 5 revolutions. The true logarithmic strain was calculated by the following equations: (1) $e = \ln(\upsilon r/h)$, at half the radius (r), where υ is the rotation angle and h is the thickness of the disk after HPT. The microstructure and mechanical properties was investigated after HPT using electron and optical microscopy, X-ray researches and measuring of microhardness.

The graphs in Fig. 1 show microhardness vs. strain ($H_\mu(e)$) for HPTed steels (I), (II), (III).Cold deformation by HPT of single crystals of Hadfield steel (I) leads to a high hardening due to intensive, simultaneous mechanical twinning in several systems. The formation of a net of thin twins promotes rapid fragmentation of the single crystal at low degrees of strain. Through an interaction between slip dislocations and twinning, the twin net is destroyed and twin boundaries are distorted and steps appear. An additional factor of strengthening is the internal twinning of primarily-twinned volumes and shear bands (SBs).

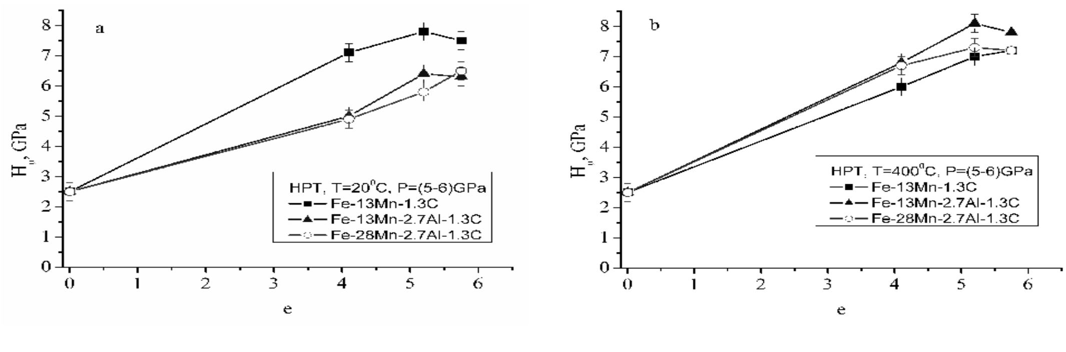

Fig.1. Microhardness vs. strain in steels subjected to HPT at 20ºC (a) and 400ºC (b)

The multistage evolution in the microhardness with strain demonstrates a strong correlation with the microstructure of the specimens. The rapid initial increase in the strength properties in the first stage ($e<4$) corresponds to the formation of a rectilinear net of thin twins. Multiple twinning was found to be the basic deformation mechanism responsible for the fast generation of an ultrafine-grained microstructure with boundaries of a special type (twins) in steel (I) single crystals after severe cold plastic deformation. As a result, the microhardness of steel increased noticeably at $e<4$. The subsequent fragmentation and destruction of the twinned structures and the formation of localized deformation bands have a less significant strengthening effect as it was also found in steels (II) and (III) (Fig 1a). Decrease in stacking fault energy suppresses twinning partially in steel (II) or fully eliminates it steel (III). Fig. 1a. demonstrates the fact that fragmentation due to slip in steels (II) and (III) has less significant effect on microhardness.

HPT at 400 ºC produces a non-equilibrium structure in steels (I), (II), and (III) containing the ultrafine particles - carbides of ~5 nm in size. These carbides are homogeneously distributed along crystals. At this temperature of HPT, there is no strong differences between strengthening mechanisms in steels investigated – refinement of initial structure due to high applied pressure, solid solution hardening and particle strengthening. As a result, the ($H_{\mu}(\varepsilon)$)-plots are weakly dependent on steel composition (Fig. 1b).

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Oral report

Microstructure of nickel alloy 718 subjected to equal-channel angular extrusion

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It is well known that ultrafine-grained and nanostructured bulk materials can be produced by severe plastic deformation (SPD) via equal channel angular extrusion (ECAE) [1] and multiple isothermal forging (MIF) [2]. The last one was used for nanostructuring of nickel based superalloys [3]. ECAE was used in present study to create an ultrafine-grained structure in Ni Alloy 718.

This paper will describe microstructure development as a function of starting grain size and process-route details. The experimental ECAE facility having channels with similar cross sections 12 mm × 24 mm, intersecting at an angle of 135 degrees was used without any back-pressure device. Extrusion was performed by route C (with 180-degree turn) on bars with fine-grained structure produced by MIF and on bars with the as-received coarse-grained structure. ECAE was performed under isothermal conditions at temperatures decreasing step-by-step in the interval 950-650°C.

Microstructural changes in the ECAE-processed alloys were studied using electron backscatter diffraction. ECAE of samples with the fine-grained structure at temperatures decreasing from 850-650°C (6 passes) led to the formation of a grain-subgrain structure with a mean size of about 400 nm. The alloy with fine-grained structure was subjected to hot deformation under conditions of ECAE in the temperature interval from 775°C to 725°C, the temperature being decreased by 25°C in accordance with the above specified route at each processing step. After 10 passes, a structure with an average grain size of about 700 nm and high dislocation density was generated in the bar.

Deformation of bars with a coarse-grained structure started at 950°C, the temperature being decreased by 25°C through route C up to 860°C. After 13 passes, the bar structure comprised of starting coarse grains of gamma phase, about 15 microns in average size, with delta phase plates, 0.5-1 microns in length and 50-200 nm thick, precipitating at grain boundaries and within the grain body interior. These plate-like precipitates divided the starting coarse grains into sub-grains with an average size of 700 nm.

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Oral report

Low temperature superplasticity and processing properties of titanium sheet alloy Ti-6Al-4V

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One of the most efficient technologies for processing sheet materials realizing advantages of superplasticity is superplastic forming (SPF) and its combination with pressure welding (PW) [1]. Many world leading aerospace companies apply and develop this technology for processing load-bearing aircraft components. Successful industrial implementation of SPF and SPF/PW technologies essentially depends on results of systematic studies of superplastic materials, their mechanical and processing properties, as well as their behavior under superplastic deformation conditions. The manuscript considers microstructure, mechanical and processing properties (formability and solid state weldability) of sheet titanium alloy Ti-6Al-4V with improved superplastic properties at low temperatures. Mechanical properties of the sheet samples having thickness of 1mm and 1.5mm were studied at room temperature and in the temperature range 650-800°C. The 1mm thickness sheet had microstructure different from that of the 1.5mm one. In all cases, the 1mm thickness sheet was found to have lower yield stress and higher plasticity that the 1.5mm thickness sheet. This titanium alloy can be applied successfully in the SPF/PW technology under conditions of low temperature superplasticity at temperatures 750-800°C. In particular, a hollow goffer type structure has been processed by the SPF/PW technology using the sheet titanium alloy Ti-6Al-4V (Fig.1).

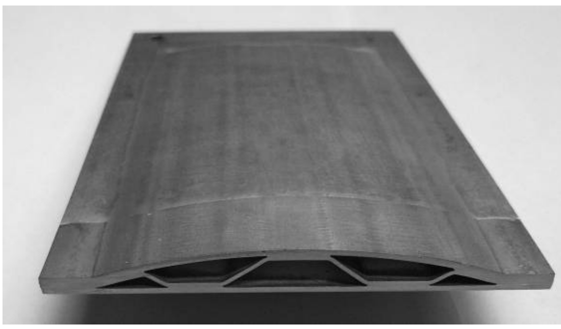

Fig. 1. Hollow goffer type structure produced by SPF/PW at T=750 °C

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26 August

Special session for young scientists and PhD students

Physical and mechanical properties of BNM

Y/Oral report

Effect of temperature-strain rate conditions of equal-channel angular pressing on the UFG structure formation and mechanical properties of the Ti-6Al-4V alloy

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The Ti-6Al-4V alloy is widely used in engineering, aviation and medical industries, due to its high strength-to-weight ratio and corrosion resistance. However, contemporary technological development demands increasingly higher requirements of structural strength in commercial alloys; bulk nano-structuring by severe plastic deformation (SPD) is considered as a potentially effective technique for the enhancement of mechanical properties in the Ti-6Al-4V alloy.

Bulk ultra-fine grained (UFG) two-phase titanium alloy billet, which can exhibit enhanced mechanical properties after processing by SPD, in particular via equal-channel angular pressing (ECAP), is nevertheless difficult to produce due to the low deformability of the material. Earlier this problem has been solved by applying high strain at elevated temperatures of 600-700 °C, combined with channel intersection angles of 120 and 135°, which consequently resulted in a decrease of strain intensity per pass, and a corresponding increase of material capability for receiving strain without the destruction of the billet.

However, α-phase grain size after such processing was approximately 400-500 nm, resulting in a low strength with a mean of 1210 Mpa, and relative elongation of 9%. It is known that lower temperature processing, with an accumulated strain of ≥ 4-6 at the intersection zone of the channels, is considered a necessary condition of refining the UFG structure in multi-pass ECAP. The present work revealed that damage-free billets were produced even at a temperature of 500°C, with total accumulated strain of $e \approx 5$, due to decrease in strain rate from $1 sec^{-1}$ to $0.13 sec^{-1}$ during ECAP; this has not been reported previously.

The microstructural investigations demonstrated the formation of UFG structure with an average grain size of 180 nm (Fig. 1); selected electron diffraction patterns demonstrate that high-angle boundaries predominate. The formation of this refined UFG structure led to an increase in ultimate tensile strength of 1335 MPa, at total elongation to failure of not less than 8%.

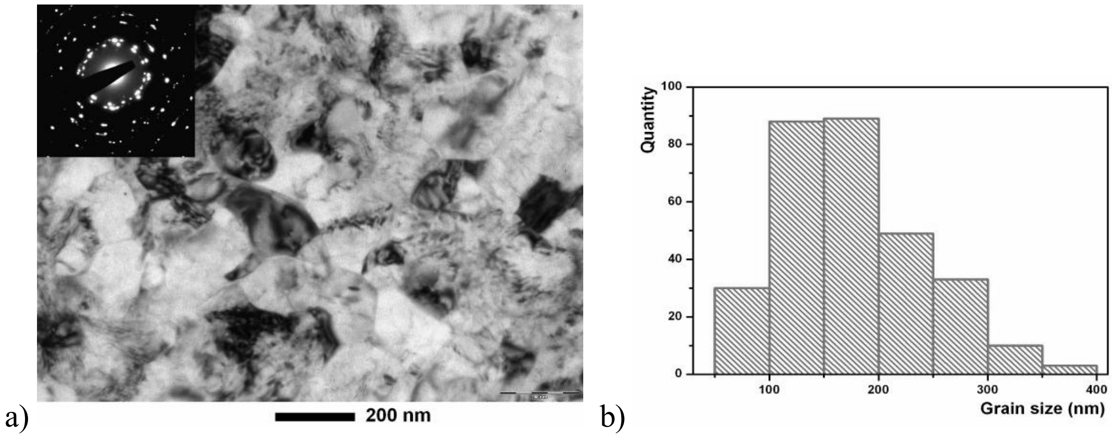

Fig. 1. Microstructure of the Ti-6Al-4V alloy after ECAP at the temperature of 500 °C.
a) TEM bright-field image and SAED patterns
b) histogram of grain sizes distribution via TEM; longitudinal section.

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Y/Oral report

Thermal stability of ultra-fine grained interstitial-free (IF) steel

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Interstitial-free (IF) steel reflects good formability but insufficient strength due to its ferritic microstructure with decreased amount of interstitial atoms. Therefore, it is generally used in plastic forming applications like deep drawing of automobile body parts due to its high formability and planar anisotropy [1]. However, low strength limits its broad applications. In order to expand its possible application areas, strengthening via grain refinement seems to be the most applicable procedure because of mono-phase microstructure of IF-steel. It was reported for many materials that ultra-fine grained (UFG) microstructure produced by severe plastic deformation (SPD) techniques caused substantially improved strength as compared to coarse-grained (CG) structure [2]. However, formation of the UFG microstructure brought about a considerable decrease in ductility for may materials as in the case of IF-steel [2]. It is obvious that low ductility constitutes a drawback for the post-SPD forming applications. Appropriate heat treatment was found to be the best way to enhance the formability of severely deformed materials by balancing their strength and ductility [3]. Therefore, he effects of annealing procedure on the UFG IF-steel were investigated systematically using optical microscopy and hardness measurements. The effect of annealing parameters on the corresponding mechanical properties and deformation behaviour were also determined by room temperature tensile tests.

The ECAE processing led to a significant refinement in the microstructure, and transforms the CG as-received structure with mean grain size of about 35 μm to the UFG structure with a mean grain sizes of 250 nm as seen in Fig.1. Effect of one hour isochronal annealing of UFG structure on hardness is represented in Fig.2. Annealing at temperatures up to 450 °C have no considerable effect on the hardness, where only recovery can took place. In the range of 450-650 °C, the hardness of the UFG structure decreased continuously with static recrystallization, above which normal grain growth stage became dominant. Stress-strain curves corresponding to the CG, UFG and UFG+annealed conditions are depicted in Fig.3. In general, the UFG structure before annealing exhibited limited strain hardening capacity as compared to the CG one. It is interesting that this capacity continued to decrease with annealing temperature down to 550 °C.

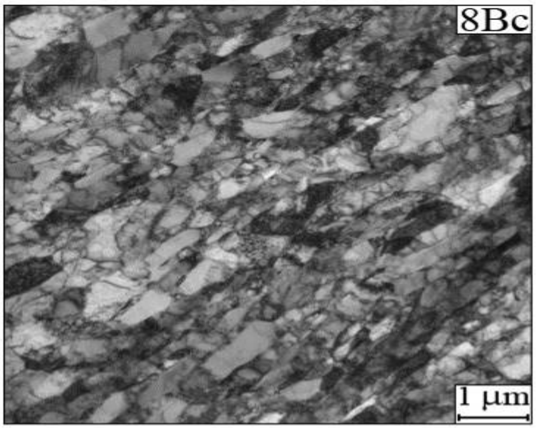

Fig. 1. Microstructure of UFG IF-steel

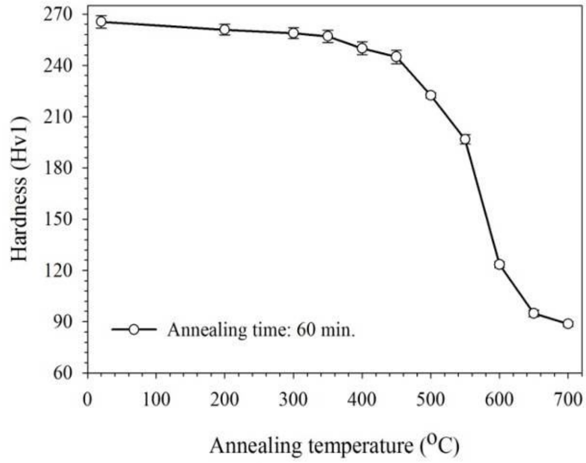

Fig. 2. Recrystallization curve of UFG IF-steel

This was attributed to the yield drop phenomena [4] as clearly seen in Fig.3. Annealing at temperatures above 550°C caused recovery of strain hardening behaviour. Therefore, it can be concluded that annealing at temperatures above 550 °C is necessary to obtain a good balance between strength and ductility for IF-steel processed by ECAE following route-Bc. This transition from yield drop to strain hardening is also confirmed by the examination of fracture surfaces, where sheared regions are clearly seen in the fracture surface of annealed at 500°C sample (Fig. 4a). Annealing at 600°C caused to formation of dimpled fracture surface due to recovered strain hardening behaviour (Fig. 4b).

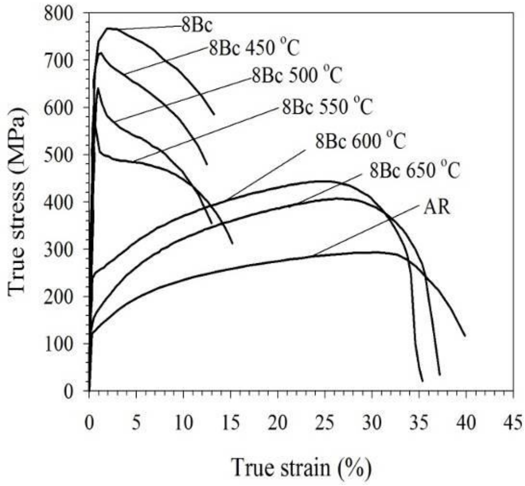

Fig. 3. Stress-strain curves of UFG IF-steel annealed at different temperatures for 1h

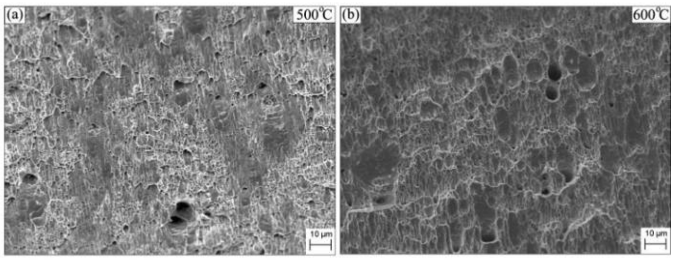

Fig. 4. Fracture surfaces of UFG IF-steel annealed at (a) 500 °C and (b)600 °C IF-steel

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Y/Oral report

Effect of high pressure torsion temperature on structure and properties of stainless steel AISI 316

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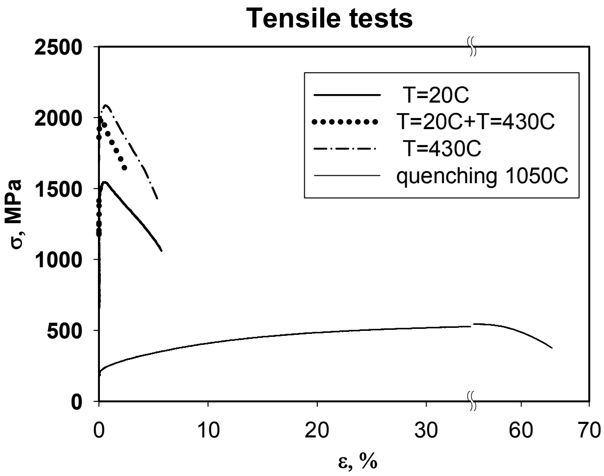
One of the principle problems of austenitic corrosion-resistant steels is low strength. It is possible to improve strength characteristics retaining the corrosion properties with the help of formation of sub-microcrystalline (SMC) or nano-crystalline (NC) structure by the methods of severe plastic deformation (SPD). However, the effect of the initial state, the phase state, the morphology and the chemical composition of the strengthening phases on the developing combination of properties of these steels is still confusing. The comprehension of contribution of every specified factor will allow to manage efficiently the process of structure development and formation of the complex properties of steel, subjected to the SPD.



The goal of this work is investigating the effect of the high pressure torsion (HPT) temperature on structure and properties of austenitic stainless steel AISI 316L (03Cr17-Ni14-Mo3).

The samples with the diameter 10 mm and thickness from 0.4 to 0.8 mm have been subjected to processing in coarse-grained austenitic state, obtained by quenching from 1050 °C. HPT has been performed at the increased temperature (430 °C), the room one (25 °C) and their combination. The microstructure and phase changes in the result of HPT applying the transmission electron microscopy (TEM) and X-ray analysis have been analyzed. The values of microhardness and mechanical properties have been determined on small samples with the base size 2 mm.

The application of HPT has allowed to get the structure with the grain size 60…100 nm and to increase the strength of steel 3…5 times (figure) in comparison to the initial state, depending on the HPT temperature. At the same time, the maximum strength has been achieved at the increased temperatures of HPT, which indicates that not only the small grain size, but also possible changes in phase composition deliver improved strength characteristics after HPT.

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Y/Oral report

High strength and ductility of ultrafine-grained pure copper processed by dynamic plastic deformation

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Abstract: Ultrafine-grained (UFG) metallic materials have been extensively researched due to their novel microstructure and properties. Particularly, simultaneous appearance of high strength and ductility are characteristic for such materials if they are fabricated by the methods of severe plastic deformation (SPD) due to non-equilibrium state of their grain boundaries [1] or due to the formation of bimodal microstructure in them [2]. The formation of bimodal microstructure has been also revealed in UFG copper obtained by SPD and then subjected to dynamic plastic deformation (DPD), performed with the strain rate over 10^3 s^{-1} [3].

The aim of the present paper is the investigation of the quasistatic deformation behavior of copper subjected to the DPD in different microstructure states.

Among the investigated samples there were the annealed initial state and the ones after an equal channel angular pressing (ECAP) at room temperature with the different number of passes (1, 4 and 8). All these states have been subjected to the DPD. The DPD has been realized by dynamic loading of split Hopkinson pressure bar with the strain rate 1431 s^{-1}. Quasi-static tensile tests have been conducted with the strain rate 2×10^{-3} s^{-1}. At least two samples have been mechanically tested for each investigated state.

The engineering stress-strain plots for the investigated states are presented in Figure. The microstructure of the initial state before the DPD has been characterized by coarse grains. The corresponding state has been characterized by low strength but high ductility. After the 1st ECAP pass the microstructure has been the fragmented one with the low angle misorientation angles. This explains the observed increased strength but decreased ductility. Cleavage fracture has been observed in the UFG copper after the 4th ECAP pass and DPD after tensile test, which implies the formation of adiabatic shear bands which is unprofitable for the exhibition of high ductility.

The increase in the number of ECAP passes up to 8 has been resulted in the development of the high angle UFG microstructure which explains the high strength. At the same time DPD of this

highly nonequilibrium state has activated the dynamic recrystallization processes. As a result, the recrystallized grains have been formed in the deformed matrix. This supplies for the state after the 8th ECAP pass plus DPD the possibility to exhibit both the high strength and high ductility simultaneously.

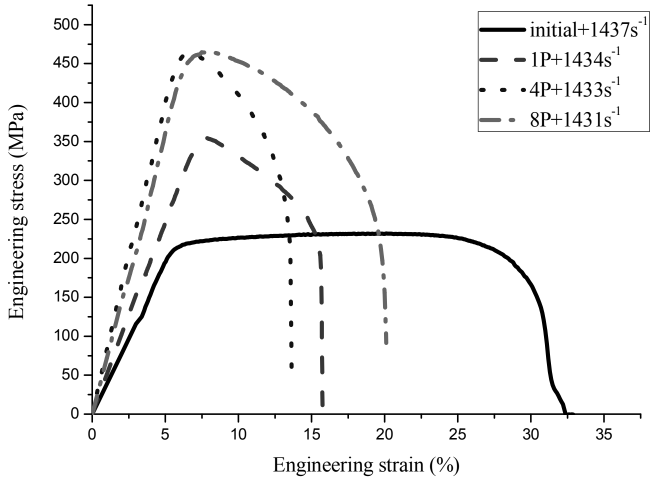

Fig. Engineering stress-strain plots

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Y/Oral report

Features of the influence of nanoparticles on mechanical and tribological properties of metal-matrix composites

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Over the last 20 years, the amount of research work performed in the area of metal-matrix composites (MMCs) has been very large and impressive. The driving force has been the fact that addition of ceramic reinforcements into metallic matrix can improve specific strength, stiffness, wear, fatigue and creep properties compared to conventional engineering materials. The use of nanosized particles (instead of microsized ones) for reinforcement of hard compounds is advantageous for the following reasons. First, according to the Orovan equation, the effectiveness of dispersion strengthening depends on the particle size of embedded particulates, so that a relatively low amount of reinforcing phase (below 5 vol. %) can be expected to markedly improve the mechanical properties of reinforced alloys. Second, the chemical activity of nanoparticles is known to be higher than that of bulk material due to better interparticle contact between the components.

The Fe-, Co-, Cu-based metall-matrix composites reinforced by nanoparticles of the WC, ZrO_2, Al_2O_3, W, and CNT (carbon nanotubes) has been studied. Initial mixtures were prepared using planetary mill followed by ultrasonic treatment of nanoparticles containing mixtures. Obtained mixtures with various content of nanosized additives were sintered by HIP in inert atmosphere. It was established that nanoparticles located on grain boundaries has a constitutive influence on mechanism and kinetic of the sintering process. The sintering kinetics was found to depend on whether or not the interaction between added nanoparticles and matrix powder takes place (using as examples inactive ZrO_2 and reactive WC nanoparticles).

An increase in the amount of added nanoparticles leads to their aggregation and accumulation of conglomerates in the porous interparticle space of the binder, which exerts a decelerating effect on the compaction process. In hot-pressed samples, the reinforcing phase was found both in the grain body and its boundary [1] (*Fig. 1*).

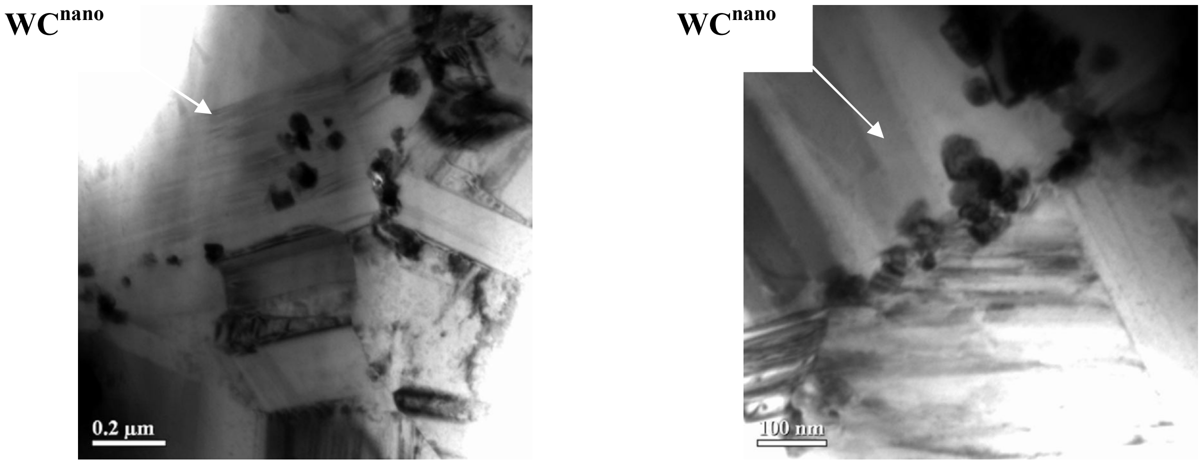

Fig. 1. TEM images of sintered Co–WC^{nano} alloy

Dispersion-strengthened MMCs produced by hot pressing technology showed an increase in the hardness (by 5–10 HRB), bending strength (by 54%), wear resistance (by a factor of 2–10) and a decrease in the friction coefficient. The application of designed alloys as a binder of diamond tools for cutting reinforced concrete gave a 2-fold increment in the service life of tools, without reduction in their cutting speed [2].

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Y/Oral report

Effect of severe plastic deformation on properties of high-carbon steels

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Improving the mechanical properties of steels of different kinds is the topic of interest, as they are widely used in various machines and mechanisms.

One of the methods used for attaining high mechanical properties of steels is refinement of their microstructure due to the severe plastic deformations (SPD).

This work investigates the possibilities to improve the mechanical properties of a high-carbon steel Fe-1%C-1,5%Cr by the method of severe plastic deformation. The equal-channel angular pressing (ECAP)has been used as one of the SPD methods.

The analysis of the technological capabilities of the SPD steel have been carried out by computer simulation method of the ECAP process, using the software DEFORM 3D. The optimum deformation speed and the friction coefficient have been chosen in the result of computer simulation, which allowed matching the efficient lubrication.

The samples for the ECAP were the cylinders with the diameter 10 mm and length 65 mm. Before the SPD the samples had been subjected to quenching from temperature 840 °C to oil. In the result the samples before the ECAP had been characterized with martensite microstructure with an insignificant share of carbides (2%).

The ECAP has been carried out in the range of temperatures from T=350 to 500 °C in the die opening with the diameter 10 mm, with the angle of channel interface 120°. The number of passes had varied from 1 to 10.

The results of mechanical tests are reduced in the table.

It has been demonstrated that after the 1^{st} ECAP pass at T = 400 °C for the samples had a fine structure, characterized with the disperse structure elements of ferritic-carbide mixture with the size 100-200 nm. This speaks for the fact that the bulk nanostructured state develops just after the 1^{st} ECAP pass. This state is characterized by high strength parameters and noticeable ductility.

Table 1. The results of mechanical tests (ECAP at T = 400 °C)

Type of processing	σ_u, MPa	$\sigma_{0.2}$, MPa	δ, %	ψ, %
Initial	680	369	29	60
heat treated	2150	1956	<1	<1
ECAP, $N = 1$	1924	1900	4	13
ECAP, $N = 2$	1728	1705	2	6,6
ECAP, $N = 4$	989	973	13	48
ECAP, $N = 10$	1590	1500	16	29

The following increase of deformation temperature or the number of ECAP cycles of steel in an initial state does not bring to the mechanical properties improvement. At the same time the nanostructures samples, obtained after the 1st ECAP pass are characterized by a high strength at the heat treated level and a significant ductility.

So, the SPD presents a perspective way to develop the bulk nanostructured states in high-carbon steels with the improved mechanical properties.

Y/Oral report

Temperature effect on theoretical strength of nanomaterials

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Ideal strength is the highest achievable stress of a defect-free crystal at zero temperature. This property of materials is of interest from both theoretical and experimental points of view. It was shown from the recent experiments that materials can response on external loading in a way that intrinsic stresses reach significant fraction of the ideal strength [1]. For instance, it is approved by recent nanoindentation experiments of graphene where intrinsic strength was measured to be 13% of corresponding Young's modulus [2]. Such a high stress values can be achieved due to dealing with small length scale of indenter tip, when strength is caused only by force of interatomic bonding, while for higher length scales it is controlled by propagation and motion of defects. These results stimulate investigations of ideal strength of different materials. For instance, by means of DFT approach *Ogata et al.* calculated ideal shear strength for wide range of metals and ceramics at zero temperature [3]. *Michal Jahnátek et al.* have analyzed the mechanism of shear deformation and ideal strength for FCC metals, but also for 0K [4], meanwhile experiments are carried out at elevated temperatures, which makes essential investigating ideal stress at finite temperatures. So far a little was done to clarify the influence of temperature on ideal strength. *Lin Yuan et al.* have established that ideal tension strength of aluminum nanofilm decreases from 2.70 GPa at 10K to 2.39 GPa at 800K [5]. *Yoshitaka Umeno et al.* performed instability analysis of nanofilm with initial notch under tension and it was also approved that finite temperature reduces material's strength [6]. However there is no comprehensive information available on the temperature effect on ideal strength of pristine metallic crystals.

The aim of the present work is the investigation of temperature effect on the ideal shear strength and instability mechanisms of FCC metals. Molecular dynamic simulations of pristine FCC metals were performed for {111}<112> shear using stress control. Having applied gradually increasing shear stress, we estimated crystal's response in terms of strain-stress relation at different temperatures and for simulation cell of different sizes.

It was established, that increasing temperature results in significant decreasing of ideal shear strength. Dependence of ideal shear strength and critical strain on temperature was built: ideal shear strength decreases linearly with increasing of temperature.

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BNM-2011

Poster Session

Modelling of superplastic forming of hollow three-layered structures made of VT6 titanium alloy to find rational geometry parameters

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The superplastic forming process of the VT6 titanium alloy three-layered hollow structures with goffered filler is investigated. Based on the results of experimental studies and on the results of finite element modeling, we determine rational parameters of the process such as gas pressure increase rate, when superplastic flow condition is satisfied for ribs, and the processing time is minimal. Various methods of metal forming find their applications for producing a broad nomenclature of structures including those widely used in the aviation and space industries. In the latter applications, the three-layered hollow structures are very important because they allow for weight reduction. Such structures are typically made of three sheets of titanium alloy with the help of the superplastic forming/diffusion bonding (SPF/DB) technology. The outer sheets form covering while the inner one gets goffered making ribs. Further development of this technology would enable production of relatively cheap and high-quality parts of aircrafts and spaceships. In the present work we aim to improve the SPF/DB technology of producing the three-layered hollow structures made of titanium alloy by using the finite-element method simulations and comparing the simulation results with the experimental ones [1-2]. Finite-element modeling of the SPF process of the three-layered hollow structure has been carried out in order to choose rational parameters of the SPF process such as gas pressure increase rate and the processing time. As the criterion of rationality we have used the fulfillment of the superplastic flow condition in the ribs. We have demanded the fulfillment of the superplastic flow condition only in the ribs because they experience the largest strain.

We have found that the SPF process of the three-layered hollow structure goes in two stages. At the first stage, the upper outer sheet (cover) is not in contact with the die and deformation of ribs takes place. During the second stage, the deformation of the regions of cover with large curvature takes place, while the strain rate of ribs becomes much smaller than at the first stage. The two-stage loading scheme was offered to minimize the total processing time.

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Homogenization of structure and properties of rods made from chromium bronze with a welded joint by equal-channel angular processing technique

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Weld seams are considered as areas of mechanical, structural and chemical heterogeneity in the material. Such areas may cause serious problems in long-length rods because they weaken an item and are corrosion zones and locations of fatigue damage development.

In the present work the problem of producing a long-length rod with a uniform structure and a welded joint was solved by the example of rods out of low-alloyed dispersion-hardened Cu-Cr alloy. The unique combination of strength and electric conductivity - 700 MPa and 70%IACS or 600MPa and 77%IACS – can be achieved in these alloys by ECAP and thermal treatment. Such functional properties of these materials are extremely attractive for production of contact wires for electric transport and, in particular, for high-speed trunk railways.

The problem of long-length ingots production still remains unsolved because the smelting volume for alloys of this system is limited by the conditions of distribution uniformity of alloying elements during hardening.

The severe plastic deformation technique, equal-channel angular processing (ECAP), and its modification ECAP-C to be applied to process long-length rods can be used not only to achieve enhanced mechanical and functional properties in the material, but also to homogenize the structure of welded joints.

It has been established that the area of weld seam spreads, however, elongates to 4 times and more and reorients towards the longitudinal axis, when a proper deformation route is chosen. The structure of material in this area is similar to the structure of the bulk. Due to such structure homogenization, mechanical properties in the seam area become similar to those in the material bulk, and fatigue damage development is less likely to occur in the seam area.

Superplasticity of Al-Mg-Sc alloy produced by ECAP and subsequent rolling

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Superplastic properties of an Al-Mg-Sc alloy subjected to hot equal channel angular pressing (ECAP) and subsequent isothermal cold and hot rolling with reductions of 80 and 87%, respectively, were examined in the temperature range of 350-520^{o}C at initial strain rates of 10^{-3}-10^{-1} s^{-1}. It was found that the cold rolling after ECAP results in formation of the microstructure that provides relatively week superplastic properties. The maximum elongation to failure was achieved to be less than 350% at all temperatures and strain rates investigated. This is caused by a fact that the heating of the sheet samples to the temperature of hot deformation promoted an extensive grain growth and formation of a non-uniform microstructure with the bimodal grain size distribution. The sizes of the coarse and fine grains in this structure were as high as 18-30 and 4-7 μm, respectively. An extensive cavitation (~5.2%) occurred during hot deformation that resulted in a rapid failure of the samples.

The isothermal hot rolling, carried out at the temperature of ECAP, resulted in significant improvement of the uniformity of the microstructure produced by ECAP and provided formation of the ultrafine-grained microstructure with an average grain size of about 1 μm and the volume fraction of ultrafine grains of about 0.9-0.95. The microstructure obtained exhibited very high thermal stability upon annealing in the temperature interval 350-450^{o}C and provided achievement of elongation to failure more than 2000% at the strain rates of about $10^{-1}s^{-1}$. The volume fraction of cavities near to fracture did not exceed ~1.5%. The difference in the superplastic behavior of the ECAPed material subjected to cold and hot rolling is discussed in detail.

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Molecular dynamics simulation of cooperative grain boundary sliding in 2D polycrystal

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During the last decades, in mechanics and solid state physics much attention is given to investigation of inhomogenities of plastic deformation at different scale levels. It is also interesting in connection with development of methods of nanostructuring of metals by severe plastic deformation [1, 2]. Grain boundary sliding is a very important mechanism of plastic deformation of metals at high temperatures. Cooperative character of grain boundary sliding [3] becomes more pronounced with decrease in grain size [4]. There exists a number of experimental data supporting the efficiency of the cooperative grain boundary sliding (CGBS) in arranging a shear in a polycrystal in a way that involves many grain boundaries and does not result in creation of discontinuities in the material [5]. Realization of CGBS at the atomic level is understood very poorly. Even though 2D models are simplifications of the real systems, they can provide results of physical relevance. The aim of this work is to investigate CGBS by means of molecular dynamics simulations.

We consider a two-dimensional polycrystal with nano-size grains based on the hexagonal lattice that can be regarded as the (111) plane of fcc lattice. We do not aim to reproduce properties of any particular material and employ simple pair interatomic potential used in [6]. CGBS is investigated at given temperature, hydrostatic pressure, and maximal shear stress. Computational cell is subjected to periodic boundary conditions. Initial grain structure included a grain that played a role of an obstacle of the CGBS. The self-organization mechanisms of CGBS that resulted in overcoming of the obstacle were analyzed. In Fig. 1 is shown the crystall microstructure at the shear strain level a) $\gamma = 0.06$ and b) $\gamma = 0.22$. CGBS takes place along the line shown by arrows. The grain playing the role of an obstacle is indicated by the circle.

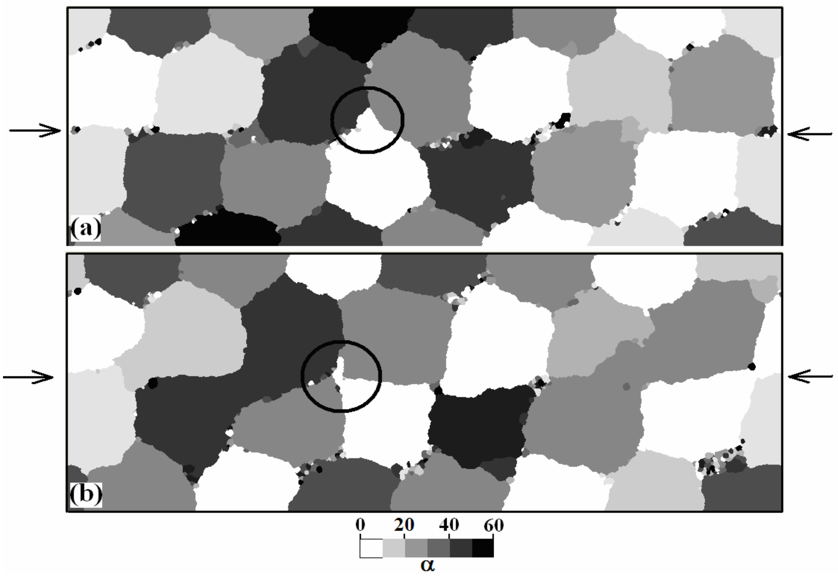

Fig. 1. Microstructure at the strain level a) $\gamma = 0.06$ *and b)* $\gamma = 0.22$*. The lattice orientation angle* α *in grains is shown according to the colour bar*

It was found that the obstacle was overcome by means of dislocation nucleation and sliding across the obstacle grain. As a result, a law-angle grain boundary was formed and later transformed into the high-angle one. Present simulations allow to better understand the mechanism of development of CGBS at atomistic level, especially for nanostructured materials. Study of CGBS is also interesting from the standpoint of the fundamental physics because it has much in common with other physical cooperative processes observed during, e.g., initiating of snow avalanches and electrical discharges. Such processes begin at a low scale length, then develop gradually by cumulating the momentum and make a way through the system at the macroscopic level. Moreover, CGBS can be characterized as a non-monotonous, wave-like process.

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Ageing behavior and properties of ultrafine-grained aluminum 6060 alloy

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The peculiarities of ultrafine-grained (UFG) structure formation, mechanical properties and electrical conductivity of the aluminum 6060 alloy of Al–Mg–Si system subjected to HPT are studied in the work.

The samples of 6060 alloy were subjected to HPT both at room temperature (RT) and at the temperature of 180°C in order to form an UFG structure.

The typical UFG structural states formed in the 6060 alloy ingots due to HPT processing are given in Fig. 1. A homogeneous UFG structure with an average grain size of 180 nm (Fig. 1.a) was formed in alloy ingots after HPT at RT. An UFG structure with average grain size of 350 nm (Fig. 1.b) was formed in the ingots after HPT at the temperature of 180°C.

There was observed a great number of precipitations of Mg_2Si nanosized strengthening phases (Fig. 1.c,d) in the UFG 6060 alloy structure after HPT at an elevated temperature. These precipitations, according to morphological characteristics, i.e., globular form and size of 20-40 nm, refer to their stable modification - β-phase, the noncoherent aluminium matrix.

The revealed change in the lattice parameter of the alloy as well as the data on 3D atomic tomography data indicate that the concentration of alloying elements in UFG matrix after HPT at the temperature of 180°C in the process of DSA decreases by the order of magnitude and leads up to that of pure Al.

Thus, such an UFG state provided high strength of alloy due to grain size decrease in accordance with Hall-Petch relation and formation of hardening phase precipitations in aluminum matrix due to aging.

The increased material electric conductivity was provided by decrease in concentration of alloying elements in the aluminum matrix because of solid solution decomposition during HPT processing and also less amount of defects in formed UFG structure.

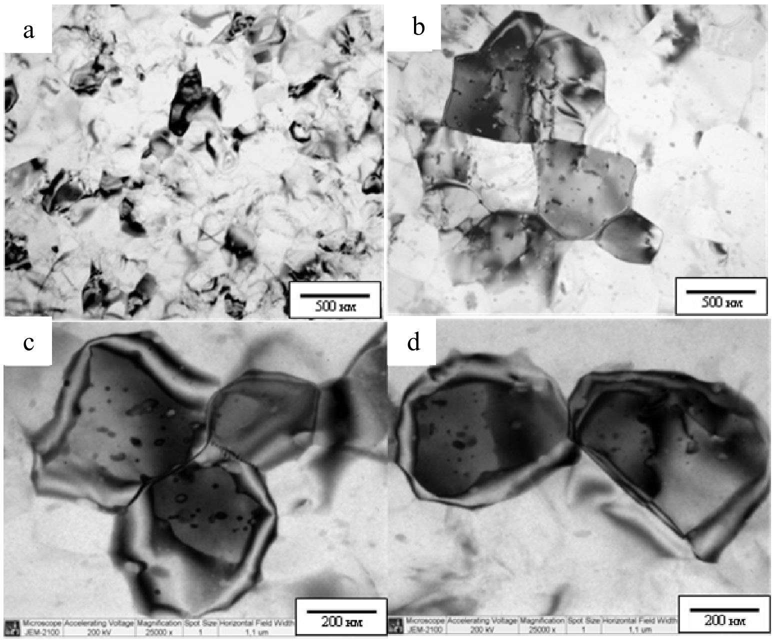

Fig. 5.1. 6060 alloy structure after HPT processing: (a) – at room temperature; (b) – at the temperature of 180°C; (c,d) – particles of strengthening phase, precipitated in the DSA process during HPT processing at the temperature of 180°C

The analysis of mechanical characteristics and electrical conductivity (ω) demonstrated that formation of such an UFG state provides higher strength and electrical conductivity in the alloy. The tensile strength was ~ by 40 % higher than after standard T6 processing, ω and IACS increase from 31.1 to 33.7 mS/m and from 53.6 to 58.1 %, respectively, leading up to pure Al. Earlier the combination of such high strength and electrical conductivity was not observed in aluminum alloys of Al-Mg-Si system.

Computer simulation of metal flow during ECAP-Conform process

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The equal channel angular pressing (ECAP) technique via the Conform scheme implements the principle of active friction forces influencing the lateral surface of a billet. It is used for high-capacity production of long-length, metal semi-products to be applied for fabrication of various items.

Influence of the scaling factor on thermal and mechanical conditions of metal processing is an important matter taken into consideration when developing the Conform pressing technique, as the scaling factor has impact on mechanical and service properties of produced items.

Research objective

Studies of thermal and mechanical conditions of ECAP-Conform processing with account of the scaling factor.

Research tasks

- Identification of quantitative relations and establishment of dependences in order to estimate the scaling factor influence (billet cross-sectional dimension) on the thermal and mechanical conditions of metal processing during ECAP-Conform;
- Development of a procedure for calculating the parameters of ECAP-Conform processing with account of the scaling factor.

Experimental procedure

- Change of the billet form was modeled with the help of the DEFORM 3D software package during investigations;
- A long-length billet made of steel 10, with a square cross-section of 10, 15 and 20 mm, with a length of 200 mm was taken for research and subjected to equal channel angular pressing via the Conform scheme. The processed billet had a square cross section. The channels intersection angle was 120º;
- The conditions of metal deformation during the Conform processing were analyzed on the basis of 9 points which were equally spaced in the billet cross-section. 9 points were chosen in the half of the cross section, as the stress-strained state is symmetric as regard to the longitudinal symmetry plane. The material points were out of plastic deformation zone at the starting time.

Conclusions

- The scaling factor has a considerable influence on the thermal and mechanical conditions of metal deformation during Conform processing of a UFG steel semi-product and may have an impact on the mechanical properties of fabricated semiproducts;
- When the billet cross section increases, non-uniformity of heat distribution in the deformation zone grows;
- When the billet cross section increases, non-uniformity of the strain state distribution over the billet section and the strain value of fibers, which are located in the section center and also near the hold-down roll surface, increase. The highest strain degree and rate in the deformation zone has been observed near the points located not far from the roll surface;
- The scaling factor has a considerable influence on the stress state, the value of stress state changes from minus to plus in the points, which are not far from the roll surface, when passing through the deformation zone;
- The plotted curves demonstrating the scaling factor dependence of thermal and mechanical conditions of metal deformation are very useful to predict these data for various sections of a billet (to 20 mm) without additional investigations.

Deformation and load-bearing parameters of equal channel angular pressing of a cylindrical metal billet

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When equal-channel angular pressing (ECAP) is applied, simple shear takes place when a billet passes through the zone of conjugation of channels with equal sections in a special die-set. The die output channel is usually constructed with some narrowing. The narrowing output channel of the die provides increase of the module of the average compressive stress in the area of deformation zone shift and production of a billet with a diameter which is less by 3…5 % than the one of the initial billet. This allows conducting easier multiple-pass deformation by feeding a billet up into the die inlet channel many times.

Objective: to establish deformation and load-bearing parameters of ECAP processing of cylindrical metal billets in the die with a narrowing outlet channel.

Tasks: - to find out the acceptable for practical application dependences for strain rate calculation and specific force of ECAP processing of a cylindrical metal billet in the die with a narrowing outlet channel;

- to compare the received simulation and calculation results with the results of experimental ECAP processing of a cylindrical metal billet;

- to draw up a methodology for calculating the technological parameters of equal channel angular pressing of cylindrical metal billets in the die with a narrowing outlet channel.

Simulation results. The process of equal channel angular pressing conducted at a temperature of 500ºC was simulated. The maximum force of 103 kN was received at the moment of formation of a plastic deformation zone.

Then the deformation force decreases, the reason for this is that the frictional area between billet and die inlet channel reduces.

Formulae derivation. The average strain rate in the areas of shear and tension in the deformation zone is: $\bar{\dot{\varepsilon}}_{i,1} = \dfrac{\cos\gamma \cdot \upsilon}{\sqrt{3}D\sin\alpha}$; $\bar{\dot{\varepsilon}}_{i,2} = \dfrac{\ln\left(D/d\right)\cdot \upsilon \cdot \pi D^2}{4V_2}$

After substitution of the expressions and formula simplification of the formula we get the dependence for the specific force of ECAP processing of a cylindrical billet:

$$p = \frac{\overline{\sigma}_S \mathrm{ctg}\gamma}{\sqrt{3}} + \overline{\sigma}_{S1} \ln(D/d) + \frac{4 f_1 \sigma_{S0}}{\sqrt{3}} \left(l_1 / D + \frac{1}{2} \left(\mathrm{ctg}\gamma - \frac{\sin\alpha}{\sin\gamma} \right) \right) + \frac{4 f \overline{\sigma}_S \sin\alpha}{\sqrt{3} \sin\gamma} + \frac{(\sigma_{S0} + \sigma_{S1})}{\sqrt{3}} \times$$

$$\times \mathrm{ctg}(\gamma + \alpha) + \frac{f_2 \overline{\sigma}_{S1} \left(1 + D^2 / d^2\right)}{\sqrt{3}} \left((D + d) / D^2 \sqrt{(D-d)^2 / 4 + l_2^2} + \mathrm{ctg}\gamma - \sin\alpha / \sin\gamma \right)$$

.

The force of equal-channel angular pressing of a billet made of ShH15 steel with a diameter of 10 mm and a length of 65 mm calculated using the formula was compared with the force value obtained through simulation. It was found out that the calculated value was close to the maximum one received by simulation.

Experiment results. The defect-free billets of ShH15 steel have been produced via equal channel angular pressing at a temperature of 500 °C. The maximum force obtained experimentally is 110 kN that exceeds by 6.7% the maximum force obtained via simulation, and by 10.9% the value obtained with the help of the formula

Conclusions:

1. The formulae for calculation of a strain rate and specific force of ECAP of a steel cylindrical billet in the die with a narrowing outlet channel have been derived.

2. The calculated force with the accuracy acceptable for practical application agrees with the force measured during experimental equal channel angular pressing of a cylindrical billet made of steel ShH15 in the die with a narrowing outlet channel.

Fracture mechanisms of ultra-finegrained aluminum alloys after dynamic pressing

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Analysis of fracture surfaces is needed to determine reasons for the difference of the mechanical properties of the samples associated with the technology of their production. Understanding of the destruction micro mechanisms of materials is an important aspect for a creating of new structural materials. Bulk samples of aluminum alloy A3003 and A7075 with structure fragment sizes of 200-400 nm were obtained by method of dynamic channel-angular pressing (DCAP). Formation of ultra- fine-grained (UFG) structure was achieved after 1 - 4 passes. High strain rate and specificity of stress-strain state of material, consisting in the imposition of impact shock and shear are the cause of the rapid kinetics of the structure dispersion. Analysis of the fractures of the tested samples was performed using a scanning electron microscope Quanta-200.

Nature of the fracture of DCAP samples of the A 7075 alloy, obtained with one cycle of compression, is viscous. The average grain size in this sample is 200 nm, but there are coarse grains in sizes of 500-700 nm and a large number of small intermetallics retained after deformation. Fracture surface consists of irregularly mixture of large and small pits of varying depth related to the heterogeneity of the material. The size of pits varies from 1 to 1.5 mkm. Fracture DCAP sample after two cycles of pressing has become more homogeneous in terms of depth and size of pits. The average size of pits decreases slightly (less than 0.9 mkm), and the depth increases (Fig.1). This corresponds to a more uniform distribution of grain size. The number of grains having size larger than 500 nm decreases, and the proportion of grains having size smaller than 200 nm increases. Increasing the depth of pits connects to the growth plasticity and confirmed by the increased values of elongation with 10 (for the sample after 1 cycle DCAP) to 14%. Plastic intracrystalline fracture occurs through the emergence, growth and coalescence of micropores on the boundaries: the matrix - the intermetallics. Micro mechanism of fracture is "pitting break away". Appearance of deformed samples of A3003 alloy showed that the fracture occurs by shear. Intracrystalline fracture mechanism was observed in all samples obtained at 1-4 cycles of DCAP (Fig 2).

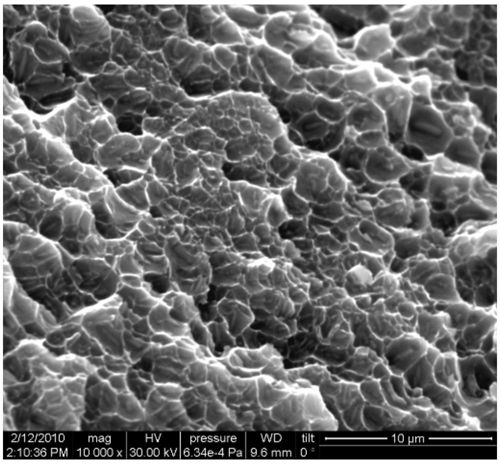

Fig.1. Fracture surface of the A7075 alloy sample

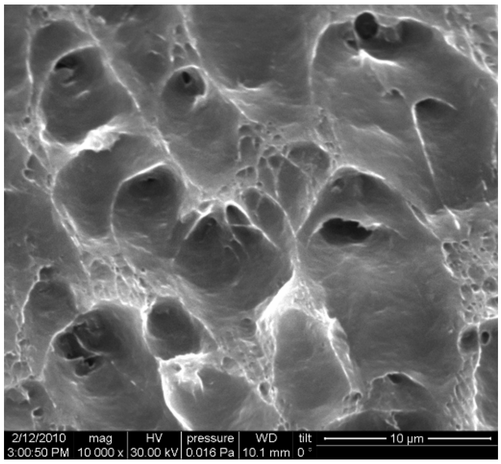

Fig.2. Fracture surface of the A3003 alloy sample

Increase in the number of cycles reduces the size of grains of UFG structure to 350-400 nm, and increases the strength of 200 MPa at the plasticity of 10%. Comparison of the nature of the fracture of deformed specimens with mechanical properties showed their correlation.
The analysis of strain relief on the basis of the New View interferometer- profilometer was carried out in samples with different grain size. It includes the correlation analysis of the micro relief of the fracture surfaces and evaluation of the Hurst index.

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Evolution of microstructure and texture during self-annealing in silver processed by Equal-Channel Angular Pressing

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The stability of the microstructure and texture of pure silver processed by equal channel angular pressing (ECAP) at room temperature was investigated by transmission electron microscopy and X-ray analysis. It is observed that after ECAP an ultrafine-grained microstructure is produced at room temperature but there is self-annealing in the form of recovery and recrystallization during long-term storage at room temperature. Silver has a low stacking fault energy (SFE) and this leads to a high degree of dislocation dissociation and so that recovery by dislocation cross-slip and climb is hindered. It is shown that the microstructures formed after the 1st pass and 4th passes are quite stable during storage at room temperature. Texture evolution also shows a negligible change during storage for samples after 1 pass and 4 passes. For samples with 8 passes and 16 passes, there is a considerable change in the microstructural features as well as texture components. The driving force for the observed changes is correlated with the high degree of dislocation density which develops due to the low SFE and the high degree of dislocation dissociation. It is concluded that these microstructural and textural changes are due to the occurrence of recrystallization during storage of the samples. It is also observed that the degree and kinetics of self-annealing depend upon the number of passes imposed by ECAP.

Two-level structure states features formed in BCC metals and alloys after severe plastic deformation

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Structure attestation results and microhardness parameters measurement data of BCC metals and alloys after torsion in Bridgman anvils subject to deformation degree were generalized. Microstructure investigation and microhardness values measurement were carried out in sections parallel and perpendicular to the anvils plane at different distances from the torsion axis.

When studying the features of nonequilibrium structural states with high continual defects density, special methods of high continuous misorientations analysis, allowing to separate «structural» (inherent in the bulk samples) crystal lattice curvature, were used [1, 2].

High microstructure anisotropy formation in high-strength BCC materials (V-4%Ti-4%Cr (wt. %), V-Mo-ZrO_2, Mo-47%Re (wt. %), Mo-47%Re-0.4%Zr (wt. %), Ta (99.99 %)) after torsion for one turn is determined. Grain sizes in torsion axis direction (perpendicular to the anvils plane) don't exceed 100 nm, while grain sizes in directions parallel to the anvil planes reach 250 – 500 nm. In this case, distinctive parameters for each material are grain and subgrain sizes and elastic-stressed state characteristics.

In investigating materials, the formation of two-level structure states was revealed after reaching true logarithmic deformation values $e > 3$. It occurs in fragmentation of submicron ($50 \div 250$ nm) grains with large-angle boundaries into nanograins ($5 \div 20$ nm) with small-angle misorientation boundaries. Such structures feature is complicated character of highdefect state defined by high crystall lattice curvature values (χ_{ij} is higher than 50 degrees/micron).

The heterogeneous strengthening character of central and peripheral specimen regions was detected in the process of microhardness measurement of all materials, investigated in this work, after one full turn rotation. It was determined, that only after reaching such deformation degrees that are characteristic for each material, microhardness parameters equalizing at different distances from the center occurs, that correlates with two-level structure states development.

Analysis of influence of investigated materials strength characteristics and relaxation ability on formation features of microstructure anisotropy and mechanical properties was conducted. Possible mechanisms of plastic deformation and crystal lattice reorientation in nanocrystalline and submicrocrystalline structure states formation process under severe deformation impact conditions were considered.

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Formation of nanostructure in CP titanium by means of sheet rolling at room and cryogenic temperatures

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Significant increase of strength in commercially pure (CP) titanium can be obtained via formation of nanostructure. High-strength nanostructured CP titanium could be used much wider than its conventional counterpart.

Several methods of severe plastic deformation (SPD) have been developed recently, including equal channel angular pressing, multiaxial deformation and high pressure torsion. Nanostructure in various metals and alloys can be formed by these methods. However use of these methods requires usually special equipments and/or can be rather laborious. Meanwhile large strain can be attained also by conventional deformation schemes, such as rolling. Authors [1] showed that structure with average grain/subgrain size about 150nm can be formed by sheet rolling of CP at room temperature. Development of such method of production of nanostructured sheet products may be very attractive. In addition it is well known that a decrease in deformation temperature considerably decreases the size of grains/subgrains forming during deformation [2]. Therefore it seems interesting to study microstructure evolution in CP titanium during cryogenic rolling as well.

Commercial pure titanium with the average grain size of 15µm was used in this study. Work pieces with dimensions $10\times30\times4$ mm^3 were sheet rolled at 20°C and -196°C. The true thickness strain per one rolling pass was about 0.02. The total strain was 2.6. Structural characterization was performed using an optical microscope Olympus GX-71, a transmission electron microscope JEOL JEM – 2100F, and a scanning electron microscope Quanta 600 FEG equipped with an electron backscattered diffraction analysis device.

Microstructure evolution during rolling at room and cryogenic temperatures was studied with emphasis on mechanical twinning and its influence on microstructure formation. Results obtained by optical, scanning and transmission electron microscopy showed that mechanical twining in CP titanium during rolling at room temperature started at initial stages of deformation and developed up to strain about 20-30 %; further deformation did not result in formation of new twins. Decrease in temperature to -196˚C caused noticeable changes in twinning process. At first, saturation of twinning was shifted to larger strains; formation of new twins was observed at strain ~60%. Second, twinning was found to be more intensive comparing to that at room temperature at the same strain.

The latter fact resulted in higher density and more homogeneous distribution of twins that leads to more intensive intersecting of twin boundaries. After sheet rolling at room (20°C) and at cryogenic (-196°C) temperature to a true thickness strain ~ 2.6 the average grain/subgrain size was found to be 150nm and 40nm, respectively.

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Phase transformations in the Cu-Cr alloys during high pressure torsion

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Severe plastic deformation (SPD) techniques allow considerably increasing strength in alloys and metals due to structure refinement to nanometer sizes [1]. In relation to dispersion-hardened low-alloyed Cu-Cr alloys, strength increase is determined by combination of structure refinement and matrix strengthening due to precipitation of dispersion-hardened (DH) second-phase particles.

The processing scheme of 3 stages is traditionally used for achieving high strength in dispersion-hardened low-alloyed Cu-Cr alloys. The first stage is high-temperature thermal treatment (1050°C) with subsequent quenching in water in order to produce supersaturated solid solution (SS) of alloying elements in the copper matrix. The second stage is cold deformation resulting in the matrix strength increase due to cold work hardening. The third stage is ageing, when solid solution decay takes place and, consequently, additional dispersion hardening of the alloy [2].

If at the second stage of deformation traditional deformation techniques, for example, rolling, drawing are substituted by any SPD technique, then there is a possibility that the nature of solution processes and second-phase particles precipitation in the copper matrix will change.

In [3] it has been demonstrated, when one of SPD techniques, namely equal-channel angular pressing (ECAP) is used to fabricate a homogeneous ultrafine-grained (UFG) structure in Cu-Cr alloys, the decay kinetics of supersaturated solid solution of alloying elements in the copper matrix changes strongly, dispersion-hardened (DH) particles appear during deformation. Probably, these processes are defined by appearance of a great number of defects, first of all vacancies occurring during severe plastic deformation. In their turn the particles also influenced the deformation flow of the material. At the same time second-phase particles dissolve. Such interaction of plastic flow and solution processes and second-phase particles precipitation in the copper matrix has allowed enhancing the margin of dispersion hardening during ageing and receiving high strength values up to 700 MPa.

The whole complex of the observed phenomena, namely change of particle sizes, distance between them and nature of distribution over the matrix, strength margin increase during ageing, demonstrates that SPD introduces sufficient local distortions into the matrix crystalline lattice, which cause non-equilibrium kinetics of phase transformations connected with solid solution decay both during SPD and ageing.

We demonstrate in this work that substitution of cold deformation in the traditional processing scheme of these alloys by high pressure torsion (HPT) (Bridgman anvils) allows enhancing the interaction effect of the above mentioned mechanisms, as the accumulated strain values are much higher during HPT than during ECAP.

It is demonstrated that HPT of dispersion-hardened Cu-Cr alloys has a considerable influence both on the matrix nanostructuring processes and decay kinetics of solid solution of alloying elements in the copper matrix in comparison with the coarse-grained state.

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Metal-insulator transition in semiconductors with magnetic nano-inclusions

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The role of large-scale fluctuations of the electric potential in traditional (*non-magnetic*) doped semiconductors is well known. Such a fluctuating potential appears usually in highly-compensated semiconductors where concentrations of charged impurities (donors and acceptors) are high and the concentration of screening mobile charge carriers is low, that results in a large screening length, defining the spatial scale of electric potential fluctuations. In that case, the average amplitude of the fluctuation potential is also high that leads to the localization of charge carriers and results in the activation character of the system conductivity: it is controlled by the thermal activation of charge carriers from the Fermi level to the percolation level and falls down exponentially with lowering temperature. In the absence of the impurity compensation, the charge carrier concentration is so high that any perturbations of the electrostatic nature are effectively screened, and the spatial scale of the potential coincides with the extent of impurity density fluctuations. The depth of such a short-scale potential relief is relatively shallow and does not lead to the charge carrier localization - the conductivity keeps being metal one.

In diluted (but nevertheless, highly-doped) *magnetic* semiconductors (of $Ga_{1-x}Mn_xAs$ type), in addition to above mentioned fluctuations of the electric potential, the new perturbation source appears - specifically, nanosized fluctuations of the "magnetic potential" concerned with fluctuations of the local magnetization in such a semiconductor. That potential is, in fact, the potential of the exchange interaction of mobile charge carriers with magnetic impurities (for instance, via the RRKKY mechanism) which fluctuates in accordance with fluctuations of the concentration and the local magnetization of those impurities.

Within the "wells" of the magnetic potential, mobile charge carriers with a certain spin direction are accumulated while the carriers of the opposite spin direction are pushed out. The spatial scale l of magnetic fluctuations is now determined not by the electrostatic screening but by the characteristic length of the magnetic interaction of impurities and the correlation length of their arrangement in the semiconductor bulk. However, in diluted magnetic semiconductors, there is usually $l \sim l_s$ and, thus, spatial scales of the magnetic (exchange) and Qoulomb potentials agree closely. That means the constructive superposition of both reliefs, and so the average total amplitude of the potential relief becomes to be higher. The medium arises where the concentration and the spin polarization of charge carriers are strongly non-uniform, and the degree of that non-uniformity is substantially defined by the local magnetization of the system. Increasing magnetization with lowering temperature promotes strengthening the spatial localization of charge carriers and in a number of cases could stimulate the metal-insulator transition. Percolative metal conductivity, characteristic for non-uniform systems, changes into the conductivity of the activation type. That occurs when under some external factors (such as temperature, magnetic field, etc.) the Fermi level falls below the percolation level. One of possible mechanisms is as follows. The fluctuating potential leads to appearing the density of states tail into which both the percolation and Fermi levels are pulled. Rates of those levels' movement are different, and if they change the relative position the metal-insulator transition occurs.

It is just the model that is investigated in the present work.

X-ray diffraction study on the nanostructured NiMn alloy fabricated by planetary ball milling

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Batches weighing 20 g of nickel and manganese commercial powders combined at a ratio of 2:3 were mechanically alloyed using a planetary ball milling instrument operating at a velocity of 400 rpm and ball-to- powder weight ratio of 12:1. X-ray diffraction (XRD) using Cu-Kα radiation was used to study the process of mechanical alloying during ball milling. Figure 1 shows XRD peak profiles obtained for powder mixtures ball milled for 5, 20 and 50 h. Accelerated evolution of the deformed structure of nickel powders was identified in terms of the preceding reduced intensity and broadening of the XRD peak profiles. Delayed evolution of the deformed structure of manganese powders was found alternatively. XRD peak profiles resembling the nanostructured crystalline state and the predominant amorphous state of the ball milled powder mixture were obtained after milling for 20 and 50 h, respectively.

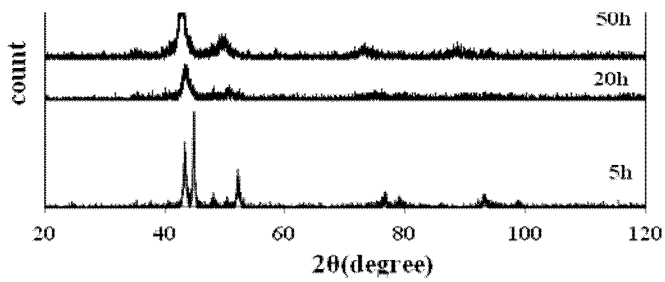

Fig. 1. XRD peak profiles obtained for powder mixtures ball milled for 5, 20 and 50 h

Fracture strength of layered material based on titanium alloy

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Metallic layered materials have received attention due to their striking characteristics [1, 2]. Diffusion bonding is capable of producing qualitative joints between metallic layers, in particular of titanium alloys specifically titanium alloys.

The study of failure behavior of layered materials at different kinds of stressing is actual objective. It is obvious that one of factors influencing on failure behavior of layered material is the arrangement of bonding interfaces relative to an extending crack. However this question isn't studied concerning layered materials from titanium alloy.

The aim of present paper was study of influence of arrangement of bonding interfaces relative to an extending crack on failure behavior of layered material from titanium alloy at shock loading.

The layered material was manufactured by diffusion bonding of microcrystalline (MC) and nanostructured (NS) sheets of Ti-6Al-4V alloy. Diffusion bonding of sheets, assembled in a package and placed in a die with wedge-type stop, was carried out in the vacuum furnace. The samples of two types with dimensions 10×10×55 mm and with U-type notch were used. The bonding interfaces were disposed perpendicularly and parallel to the notch.

In the present paper it is shown that the failure behavior at shock loading of layered material manufactured by diffusion bonding of sheets of titanium alloy depends on arrangement of bonding interfaces relative to an extending crack. The samples of layered material prone to delamination have the high impact toughness.

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The pressure welding of VT6 titanium alloy through ultrafine-grained pad

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The titanium alloys and their welded constructions are widely used in different branches of industry. Pressure welding is one of effective technological methods of obtaining qualitative joint of structural material.

The different methods of quality assessment of solid state joint (SSJ) are apply now. Bending impact test is simple and standardized method, impact toughness is one of most sensitive characteristics of quality SSJ. The aim of this paper is quality assessment solid state joint by means of definition of impact toughness and carrying out the fractography analysis.

The material used for this study was the ($\alpha + \beta$) - titanium Ti-6wt%Al-4wt%V alloy. It was used in the form of Ø 40 mm rod with microcrystalline structure and sheets 1 mm thick in micro- and nanostructured (MC and NS) states: 1 - average grain size of 3-5 μm; 2 - average grain size of 1.2 μm; 3 - average grain (fragment) size of 0.5 μm; 4 - average grain (fragment) size of 0.2 μm. States 1 and 2 were produced VSMPO, Verhnyaya Salda, Russia. State 3 was processed by multiple step forging [1], state 4 - multiple step forging and subsequent isothermal rolling [2]. Four types of welded specimens were investigated. The welded specimens (fig. 1a) was manufactured by pressure welding of two cylindrical specimens through pad in the state 1 (A type), 2 (B type), 3 (C type), 4 (D type).

The standard specimens with dimensions 10×10×55 mm with U-type notch and radius of concentrator R=1mm were used for definition impact toughness. The notch was disposed along SSJ (fig. 1b).

The zone of SSJ of all welded specimens contains isolated pores, size and amount of pores is equally. The welded specimens of type A have the highest impact toughness, the specimens of type D have the lowest impact toughness, the specimens of type B and C have the equal level of properties. The results of fractography analysis coordinate with results of mechanical tests.

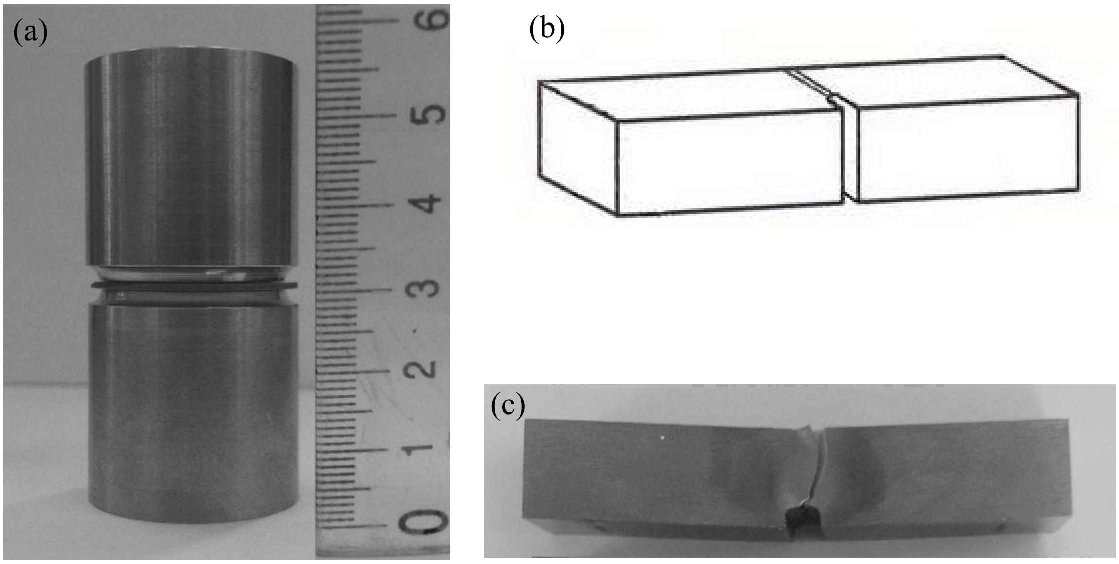

Fig. 1. (a) – welding specimen, (b) – design of specimen for impact tests, (c) – form of fractured specimen

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Microstructural evolution in an Al-Cu-Mg-Ag alloy during ECAP at 250°C

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Microstructural evolution in a 0.17%Sc and 0.12%Zr modified age-hardenable Al-Cu-Mg-Ag alloy was examined under equal channel angular pressing (ECAP) with a back pressure by orientation image microscopy (OIM). The samples were initially subjected to solution treatment followed by water quenching. Next, these samples were overaged at 250°C for 5 h to produce a dispersion of Ω-phase. ECAP was carried out at 250°C using route B_C. Samples were strained to total strains of 1, 2, 4, 8 and 12. It was shown that a fully recrystallized structure is evolved after a total strain ε~12. Particle stimulated nucleation (PSN) plays an important role in extensive grain refinement under ECAP. Features of grain refinement mechanism associated with PSN are considered.

Grain boundary sliding in submicrocrystalline titanium at room temperature

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Grain-boundary sliding (GBS) is one of the mechanisms of deformation of metals and alloys. This mechanism of deformation is caused by mutual displacement of adjacent grains on the surface of the boundary separating them. Generally, this mechanism is typical for the high homologous temperature (T> 0,5 Tm). Recently, however, it has been shown experimentally that in nanostructured (NS) and submicrocrystalline (SMC) materials obtained by severe plastic deformation GBS is developed at lower temperatures (T ~ 0.2-0.3Tpl) - close to room or even at room temperature. There is a change of mechanisms of plastic flow with the grain size decreasing: from dislocation slip to the mechanisms which are controlled by diffusion - diffusion creep and grain-boundary sliding. These mechanisms of deformation in the SMC and NS metals and alloys are competing with each other. The dominance of one or another mechanism is determined by the grain size.

In this paper the VT1-0 titanium alloy (Ti – base, C – 0,004; N – 0,003; Fe – 0,12; O – 0,143; H – 0,0008; Al – 0,01; Si – 0,002) was investigated in submicrocrystalline state obtained by severe plastic deformation method that combines longitudinal and helical rolling. It is shown that GBS can be realized at room temperature under uniaxial tension, which is realized in the form of cooperative GBS. Grain-boundary sliding was found according to the shift and the break of the scratches pre-deposited on the surface. Areas in which the break of scratches was observed represent the mesoscopic bands of the length from about 0.5 to 2 microns. The thickness of these mesoscopic bands corresponds to the thickness of the grain boundary. In the study of the sample surface it was found that the bands of cooperative GBS were distributed uniformly along the entire working part of the specimen and intersect the all cross section. Herewith, they break off and re-appear at a distance of up to several tens of microns along each line. These mesoscopic bands are located at 45° to the direction of strain, that corresponds to the maximum value of shear stress. The above description fully corresponds to the typical fringe patterns of cooperative grain boundary sliding, well-known in the literature. In the study of the cross section it was found that the presence of a step for the band of cooperative GBS is typical. The height of this step corresponds to the distance of relative displacement of scratches during their break. From a distance of the break of scratches it was found that the contribution of cooperative grain boundary sliding to total deformation may take up to 10%.

Characterization of oxide film in pure iron by nanoindentation

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The metal and alloy products widely apply in modern engineering. Their surfaces are covered by oxide films in environmental conditions. That is why operating abilities, for example corrosion stability, depend on capability of surface oxide films to protect metal against damages. Mechanical properties of oxide films on the Armco iron were investigated by nanoindentation. Nanoindentation is now proven to be a powerful method for elucidating mechanical properties at nanoscale level, comparable to the mean grain size of materials. Nanoindentation with a three-sided pyramid (Berkovich) indenter was conducted on a commercial instrument Hysitron TriboIndenter TI 900. This equipment is ideal for nanoindentation (measure Young's modulus, hardness, fracture toughness and other mechanical properties) and scratch testing (quantify scratch resistance, critical delamination forces, friction coefficients and more with simultaneous normal and lateral force and displacement monitoring). The iron specimens were annealed with different temperatures and curing times, after that the indentation curves were constructed. The surface topography and indent SPM images were got with atomic-force microscope. The results of the present nanoindentation experiments showed that mechanical properties of Armco iron oxide films depend on temperature, environment, gaseous atmospheres, surface preparation and change with the course of time.

The research was supported by Russian Fund of Fundamental Research Grant "RFFI-URAL" № 10-08-96050 and Program with Institute of applied and theoretical mechanics SR RAN 09-C-1-1003.

Influence of thermal cycling on micro hardness and microstructure of NC TiNi alloys

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It is known that thermal cycling allows increasing the dislocation density (work hardening) in initially coarse-grained TiNi alloys – multiple cooling and heating of alloys are above and below the points of martensitic transformations, as each cycle of martensitic transformation results in accumulation of residual dislocation in material. It is of great interest whether it is possible to increase dislocation density in nanocrystalline TiNi alloys by means of thermo cycling. The investigations have been carried out on the $TiNi_{50.6}$ alloy with the temperature of the martensitic transformation B2 → B19' (Ms) of about 15°C. The amorphized $TiNi_{50.6}$ alloy's samples have been processed by HPT at room temperature. Subsequent annealings at the temperature of 400 °C have resulted in formation of NC structure with the grain size of about 20 nm [1]. Samples in the initial coarse-grained (CG), amorphized and NC conditions have been subjected to thermal cycling with the number of heating-cooling cycles n =20. Cycles of thermal cycling have been performed by cooling down to the temperature of liquid nitrogen T_N (-196 °C) and by heating up to 150°C, these values which are consciously below and above the temperatures of transformation in the CG alloy. The microhardness Hv of samples has been measured in the initial condition after thermal cycling with final cooling to -196°C (Table 1 – «thermal cycling»), when the residual martensite may remain in the material, and after final heating up to 150 °C, (Table 1 – «thermal cycling + heating to 150 °C »), when the material transfers into the austenitic state.

The microhardness of CG alloy has increased slightly as a result of thermal cycling (Table 1). However, the subsequent final heating of the alloy to the temperature of 150 °C and its transformation into the austenitic state have resulted in Hv decrease to the former level. The H_v of amorphized alloy has not practically changed during thermal cycling. It is quite reasonable because martensitic transformations do not occur in amorphous material. At the same time there is a considerable increase of Hv in NC alloy after thermal cycling (Table1). The alloy's grain size makes approximately 20 nm after HPT and annealing at the temperature of 400 °C. The grain size of NC alloy has not considerably changed after thermal cycling. However, obviously, in nanograins dislocations and twins have been formed, (Fig. 1).

Table 1 Dependence of microhardness Hv of CG, amorphized and NC $TiNi_{50.2}$ alloy on thermal cycling process (n=20)

State		initial	Thermal cycling	Thermal cycling + heating to 150 °C
Coarse-grained		240	275	245
Amorphized by HPT	Hv	615	625	
NC		430	535	470

Thus, thermal cycling also causes formation and accumulation of dislocations in nanograins in the NC TiNi alloys with grain size about 20 nm. Nevertheless, as it is known the transformation B2 $\rightarrow$ B19' in the TiNi alloys with NC structure (with D<= 20 nm) is blocked if the alloy is cooled to T_N. Probably the dislocations in NC $TiNi_{50.6}$ alloy is formed by means of transformation B2 $\rightarrow$ R $\rightarrow$ B2 (which may be in NC TiNi alloys with the grain size of about 20 nm). This question requires further investigations.

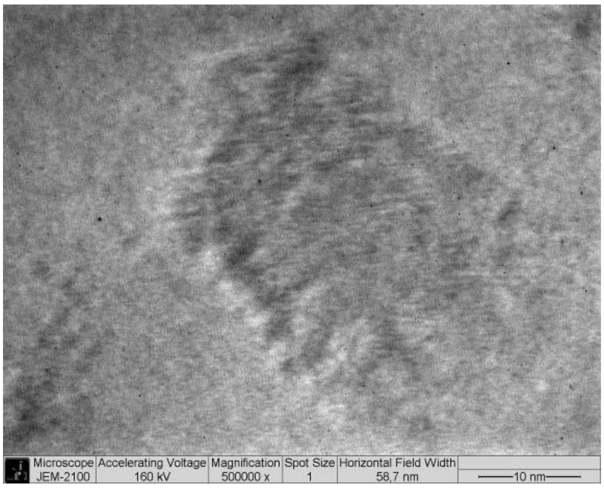

Fig. 1 Microstructure, HPT TiNi50.6 after annealing at the temperature of 400 °C for 1 hour and after thermal cycling process n=20, TEM

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Deformation and cold resistance of ultrafine-grained steels

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The questions of using of traditional thermomechanical treatment methods of metallic materials and severe plastic deformation methods with thermal treatment combination are examined. Comparation of results obtained by thoes methods is presented.

Data of mechanical properties and impact toughness of steels subjected by mechanical (forging and equal channel angular pressing) and thermal treatment are presented.

Results of energy dissipation during the process of plastic deformation of steel in different structural stage are presented. For the evoluation of storage energy of steel during the process of material plastic flow is applicated special calculation-experimential technic [1].

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Structural and phase transformations during the severe plastic deformation and heat treatment of steels

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The issues of studying the structure, mechanical properties and phase state of nickel alloy in the initial state and after equal-channel angular pressing are discussed. The equal-channel angular pressing was performed in 14 passes on the route C at 623 K.

The data on the grain size, microhardness, characteristics of strength and ductility, as well as the phase composition of nickel alloy are presented.

Fractographic study of the microstructure were performed on raster electron microscope „XL-20 Philips", the mechanical properties were determined by mechanical tests on the machine „Instron 1195", X-ray studies were made on X-ray diffractometer „DRON-3M".

Atomistic simulation of order-disorder phase transitions in binary alloys driven by vacancy diffusion mechanism

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For the alloys of stoicheometry AB based on fcc and bcc lattices we study the influence of pair interatomic energies $\varphi_{AA}^{(i)}$, $\varphi_{BB}^{(i)}$, $\varphi_{AB}^{(i)}$ on the ordering kinetics at fixed ordering energies, $\omega_i = \varphi_{AA}^{(i)} + \varphi_{BB}^{(i)} - 2\varphi_{AB}^{(i)}$, where i is the number of coordination sphere. Ordering occurs by the vacancy diffusion mechanism. Probability for an atom to occupy the vacant lattice point depends on the energy difference associated with this process and on temperature. The computational model and basic relations are presented for interatomic interactions in arbitrary number of coordination shells. Examples are given for interactions in the two first coordination shells. It is demonstrated that the speed of ordering is maximal when φ_{AA}^{i} are equal to φ_{BB}^{i}, and it decreases when the difference between φ_{AA}^{i} and φ_{BB}^{i} increases. This conclusion is true for both fcc and bcc lattices. Our results contribute to the understanding of how the energies of pair interatomic interactions influence the ordering kinetics of alloys.

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Gradient microstructure in Fe-Cr-Co system hard magnetic alloy subjected to tension combined with torsion

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The development of new very-high-speed electrical machines in recent times requires high strength characteristics from the magnetic materials used. Most magnetic materials of today possess high magnetic characteristics but are brittle and have low ultimate rupture strength. The highest level of mechanical properties in magnetically hard materials is realized in commercial alloys of the Fe – Cr – Co system.

Complex loading by compression, stretching, and torsion at an elevated temperature is rather new method of severe plastic deformation. It ensures a substantially refined structure without changing the shape of the specimen. Depending on the mode of the deformation chosen, this method makes it possible to localize strain in specific regions of the perform and ensures formation of a gradient structure with different combinations of magnetic and mechanical properties in different regions.

The aim of the present work consisted in studying the evolution of the structure and microhardness of Fe-30Cr-8Co hard magnetic alloy during complex two-stage tension–torsion under isothermal conditions at different temperatures. The generated structure is non-uniform through the body of the samples and has gradient character which is coarse grained in the center and ultrafine grained in the peripheral part. Torsion with tension of alloy Fe-30Cr-8Co with fixed grips enables fabrication of cylindrical specimens with gradient structure. This in turn causes plasticization the surface layers of the specimens for subsequent drawing in the preliminarily aged state in order to obtain a strong uniaxial texture ensuring maximum magnetic properties in the alloy.

The microstructure of the Ni-Al-V alloys prepared by levitation, rapid quenching and high pressure torsion

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The Ni-based intermetalic phases like Ni_3Al or NiAl reveal many interesting properties as constructive materials, however commonly exhibit also high brittleness at room temperature. The NiAlV alloys belonging to the pseudo-binary, Ni_3Al-Ni_3V cross-section through the ternary phase diagram posses at room temperature characteristic microstructure resulting from the eutectoidal decomposition below 1000°C, which leads to the Ni_3Al–Ni_3V phase composition. The structures of these phases belong to the highly ordered dens packed L12 and D022 structures, with very high degree of coherence at the interfaces. Also, mechanical properties of such alloys investigated in the creep tests are promising. The path of the microstructure formation in eutectoidal decomposition as well as the influence of the prolonged annealing, which leads to the lamellar microstructure remain not completely clear.

The report presents results of investigation of three NiAlV alloys, of composition belonging to the Ni_3Al-Ni_3V pseudo-binary cross-section. The samples were prepared by the cold crucible levitation melting (CCLM) and by re-melting and crystallizing in the small volume copper mould. The phase composition of the samples, which should result from the eutectoidal decomposition was not found. Instead the $Ni_3(Al,V)$ and Ni(Al,V) solid solution or seldom disordered solid solution were retained due to the relatively high cooling rates. The samples after high pressure torsion revealed much smaller size than after rapid quenching while the phase composition remained similar to that in the case of rapidly quenched samples.

Investigation of the deformation mechanisms in 2024 Al-alloy during ECAP

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Severe plastic deformation is a promising technique for enhancement of mechanical properties via grain refinement as a result of high shear strains [1]. Being preferential and advantageous over other methods in many aspects, ECAP (Equal Channel Angular Pressing) has high potential for commercialization due to its simplicity and applicability to various materials and various sizes, and not causing any significant dimensional change [2]. Although the procedure is simple, it involves complex deformation mechanisms related to boundaries, dislocations and precipitates which need clarification in order to maintain stability and maximum efficiency [3]. This study covers the HREM, RXD and nano-indentation investigations of three sample groups of 2024 Al-alloy: solutionized, solutionized-ECAPed, solutionized-ECAPed-aged. The research was focused on understanding of dislocation-boundary interactions, boundary characteristics of the cells, dislocation distribution in the absence and presence of dispersoids, and the interactions among dislocation-boundary-dispersoid.

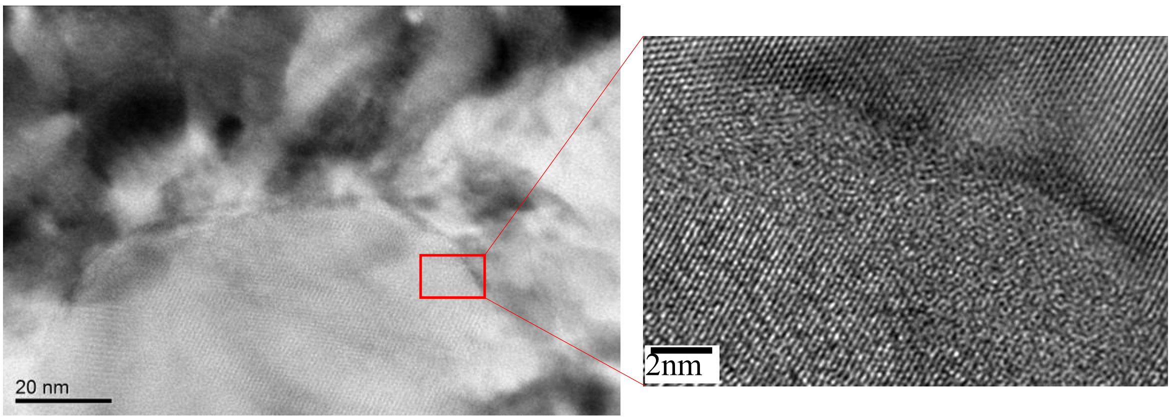

Fig. 1. TEM micrograph of ECAPed-aged 2024 Al-alloy

The figure shows a TEM image of solutionized-ECAPed 2024 Al-alloy containing T-phase dispersoid. A boundary region of 4-5 nm thickness could be observed between dispersoid and ECAPed Al matrix.

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Cooperative grain boundary sliding at high strains in UFG and NC Pd Alloys

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A major impediment to studies of the deformation of nanostructured metals is their typically very limited ductility in tension, which restricts the experimentally accessible parameter space. Torsion testing under high pressure offers an important opportunity, since it allows practically unlimited shear strain without fracture. This is especially important for brittle samples like nanocrystalline (nc) materials with a grain size less than 30 nm. Here, we present an overview of the recent results of mechanical behaviour and microstructure development in nc Pd and its alloys at severe plastic deformation by instrumented high pressure torsion up to shear strain 300. We have studied microstructures formed in initially coarse crystalline and nc Pd and its alloys, and revealed signatures of similar processes occurring in all these materials. In particular, we found traces of cooperative grain boundary sliding in the form of aligned in parallel segments of boundaries of several grains with straightened triple points. Fracture surfaces contained shear bands. Texture measurements revealed lower dislocation activity in nanocrystalline state as compared with coarse crystalline one. Therefore we argue that cooperative grain boundary sliding is an important deformation mechanism at large strain which develops in both ultrafine grained (ufg) and nanocrystalline materials. In nc and ufg materials planes of cooperative grain boundary sliding act as precursors of shear bands and shear occurs along planes formed by numerous grain boundaries.

On tensile strength of cryorolled commercial heat hardenable aluminum alloy with multilevel nanostructure

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Developing new methods for strengthening of metallic materials has caused appreciable interest to severe plastic deformation (SPD) at low homological temperatures. Such processing of pure metals and solid solutions usually forms well-developed deformation structure with features of nanocrystalline one of fragment type, accompanied by a material unique strength at a room temperature. The latter is caused by structural hardening predominantly due to two factors - strong (sub) grain refinement and high densities of lattice dislocations. It was of great interest to evaluate the potential of such processing for highly alloyed precipitation-hardened material, to analyze the combining effect of low temperature SPD and heat treatment resulting in the alloy structural and dispersional strengthening.

Plates of commercial aluminum alloy D16 of 5 mm in thick cut from conventional hot pressed rod, were preliminary quenched and then rolled to strains up to e = 3.5 in isothermal conditions at a temperature of liquid nitrogen. After that, the rolled samples were artificially aged in a wide temperature-time range. Dislocation and grain structure before and after cryorolling were studied by optical and transmission electron microscopy (OM and TEM), and X-ray diffraction (XRD) analysis. Parameters of the alloy tensile strength at a room temperature were measured on samples with a gage part of 1x5x15 mm. Microhardness (Hv) was estimated by standard procedure.

OM and TEM have shown that cryorolling does not qualitatively change the type of the alloy structure of the matrix remaining coarse-fibered after all strains investigated. Inside the fibers, instead of hot-pressed substructure the cellular structure with high dislocation density was developed. Strain increase from 0.9 to 3.5 did not result in decrease in cell size stabilizing at a value of ~100-200 nm. Meanwhile, TEM images from their boundaries became sharper and of less width, causing increase in cell misorientations. However, only insignificant fraction of boundaries demonstrated a contrast typical for non-equilibrium boundaries of nanocrystalline materials produced by "cold" SPD. As a result, rolling to maximum strain has formed separate nanograins with size less than 100 nm, which volume fraction did not exceed 10%, testifying an initial stage of nanocrystalline structure formation only.

XRD analysis has shown reduction to 50 nm in the alloy coherent domain size (CDS), similar to the cell size changes with strain, i.e. the CDS reached its minimum at the strain of 0.9 and did not changed with further deformation. On the contrary, microstrain of matrix firstly increased from 0.07 to 0.21 at e= 0.9, and then slightly decreased to 0.17%, testifying transformations in cellular structure leading to increase in cell boundary misorientations.

Mechanical tests of the cryorolled with e~2 state have been revealed the alloy ultrahigh-strength (YS = 630 MPa, and UTS = 645 MPa) and hardness (~180 Hv), but low plasticity (El ≤ 3 %). Thus, strength parameters of the as-processed alloy exceed their levels in conventionally T6 and T8 processed sheets. Subsequent annealing under conventional T6 artificial aging conditions led to rather enhanced alloy plasticity. However, it was accompanied by strength decrease to conventional values. It is also shown, that aging by correct regimes provides much higher both the alloy strength and plasticity. This effect is reasoned by less softening of deformed material under recovery and continuous recrystallization, and by higher simultaneous dispersional strengthening. As a result, the alloy with unique balance of room temperature mechanical properties, causing by formation of multilevel nanostructure – mixed nanoprecipitation strengthened nano(sub)grain one (i.e. mixed nano(sub)grain structure strengthened by mixed nanoprecipitates), has been obtained.

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Physical and chemical properties of large-dimensional nano- and ultra structured ceramic-composite materials on the basis of a secondary silicon waste

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It is known that in metallurgical, chemical and other industries a secondary waste is produced in the course of a manufacture which is in the form of multiton metal waste are formed.

The world problem of economic metal waste recycling is solved in a greater degree for colour and black metallurgical manufacture, and partially for mechanical engineering and construction engineering, but practically isn't solved for silicon manufacture.

In the course of manufacture of silicon plates is lost about 40-45 % (masses) of expensive initial single-crystal silicon in the form of the various silicon waste, formed in the process of calibration grinding, polishing of silicon plates. This waste at worst is thrown out in a sailing, and in the best is stored in special separate adjoining territories of the enterprises, thus polluting an environment [1; 2].

The National Academy of Sciences of the Kyrghyz Republic at Institute of physicotechnical problems and materials technology develops the technology of reception of large volume nano – and ultrastructured ceramic-composite materials (NUCCM) which structure is presented on Fig. 1.

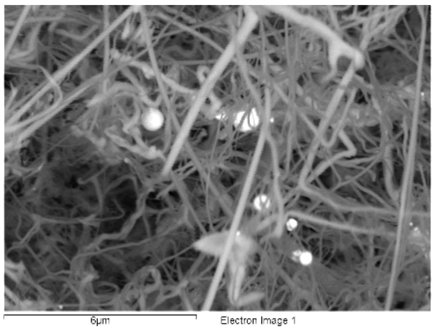
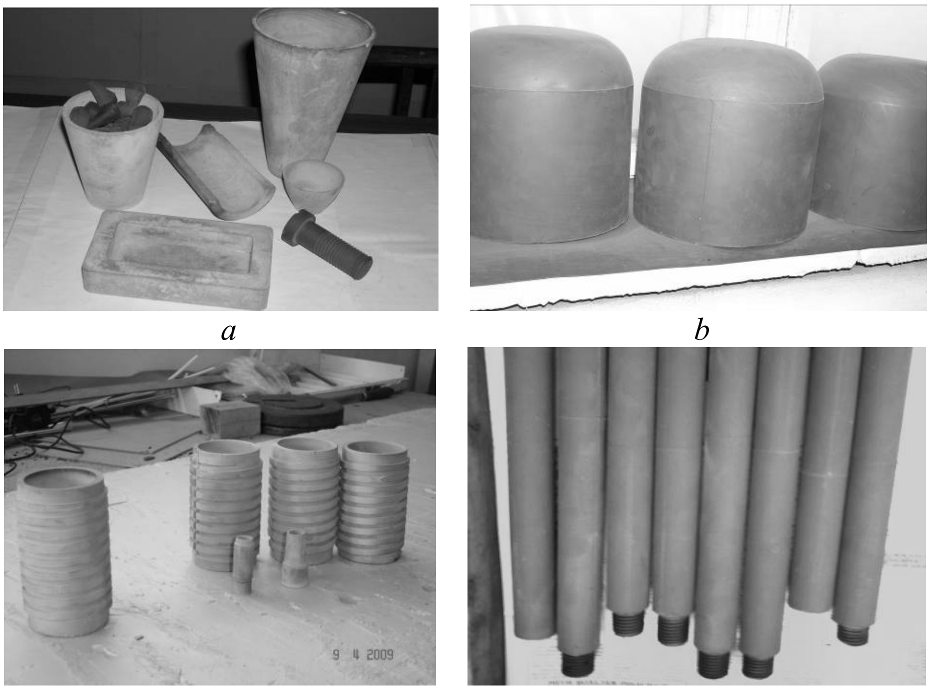


Fig. 1. Threadlike crystals in ceramic-composite nitridesilicon material

Given NUCCM materials, on the basis of nitride and Si carbonitride are 2.5 times cheaper in comparison to nitridesilicon materials, which are received in the traditional-classical ways. Reactionary-synthesised NUCCM (Fig. 2.) have high physical and chemical properties: durability, hardness and thermal stability, low relative density, etc., this is thanks to a formation of synthesis of two types of isomorphic phases in a form of nano - and ultrathreadlike crystals ß-Si_3N_4 and unequal ß′-$Si_3(C_xN_y)_4$ in structure of NUCCM in a process [3,4].

In the present report results on measurement it low-temperature thermal capacities of NUCCM will be presented and discussed in comparison to thermal capacities of a classical ceramic material on the basis of nitride of silicon and initial monosilicon. Along with its results on a research of physical and chemical properties, influence of various acids, alkalis and other specific environments on synthesised NUCCM with nano - and ultradisperse structure will be presented and considered.



a *b*

c *d*

Fig. 2. The large-volume nanostructures ceramic-composite materials:
(a) Highly temperature bolts and other products;
(b) Large dimensional crucible for monosilicon cultivation;
(c) Ceramic glasses for fusion of gold;
(d) Tubular covers for thermocouples.

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Generation of non-equilibrium point defects and anomalous diffusion in nanostructures of metals and alloys during mechanical alloying

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Processes of structural changes which account for the generation of non-equilibrium point defects and anomalously fast diffusion of interstitial impurities in nanostructured metals and alloy during plastic deformation are studied. Extra vacancies generated by intercrystalline boundaries due to their migration are shown to be the main cause of the diffusional mass transport enhancement. Non-equilibrium vacancy concentration profiles are calculated for nanostructures with grain size 5–100 nm under mechanical alloying conditions. It is shown that in materials with grain size larger than 100nm the diffusion enhancement occurs only in the regions near the grain boundaries, whereas at grain sizes smaller than 10 nm the enhancement is possible even in the grains bulk. The effect of interstitial atoms on the diffusion and processes of recrystallization is negligible.

On the theory of grain-boundary diffusion in nanostructured materials produced by SPD

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Diffusion properties of nano- and ultra-fine grained materials produced by severe plastic deformation are commonly related to non-equilibrium grain boundaries. This study presents a theory of grain-boundary diffusion for the case when bulk diffusion coefficient varies strongly near the structural boundary of the grain, which was modeled by as a system of chaotically distributed dislocations. The diffusion conditions that corresponded to regime "C" in conventional polycrystalline materials (when diffusion path of intergranular bulk diffusion is much smaller than boundary thickness) were analyzed using an asymptotic method of solving diffusion equations and a Laplace transform. The analysis of the layer activity measured in the diffusion experiment was performed on copper example and a significant difference of concentration profiles from the case of equilibrium grain boundaries was found. Results of numerical calculations and qualitative estimates are given, which make it possible to establish the field of the applicability of the approach suggested.

Effect of rolling scheme on structure and properties of copper sheets

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Formation of required microstructure in copper by termomechanical treatment is a promising way to achieve considerable improvement in its structural properties [1]. Grain refinement by severe plastic deformation methods like multi axial forging and ECAP lead to increase of strength in copper in 1.5-2 times and makes usage of this material highly perspective [2,3]. Technological limits of those processes don't allow producing work pieces, for example sheets, which would be required by final consumer. The flat rolling should be used for manufacturing of such work pieces. However, absence of any information about the effect of rolling order on microstructure and mechanical properties of copper sheet billet causes relevance in carrying out research in this direction.

In present work the fine grained copper billet rolled with total reduction 60% was examined. The rolling schemes were: 1. Without any changes of rolling direction; 2. Reverse rolling; 3. One time change of rolling direction after reduction 30%; 4. Changes the rolling direction by rotation at 90° after each pass of rolling.

The strain-state analysis in considered point of work pieces was completed. It was shown that through the changes of rolling direction the impositions of hydrostatic pressure and maximum strain rates succession are varied. That means that the at first pass the values of hydrostatic pressure changed from -1 to -1.5 and after rotation of rolling direction by 180° they changed from -1.5 to -1. The strain rates had the same order of magnitude and varied from 3.5 to 1.5 s^{-1}.

The microstructure analysis was carried out. The magnitude of average grain size lead in range from 1.04 to 2.04 μm and depended on rolling schemes. The volume fractions of high angular boundaries were in range from 38 to 64%.

The mechanical properties of produced copper sheets were estimated. The ultimate tensile stresses were in range from 357 MPa to 377MPa, the values of reduction in area were from 25% to 31% depending on rolling scheme.

The effect of rolling scheme on structure and mechanical properties was discussed.

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Effect of further deformation treatment on structure and properties of nanostructured nickel thin strip

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The effect of severe plastic deformation (SPD) processes on microstructure and mechanical properties of metals and alloys have been thoroughly considered in recent research. The heightened interest for this issue is caused by considerable changes of structural properties those materials due to grains refinement and high angular boundaries formation during plastic deformation. The features of nanostructure formation in Nickel by SPD are studied in detail research [1,2]. It was shown that the uniform nanostructure state is formed by those methods. However the limitations of work piece dimensions don't permit to use it in as received shape. The solution of this problem could be usage of following deformation of nanostructure materials to obtain required shape [3]. One of the widely used types of the material is thin strip. Upsetting and rolling should be applied for its manufacturing. Though, the effect of following forming on structure and properties of nanostructure nickel strip isn't still clearly.

Initial nickel rod had average grain size more than 500μm. It was subjected to ECAP at temperature 250°C for 12 passes by route B_C. A work piece with diameter 39 mm and length 180 mm was obtained. It was cut on two equal parts, machined to get rid of defects and upset to 15 mm in height. Strain corresponding to upsetting was equal to 62.5%. After opposite sides machining of work piece its height was 13 mm. From this work piece the strip with thickness 0.3 mm was produced by flat rolling.

After ECAP structure consisted from wide and thin elongated grains with average width 1.8 μm and 0.34 μm correspondingly. The volume fraction of HABs was 60%. Structure became more homogenous after cold upsetting. Average width of wide grains was about 1.5 μm. Width of the thin ones was equal to previous value. Low angle grain boundaries were found inside most wide grains. The number of HAB decreased to 50%. After cold rolling with strain 98% elongated grains with uniform width 0.09 μm were found. HABs fraction was 48%. The relationship between microstructure features and accumulate plastic strain would be discussed.

The ultimate tensile stress of nickel after 12 passes of ECAP was 1005 MPa[4]. The following cold deformation increased this value to 1275 MPa. Increase of strength resulted in elongation decrease from 4.2 to 3.25%. Strength anisotropy of strip didn't exceed 15%. Dependence of microhardness value on annealing temperature showed that the softening processes took place after annealing at 300°C.

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Defect structure features of nanocrystalline Ta obtained by high pressure torsion

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Electron microscopy investigation of pure Ta (99.99 %) microstructure features after high pressure torsion at room temperature up to various deformation degrees was conducted. The investigation was carried out in section perpendicular to the anvils plane at different distances from the torsion axis. Grain and defect structure quantitative parameters were determined with the use of special methods of dark-field analysis of discrete and continuous misorientations [1, 2].

The investigation process showed, that on the background of strong grain structure anisotropy, formed during torsion under pressure deformation, periodic striped or spotted diffraction contrast is observed. To determine its origin nature, detailed investigation of defect structure features of nanodimensional (3-10 nm) areas was carried out.

It was found, that in pure Ta after reaching certain plastic deformation values begins an intensive formation of two-level structure states, which are similar to those previously observed in high-strength alloys based on Mo-Re and V [3]. The analysis of such structural states formation features related to plastic deformation degree was conducted. Corresponding microstructure parameters were quantified. On the basis of crystal lattice curvature experimental data, the analysis of values of the local internal stresses and their gradients at nanoscale level was carried out.

Measurements of microhardness H_v subject to deformation degree and distance from the torsion axis were carried out in sections, perpendicular to the anvils plane. It was shown, that H_v value increases several times during the deformation process. Besides that, heterogeneous strengthening character of central and peripheral specimen regions was detected at initial deformation stage. With further deformation, gradual equalizing of H_v parameters and their saturation occurs. It was found, that such H_v values change is accompanied with initiation and development of two-level structure states.

Possible mechanisms of pure Ta plastic deformation that provides high-defect structure states formation at different stages of deformation impact were considered. Generalization and comparison of experimental results with earlier data relevant to microstructure parameters of high-strength BCC materials after torsion in Bridgman anvils [3] was conducted. Initial strengthening mode influence on the parameters of high-defect structure states forming in severe deformation process in BCC metals and alloys was analyzed.

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Mechanical properties of an Al-5.4%Mg-0.5%Mn-0.1%Zr alloy processed by ECAP at elevated temperature

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Mechanical properties of a commercial Al-5.4%Mg-0.5%Mn-0.1%Zr alloy subjected to equal-channel angular pressing (ECAP) with back pressure at 300 °C using route B_C up to a total true strain of about 12 lead to the formation of uniform recrystallized structure with an average grain size of about 0.7 μm. Mechanical properties of this material was studied. It was shown that ECAP provided a +30% increase in yield stress; ductility remains at sufficiently high level. Contribution of structure hardening, strain hardening and dispersoid hardening into overall increment in strength is considered.

Influence of deformation and heat temperature on structure and mechanical properties of titanium alloy TI-6AL-4V

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Titanium alloys are widely used as structural materials in many industries: from aerospace to medicine. This is justified by the presence of the following properties: low weight, high strength, corrosion resistance in many aggressive environments, manufacturability and bondability. For titanium alloy VT6 (Ti-6Al-4V) exist the technology of hardening heat treatment, which consists in successive operations quenching and aging. Currently, the efforts of researchers both theoretically and practically justified the use of severe plastic deformation (SPD) in connection with the possibility of obtaining SPD methods ultrafine-grained states in the traditional metals and alloys including titanium alloys. Materials with ultrafine structure possess a unique combination of properties

Different types of the deformation and heat treatment of alloy VT6 lead to obtain samples with different microstructures as the morphology of phases and the size of structural components: heat hardening treatment led to the formation of the alloy VT6 lamellar structure: after quenching (1010°C) and aging was formed coarse-grained structure consisting of β-grain size of 260 microns, which is located inside the plate α-and β-phases, after quenching (940°C) and aging was formed microcrystalline structure with α-phase plates with a length of 13 microns and a width of 5 microns; severe plastic deformation led to the formation of the alloy VT6 globular structure: after equal-channel angular pressing (ECAP) formed structure with the size of α-grains 5 microns and dispersed with a mixture of α-and β-phase size 0.5 microns, after multiple step forging formed a homogeneous microstructure with size microfragments α-and β-phases 0.3 microns.

Strength properties of alloy VT6 with lamellar microcrystalline structure at 100 MPa lower than those of coarse-grained alloy (Table 1). The mechanical properties of specimens of VT6 after hardening heat treatment on the same level with the properties of the samples after ECAP, however, the plasticity of alloy VT6 after ECAP is 2 times higher plasticity, heat-strengthened alloy. Multiple step forging led to high strength (1185MPa) and plasticity (δ = 26%). Globular structure of alloy VT6, which was formed of ECAP and multiple steps forging, provides the best combination of strength and ductility compared with the lamellar structure.

Fractures of samples after ECAP and multiple step forging predominantly viscous. Fractures of samples alloy VT6 after hardening heat treatment of fragility. Fragility fractures of alloy VT6 after hardening heat treatment can be associated with lamellar structure, which was formed as a result of quenching and aging.

Table 1. Mechanical properties of Ti-6Al-4V alloy.

Type of processing	σ_B, MPa	$\sigma_{0,2}$, MPa	δ,%	ψ, %
Quenching (1010°C) + aging (500°C)	1037	992	8	10
Quenching (940°C) + aging (500°C)	951	854	10	12
ECAP	1043	1025	16	46
Multiple step forging	1185	1134	26	50

Effect of precipitates on nanostructuring and tensile strength of severely deformed 1965 aluminum alloy

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High-strength 1965 type aluminum alloy with scandium and zirconium additions (Al 8Zn 2.5Mg 2Cu 0.1Zr 0.27Sc) was subjected to severe plastic deformation (SPD) by high-pressure torsion (10 rotations at pressure of 6GPa) at a room temperature. Before SPD the homogenized ingot was solution treated and water quenched (AQ) and then aged (AA) at 170 ^{0}C from 1 to 10 hours. In distinction with as-quenched state with unimodal distribution by size of precipitates of secondary coherent Al(Zr,Sc) phases, the AA alloy has bimodal distribution due to precipitation of strengthening η-phase in addition to mentioned aluminides of transition metals.

TEM and X-ray analysis of the SPD-ed alloy conditions indicate the formation of nanocrystalline (NC) structure with a mean (sub)grain size of ~80 nm in AQ alloy only (Figure).

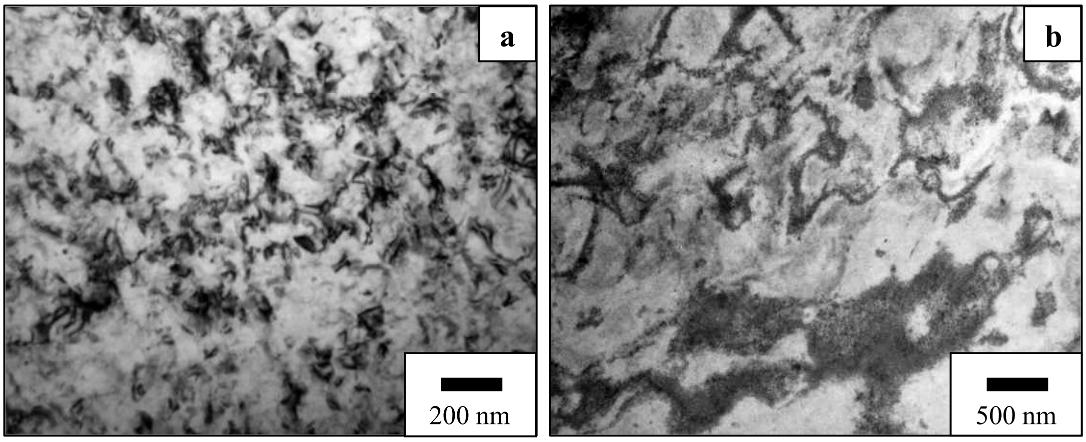

Fig. TEM structure of severely deformed (HPT 10 rot., P=6 GPa, T=20 ^{0}C) alloy in initially quenched (a) and aged (b) conditions

Independent of time of aging the AA alloy structure was highly work-hardened containing high densities of regularly distributed dislocations. No any processes of deformation nanostructuring due to recrystallization, polygonization or cell formation were found, as no crystal boundaries were TEM visible. Moreover, no dissolution of η-phase precipitates was also detectable by both the methods used for analysis.

The initial alloy state significantly affects the mechanical behavior of SPD processed alloy (Table). In case of prior-quenching the level of parameters of the alloy strength and plasticity under tension

(YS, UTS and El.) and hardness are quite common for HPT processed nanostructured heat hardenable aluminum alloys [1, 2]. Meanwhile, the preliminary aged alloy demonstrates comparatively low strength parameters accompanied by high alloy elongation to failure. Such a difference means the direct effect of nanostructuring on the alloy properties.

Table. Mechanical properties of the SPD-ed alloy 1965 at room temperature

Initial state	NC structure processed	YS, MPa	UTS, MPa	El, %
Quenched	yes	990	1030	2.1
Aged at 170 °C, 1 hr	no	730	780	7.0
Aged at 170 °C, 10 hrs	no	750	795	7.4

It is concluded the strong importance of the concept of optimal alloy heterogeneity of structure in nanostructure processing involving SPD. This approach should be taken from aluminum alloys conventional grain refinement processing [3], demand specification of it parameters [4] and spread on severe straining of commercial and novel alloy compositions.

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Microstructure and orientation analysis of <111> nickel single crystal subjected to hydrostatic extrusion

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The aim of the present paper was to investigate the microstructure formation and the orientation transformation of nickel single crystals subjected to severe plastic deformation (SPD) by the hydrostatic extrusion process (HE). The experiment were carried out on the sample cuted out by spark erosion saw from the nickel single crystal with axis <111> parallel to the crystallographic direction. The sample was deformed by hydrostatic extrusion within two steps to achieve the final true strain ε_r=2.4. The microstructure and the orientation evolution were investigated using Transmission Electron Microscopy (TEM), Electron Back – Scattered Diffraction (EBSD) and x-ray diffraction (XRD).

The TEM investigation after HE reveals inhomogeneous ultrafine – grained structure with the average grain diameter about 300 nm. The EBSD technique proved that the majority of grain boundaries are of a high angle disorientation. The XRD analysis showed a domination of <111> orientation after HE process which means that the single crystal <111> does not transform to any other orientation during the deformation (even if it is severe deformation).

Effect of ECAP on mechanical properties of an Al-Mg-Sc-Zr alloy

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Effect of intense plastic straining on mechanical properties at room temperature of an Al-Mg-Sc-Zr alloy is considered. This alloy was subjected to equal channel angular pressing (ECAP) at a temperature of 200 ^{o}C using a die with rectangular shape of channels up to true strains of 1, 2, 4, 8. It was shown that ECAP leads to the formation of subgrain structure. Accumulation of total strain imposed by ECAP leads to increasing misorientation and the formation of high-angle boundaries. Yield stress and ultimate tensile strength tend to increase with straining, while elongation-to-failure decreases. This Al-Mg-Sc-Zr alloy exhibits jerky flow associated with Portevin – Le Chatelier (PLC) phenomenon. However, type of serrations is dependent on deformation structure. Role of strain hardening and structural hardening in increment of yield stress is discussed.

The influence of surface roughness of VT6 alloy processed sheets on quality of solid state joining under conditions of low temperature superplasticity

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The terms of solid state joining are conditioned by the surface state of sheets to be joint including surface microgeometry processed from superplastic deformation prior to solid state joining (SSJ). The data on increasing surface roughness in samples with microcrystalline structure during superplastic deformation are given in [1] and it is assumed that roughness exerts a negative effect on mechanical properties of SSJ. The data on the influence of surface roughness on weldability of nanocrystalline materials are rather poor. In this respect it seems interesting to study the influence of surface roughness on SSJ quality under conditions of low temperature superplasrticity.

Sheets of two phase titanium alloy VT6, 0, 8 mm in thick, with micro- and nanocrystalline structures were used. The initial processed sheets had MC structure with a mean grain size of 3-5 μm. After multi-step forging a nanocrystalline structure was processed (a mean grain size – 0, 2 μm) [2]. The processed sheets were subjected to different surface treatment: mechanical diamond paste polishing, zero grain grinding, acid etching and cleaning by a hog.

Two type constructions were processed. Construction 1 consists of processed sheets with MC structure. Construction 2 – sheets with NC and MC structures. Pressure welding was performed at temperature 750° C. The quality of SSJ was evaluated metallographically regarding relative long range areas of pores in the zone of joining.

The obtained results have shown that the least relative volume fraction of pores in the zone of joining can be processed after polishing of the surfaces to be joining and the largest volume fraction can be processed after cleaning by a hop. The application of NC alloy for pressure welding provides decreasing parameters of roughness as compared the MC alloy.

So, the investigations of microstructures of SSJ with different roughness have shown that with decreasing roughness the quality of SSJ improves. Low temperature superplasticity provides conditions for decreasing long range areas and sizes of pores that influences the SSJ quality.

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The influence of severe plastic deformation and heat treatment on structure and mechanical properties of nickel-iron superalloy

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It is known that nanostructured (NS) nickel-iron based Alloy 718 displays superplasticity even at 600°C, at that a strain rate sensitivity coefficient m=0.37 and relative elongation δ=350% [1], that is important for hard-to-deform superalloy. The present paper deals with the review of earlier studies and original investigations of NS Alloy 718 processed by severe plastic deformation (SPD) via high pressure torsion (HPT) and multiple isothermal forging (MIF). The initial structure consisted of γ-phase grains with disperse precipitations of γ″-phase in the forms of discs, 50-75 nm in diameter and 20 nm in thickness. The NS alloy with a mean grain size of 500 nm - 50 nm has been studied in terms of its phase composition and mechanical properties. It was shown that temperature and strain of SPD have effect not only on grain size but also on phase composition of alloy. Hardness, strength and fatigue properties of NS alloy were carried out at room temperature. Hardness was twice larger than that of a coarse-grained alloy. Tensile tests of NS alloy after MIF have shown very high strength and reduction of plasticity. The ultimate strength of NS alloy (d=80 nm) at room temperature is 1920 MPa, that is higher by the factor of 1.5 than in coarse-grained alloy [2]. Heat treatment is generally utilized for Alloy 718, working at elevated temperatures [3]. Therefore NS alloy was submitted to strengthening heat treatment. Mechanical properties were determined at room and elevated temperatures. The data of heat treatment for the NS alloy correspond to the Aerospace Material Specification requirements (AMS) [3]. Comparative fatigue tests of the samples at room temperature have shown that the properties of NS alloy on the scale of 10^5 cycles are higher by the factor of 1.6 than those stipulated elsewhere [3]. Stress rupture data shown that with decreasing a mean grain size of γ-phase one observes the tendency towards decrease of stress rupture and increase of ductility. Nevertheless, presented condition meets the AMS requirements. The investigation results show that for additional increasing alloy's strength properties at operating temperatures it is appropriate to have δ-phase totally dissolved in order to increase the quantity of strengthening γ″-phase precipitates during aging.

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The Effect of Grain Boundary on Strain Characteristics of Nanostructured Metals Produced by SPD Techniques

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Due to high density of non-equilibrium grain boundaries (GB) the nanostructured (NS) metals and alloys produced by severe plastic deformation (SPD) techniques demonstrate at ambient temperatures ($<0.4T_M$) the attractive mechanical properties in comparison with coarse grained (CG) conterparts. It is caused by prohibition with GB network of intragranular deformation propagation in accordance with Hall-Petch relationship. In addition in the NS materials even at ambient temperatures the diffusion-controlled mechanisms of plastic straining can be realized that are typical for elevated temperatures in case of coarse grained ones. For instance, the temperature dependence of internal friction obtained for nanostructured titanium shows a peak corresponding to true grain-boundary sliding, which is shifted to the low-temperature region by 120 degrees relative to CG titanium. Besides, superplasticity involving grain-boundary sliding is frequently realized in NS metals and alloys processed by SPD at less elevated temperatures (about 200 degrees) relative to their CG counterparts.

In this work the investigations of deformation process development are discussed which were carried out by tension and creep in the temperature range $T<0.4T_M$ (here T_M is the absolute melting point of material) for nanostructured metals produced by the methods of severe plastic deformation. The contribution of grain boundary sliding to the total deformation in the above temperature interval is also considered. An analysis is made of the effect of grain size and grain boundary state on the evolution of individual and cooperative grain boundary sliding in nanostructured metals.

Effect of prior-SPD aging on tensile strength of nanocrystalline D16 aluminum alloy

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Commercial hot-pressed rod of heat hardenable D16 (Al-4.4Cu-1.4Mg-0.7Mn) alloy was subjected to severe plastic deformation (SPD) by high-pressure torsion (HPT) (10 rotations under 6 GPa of 20 mm in diam. samples) at a room temperature. To analyze the effect of heterogeneity of initial structure on the alloy nanostructuring and properties two types of prior-SPD heat treatment were compared. Under the first one, the alloy was solution treated and water quenched, and under the second one, it was additionally artificially aged at a temperature of conventional T6 temper (190 $^{\circ}$C) from 1 to 10 hours. In distinction with the quenched state having quite homogeneous distribution of comparatively coarse particles of secondary T-phase ($Al_{20}Mn_3Cu_2$), the matrix in the aged alloy was additionally strengthened by S-phase (Al_2MgCu) nanoprecipitates of different densities and size.

TEM and X-ray analysis of SPD-ed alloy indicate rather uniform and highly non-equilibrium nanocrystalline structures in both the states investigated, having (sub)grains of 70-100 nm in size and high densities of lattice dislocations. Besides, no significant difference in the type of nanostructure was found due to changes in time of prior HPT aging too. However, the initial alloy state significantly affects the morphology of the matrix structure formed. In the aged alloy, it is more mixed and composed of equiaxed and elongated crystallites, whereas the pre-quenched one predominantly consists of equiaxed crystallites. Moreover, its nanostructure is characterized by a bit smaller size of (sub) grains and higher fraction of high-angle boundaries.

The data on the alloy tensile tests, which have been performed at a room temperature on samples with a gage part of 1x1x3 mm cut from near the half of the HPT sample radius, are presented in the Table. It is found, that maximum the alloy tensile strength parameters are observed in the initially quenched condition and exceed 1000 MPa. Preliminary alloy processing by strengthening heat treatment under the conventional T6 regimes has led to it slightly less strength parameters. Meanwhile, such a treatment can result in significant improvement in the alloy plasticity.

Table 1. Mechanical properties of the SPD-ed alloy D16 at room temperature

Initial state	YS, MPa	UTS, MPa	El (min), %
Quenched	1020	1055	1.8 (0)
Aged at 190 ^{0}C, 5 hrs	995	1020	3.2 (2.5)
Aged at 190 ^{0}C, 10 hrs	990	1030	2.7 (0.8)

Nature of microstructure and phase transformations during the alloy SPD processing, as reasons of it mechanical behavior observed are discussed in detail.

It is concluded, that initial heterogeneity of structure of the D16 alloy in the investigated range has insignificant influence on it nanostructuring and room temperature properties formed under the SPD processing involving HPT at room temperature. However, this factor have to be taken into consideration and further analysis, as SPD-ed aluminum alloys of different systems are planned for wide commercialization and applications in different environments and conditions, requiring high corrosion and fatigue resistance.

Corrosion damage in the aggressive environment of the Zr-2,5%Nb alloy processed by equal-channel angular pressing

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A comparative study of resistance to stress corrosion cracking (SCC) of the Zr-2,5% Nb alloy with ultrafine grained (UFG) structure (grain size 50 ... 200 nm) formed by equal-channel angular pressing (ECAP), and the same alloy in coarse-grained (CG) state (grain size 1 ... 5 μm) was perfomed.

Additionally, for comparison, specimens of the Zr-2,5% Nb alloy after cold rolling (grain size 4 ... 6 μm) were tested.

ECAP was performed at 425 ºC with the number of passes N = 4 at an angle between the channels 90º for the normal state of the Zr-2,5% Nb alloy after annealing 530 ºC.

Comparative SCC-tests on the Zr-2,5% Nb alloy specimens in all three states have been carried out by a specially developed original method of "express" SCC-tests with quantitative assessment of corrosion damage of the specimens after testing (measuring the number and size of pits and cracks).

SCC-tests were performed on sheet specimens (size 20.0 x 4, 5 mm and a thickness of 0.6 mm) using loading by bending at the exposure time to corrosive solution (1 % iodine solution in methanol) 100 and 200 hours and the same level of stress (~ $0{,}8\sigma_{0{,}2}$ for the alloy of the state). Corrosive environment of 1 % iodine solution in methanol was chosen as the most aggressive for zirconium alloys, and allowed for a relatively short period of time trials to observe the various stages of corrosion damage.

On the surface of the specimens after cold rolling after 2-4 h exposure during SCC-tests there have been observed cracks with length up to 1,5 mm, extending from the edges of the specimen, as well as pits with radiating from them small cracks with length of about 20-200 microns. On the surface of the specimens after annealing there were observed pits, and only some of them depart some few cracks with length of about 20 microns. Large cracks, as in cold rolling specimens, in this case were not observed.

Formation of submicrocrystalline structure at ECAP Zr-2,5% Nb alloy does not change the characteristic of CG zirconium alloys mechanisms of SCC associated with pits formation, but leads to increased resistance to corrosion damage under stress compared with the alloy with the initial CG structure: at the same time of exposure in a solution, the number of pits are 2-4 times smaller, and their average size is 4-7 times less, compared with the alloy with the CG structure. The increase in test time from 100 to 200 hours does not lead to a significant increase of pits size. For the alloy with UFG structure after ECAP during testing at the time of exposure to 200 hours there no cracks were observed comparing to the CG alloys. For the UFG alloy in the local areas there are concentrations of very fine pits with a diameter of about 2 microns. The accumulation of corrosion damage with increasing exposure time from 100 to 200 hours is mainly due to the appearance of new pits, but not of their growth.

Thus, the Zr-2,5% Nb alloy with UFG structure is less susceptible to corrosion damage in comparison with the CG structure (annealed or cold rolling).

The texture strengthening effect in a magnesium alloy subjected to severe plastic deformation

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It is known that application of equal channel angular pressing (ECAP) can lead to an increase of strength in the magnesium alloys due to both the grain refinement and formation of crystallographic texture [1].

The focus of the present work is to compare the structure and mechanical properties of a magnesium alloy after ECAP, simple compression and homogenization.

The AM60 magnesium alloy with chemical composition Mg-6%Al-0, 5%Zn-0, 13%Mn has been selected as initial material for investigations. The quenched samples were subjected to 8 passes ECAP at 300ºC in the die with channel intersection angle 120º using rout B_c and additional 4 passes at 210ºC. For comparison the alloy was also strained by hot compression to 30% at 350ºC.

Equiaxed grains with a mean size of 23 μm were observed in microstructure of the ECAP samples after 8 passes. Bimodal structure with an average size of 4.8 μm for ultrafine grains and 23 μm for coarsened grains was typical for samples processed by 12 passes of ECAP.

Microstructure of the alloy after hot compression was characterized by appearance of the deformation twins inside of initial grains of 60 μm in size.

Pole figures developed by X-ray analysis have demonstrated the $(10\bar{1}0)$ texture after 12 passes of ECAP.

Table 1. Mechanical properties of the AM60B alloy

state		$\sigma_{0,2}$, MPa		σ_B, MPa		δ, %	
		long	cross.	long	cross.	long.	cross.
Gomogenization	1	80	80	160	160	11	11
Compression 30%	2	95	95	240	240	10	10
ECAP 8 pass.	3	180	170	260	265	10	7
ECAP 12 pass.	4	160	310	285	430	13	10

Tensile tests of the ECAP samples were carried out for longitudinal direction and cross section. Samples undergoing compression were cut in diametrical section to be paralleled compression axis.

Strengthening effect of the alloy after simple compression is conditioned obviously by appearance of new deformation twins. It should be noted that the influence of twins on enhancement of strength for example in copper was investigated earlier on numerous papers [2].

The strength after 8 passes ECAP is higher by 10-15 MPa in comparison with samples subjected to hot compression. It means that strengthening due to grain refinement to 23 μm has the same effect to be compared with strengthening from twins.

The alloy after 12 passes of ECAP has demonstrated a highest strength and strong anisotropy is a result of grain refinement and texture forming. Ultimate tensile strength in longitudinal direction after 12 passes is lower, than after 8 passes, testifying to favorable plane orientations for basal sliding in volume of a material having a texture peak. The highest ultimate tensile strength of 430 MPa for the investigated alloy has been achieved due to "texture strengthening" observed earlier in other alloys with hexagonal crystal lattice [2].

It should be noted, that further grain refinement of the alloy by rolling leads to a decrease of tensile strength due to forming of traditional rolling texture [3].

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Thermal stability of 5483 Al alloy processed by ECAP

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It is well known that microstructure of materials processed by equal channel angular pressing (ECAP) in not very stable. There were published many experimental and theoretical evidences of this fact obtained by various methods such as microstructure observations, micro-hardness measurements and computer modeling.

The aim of presented paper was to investigate the thermal stability of microstructure and mechanical properties of the Al 5483 alloy processed by ECAP. Rectangular samples of 5483 Al alloy processed by ECAP in an L-shaped, 90^{O} channel at 180 ^{O}C using route Bc were used for investigations. For the aim of the investigations there were performed standard tests, such as micro-hardness measurements, TEM observations and tensile tests of non-standard micro-specimens cut-out from various parts of the samples processed by ECAP. The investigation was carried out for samples subjected to 1, 4 and 8 passes of ECAP.

It was found that microstructure and mechanical properties (microhardness, yield strength, tensile strength and elongation to rupture) are stable up to about 250 ^{O}C. It was also found that annealing in the temperature range 250-300 ^{O}C results in the strong changes of grain size and mechanical properties. At the higher temperatures (300-350 ^{O}C) stabilization of mechanical properties was observed. Materials annealed in the temperature range 265-350 ^{O}C had higher yield strength, tensile strength and elongation to failure than the material in the initial state.

Effect of the severe plastic deformation on magnetic properties of amorphous alloys

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The plastic deformation, especially severe plastic deformation (SPD), is a complicated process which not only results in the forming of the deformable solid body but also causes significant changes in the structure and properties of the material [1]. The deformation particularly stimulates the mass transfer and the change of the chemical composition both on macroscale and microscale levels [2]. The redistribution of the components of solid solution in course of plastic deformation in its turn can alter a number of physical properties of materials and particularly their magnetic properties.

The effect of severe plastic deformation in Bridgman's chamber on magnetic properties of the Fe-based amorphous alloys obtained by the melt quenching was studied. The structure of the deformed samples was studied by transmission electronic microscopy and X-ray-structural analysis. The magnetic properties were determined using vibrating anisotropy torque meter.

The substantial alteration of saturation magnetization (M_s) depending on the number of ferromagnetic and antiferromagnetic components in the alloy was revealed. It has been revealed that the change of the positive effect of the saturation magnetization as the result of severe plastic deformation is more the more is the number of ferromagnetic components in amorphous alloy. The character of change of coercivity (H_c) with the growth of turn (N) is the same in all studied amorphous alloys. The dependence $H_c(N)$ shows the abrupt growth of the H_c value at $N = 0.5–1$ at the beginning with the following gradual decrease down to the values slightly exceeding the initial ones.

The structural state of the alloys corresponding to the maximum M_s values after SPD is characterized by the presence of the expressed skewness of a profile of the intensity of the main halo corresponding to the amorphous state in X-ray diffraction images. In this case both X-ray and electron microscopic studies do not reveal the existence of nanocrystalline phases in these states in majority of cases. It seems that the internal separation into nanoscaled areas (nanoclusters) enriched by various components occurs under the action of severe shear stress in a random multicomponent structure. Such separation can be stimulated by both elastic and chemical interactions between the atoms of metals and nonmetals in the amorphous matrix.

There is a possibility to increase the value of saturation magnetization significantly, which is the weak point of amorphous and nanocrystalline soft magnetic alloys, with the retention of low values of coercivity as a result of SPD treatment in optimal regimes.

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Increase of ultimate tensile strength of ECAP-processed titanium Grade-4 during drawing

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The high productive technology based on equal channel angular pressing via the «Conform» scheme (ECAP-C) [1-3] is perspective for achieving high strength semi products of nanostructural (NS) titanium. This technology allows producing rods of NS titanium 3 meters long and of 5-7 mm diameter, semi-products for medical implants.

In this work influence of drawing on microstructure and mechanical properties of CP Ti Grade-4 which was preliminary subjected to a various number of ECAP-C passes were studied. UTS of titanium after n=6, 8 and 10 passes ECAP-C is practically identical. The probable reason is that when the number of passes increases from 6 to 10 the dislocations move into grain boundaries and misorientations between grains grow but he dislocation density decreases. At the same time ultimate tensile strength of titanium ECAP-C n=10 after additional deformation by drawing is reasonably higher than after ECAP-C n = 6 and drawing (Fig 1). This can be explained by the fact that the structure «ECAP-C n=10» with ultra-fine grains and high-angle grain boundaries provides a stronger additional structure refinement during subsequent drawing, than in case of «ECAP-C n=10». On the other hand, additional research is necessary for a more reliable explanation of the obtained results.

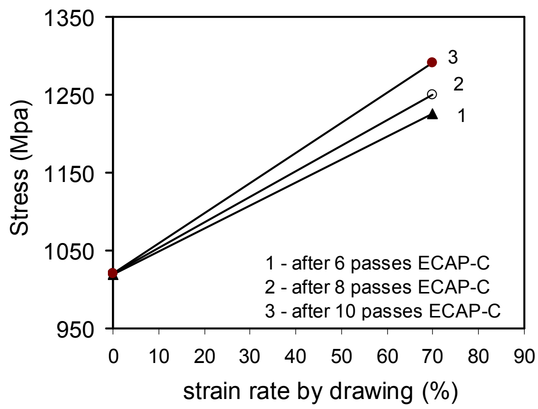

Fig. 1. Increase of ultimate tensile strength of titanium Grade-4 during drawing after 6, 8 and 10 passes ECAP-C

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Simulation of a promising severe plastic deformation technique for producing high-strength semi-products

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Strength enhancement in materials is one of the key tasks in modern metallurgical and mechanical engineering industries.

The techniques, which enable strengthening metallic materials 1.5-3 times via severe plastic deformation (SPD), have been recently developed for these purposes. Production of high-strength long-length steel rods and wires, which are used for fabrication of a broad range of fasteners, is of particular interest.

The majority of known processes on production of high-strength semi-products includes SPD usage, for example, equal channel angular pressing, twist extrusion, and additional deformation by the most popular metal forming techniques such as drawing, rolling and etc.

The proposed method of shear drawing [1, 2] (Fig.1) allows combining severe plastic deformation and material reducing processing.

Low carbon steel with a carbon content of 0.1 % was used for investigations.

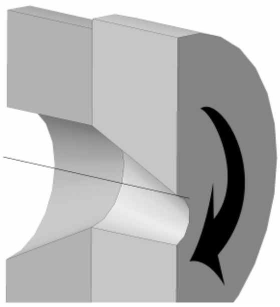

Fig.1 Scheme of drawing with shear

The stress-strained state of the shear drawing was investigated by the finite-element method in the Deform3D package. The geometry of a forming tool was optimized by using the factorial experiment according to the virtual data. The achieved levels of accumulated strain within 5-6 cycles of processing are e = 4–8, which enables predicting effective structure refinement and strength enhancement. A physical experiment is planned to be conducted in future for verification of virtual results.

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Effect of severe plastic deformation on decomposition of supersaturated solid solution in magnesium alloys

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The severe plastic deformation (SPD) is well known as the way to improve significantly the strength properties of metals and alloys [1]. It is especially attractive for application to the light structural materials, such as Al- and Mg-based alloys, which are used commonly in the machinery areas, where the weight saving of machines (airplanes, cars and others) is of a great importance. Therefore, the main requirement to such alloys is the high strength properties. Both Al and Mg alloys are known to be strengthened at most by aging resulting in decomposition of the supersaturated solid solutions and widely used in such conditions. It is reasonably to suppose, that the strength properties of the age-hardenable alloys may be improved by additional SPD. In the report the main results of the works in this direction on the Mg-base alloys are presented. The works were conducted on the Mg alloys, where the aging was known to improve notably their strength owing to a possibility of the Mg supersaturated solid solution formation and its decomposition during aging with strengthening effect. These are the binary Mg-Sm, the ternary Mg-Sm-Y and the quasibinary Mg-Al_2Ca alloys. The alloys of these systems with different contents of the alloying elements were prepared and solution treated then aiming to obtain the Mg supersaturated solid solution. After solution treatment the alloys were subjected aging using different regimes and SPD in various combinations. SPD was performed at room temperature and 200 ºC by torsion under a pressure of 4 or 6 GPa to $\varepsilon \approx 6$ (5revolutions). The strengthening of the alloys was estimated by the hardness measurements. Investigations discover a number of the general regularities of the strength characteristics change of the alloys of all systems depending on the combine action of aging and SPD and a possibility to improve them notably at certain conditions. The general regularities are, as follows: 1) SPD of the alloys results in increase of their hardness at all aging regimes and both SPD temperatures, although the observed hardness increase depends on the system of the alloys, their composition and aging regime. 2) SPD activates and accelerates decomposition of the Mg supersaturated solid solution resulting in a possibility to reach higher hardness during aging, than during aging without SPD. This regularity is evident from the plot presented in Fig.1, 2 for the two studied alloys. Acceleration of the solid solution decomposition due to SPD can be seen from the shift of the hardness maximum on the curves to shorter aging time (Fig.1). Activation of the hardness response due to SPD is seen comparing the hardness curves for the alloy aged after quenching + annealing at 200 or 300ºC without and with additional SPD. 3) Strengthening of the alloys resulting from SPD is retained after annealing at enough high temperatures corresponding to the possible operation of Mg alloys (up to 300ºC). With increasing annealing temperature in these limits the hardness of the alloys subjected to SPD decreases in accordance with formation of the enough coarse particles of the phases precipitated from the Mg solid solution and processes of recovery without recrystallization.

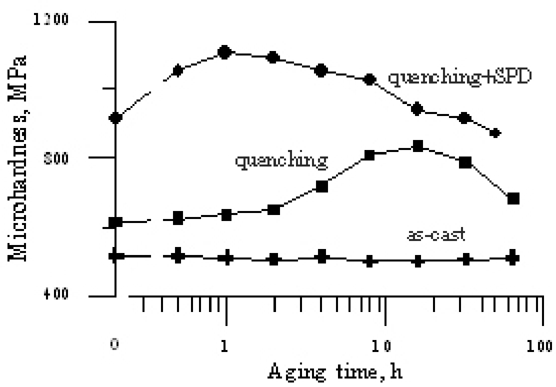

Fig. 1.Microhardness of the Mg-0.62% Al-0.57% Ca alloy vs. aging time at 175 °C

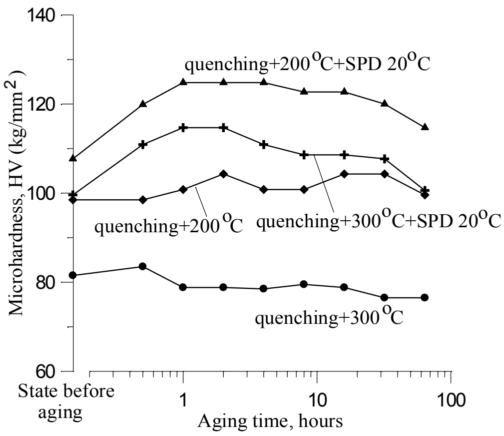

Fig. 2. Micro-hardness of the Mg-4.5% Sm alloy vs. aging time at 200 °C

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The structure features and mechanical properties of low carbon steels processed by severe warm deformation

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Processing high-strength state in low-carbon lean alloy steels is an actual problem for modern mechanical engineering industry since conventional methods of heat treatment do not allow the high level of mechanical properties to be reached. Such steels are widely used, for example, in the construction of main petroleum and gas pipelines operating in inclement climatic conditions and under high pressures. The challenge for these steels is the enhancement of mechanical properties and corrosion resistance. To produce of steels with nanocrystalline and ultrafine grained structure is one of the ways of solving this problem.

This paper is concerned with the effect of severe warm deformation on the processes of generating ultrafine grained structures in low carbon lean alloy steels. A detailed certification of steels' structures produced by warm deformation has been performed. It has been shown that various type of an ultrafine grain structure is gained at the different scheme of the warm deformation.

The mechanisms of changing mechanical properties of steels depending on the structural state of steels have been established.

It was determined that stells in ultrafine grained states displayed especially high strength properties that 2-3 times higher than those of a coarse-grained state, at the same time ultrafine grained materials possess enhanced ductility characteristics at room and lower temperatures.

Impact strength increasing of low-carbon steels with ultrafine grained structure has been established.

The nature of the high mechanical properties of steels with different types of ultrafine grained structures was analyzed.

Finite element method simulation of the diffusion bonding and superplastic forming processes to produce three-layered structures of vt6 titanium alloy

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Technology that combines diffusion bonding and superplastic forming is used to produce complex shape three-layered structures that include two covering sheets and filler sheet that plays the role stiffening ribs. It is very important to find rational geometry parameters, such as thickness ratio of the covering sheets and the filler and the entanglement angle of the ribs. Another important problem is finding rational values of gas pressure and duration of bonding and forming processes. Computer simulations can be very helpful in solving these and other related problems.

We report on the results of diffusion bonding and superplastic forming of three-layered structures of two types. First type structure is elongated with nearly parallel stiffening ribs, while the second one has axial symmetry. In the first case the forming process can be effectively modeled under the assumption of plane-strain condition, while in the second one the axisymmetric problem should be solved. Constitutive relations describing superplastic flow of titanium alloy must be found from the tests where material is loaded closely to the stress state that is realized during forming of real structure. In our simulations we use the constitutive relation parameters found from the wedge-cup tests and from cone-cup tests for the two abovementioned types of structures.

Diffusion bonding of sheets is one of the key operations of production titanium alloy three-layered structures. To obtain a desired shape of the structure, a stop off covering is made on a part of the sheet surfaces prior to welding. In the present work, by means of finite-element simulations, we investigate the behavior of the top off coating during diffusion bonding. Two ways of covering are compared, with and without grooving along the border of covering. It is demonstrated that grooving prevents penetration of the stop off coveting into the welding zone.

Deformation of the alloy $Ti_{50}Ni_{25}Cu_{25}$ in Bridgman chamber

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The effect of severe deformation on the structural and phase transitions of alloy $Ti_{50}Ni_{25}Cu_{25}$ in amorphous and crystalline states has been investigated. An amorphous ribbon (50 μm thickness; 8 mm width) obtained by melt spinning in the argon atmosphere was used. A crystalline state was obtained by annealing the initial amorphous ribbon. Severe deformation was carried out in the Bridgman chamber at room temperature under hydrostatic pressure P = 4 GPa. The strain rate varied from 2.8 to 4.2 s^{-1}. The number of turns of the mobile anvil, *n*, varied as follows: $^{1}/_{8}$, ¼, ½, 1, 2, 3, 4, 5, 6, 7, 8 and 9. The initial stages of the severe deformation of the amorphous state [1] ($n = {}^{1}/_{8}$ - 1) were characterized by the appearance of crystalline phases (the volume fraction of the crystalline phase at *n* = ½ is ~ 80%). Then the volume fraction of the crystalline phase decreased with the increasing strain, and at *n* = 4 the alloy was completely amorphized. However, for *n* = 6, a two-phase amorphous-crystalline structure was revealed through the X-ray and electro-microscope investigations. Further increase in the severe deformation up to *n* = 8 led to the amorphization of the alloy again. It was observed that the reduction of the deformation rate of the amorphous material from 4.2 to 2.8 s^{-1} led to a significant reduction in the intensity of the primary crystallization. The initial structure of the crystalline state of alloy $Ti_{50}Ni_{25}Cu_{25}$ was characterized by lamellar martensite B19. With the increase in the strain, degradation and destruction of the initial lamellar martensite structure and the transformation into an amorphous state through the deformation dissolution of the crystalline phase fragments was observed. Under the deformation corresponding to *n*=4 the original crystalline structure became completely X-ray amorphous. The subsequent deformation was structurally observed through the repetition of the "crystallization – amorphization" cycle [2]. Thus, the study has demonstrated that three cycles of direct and reverse structural phase transitions are realized through a successively increasing deformation of both initially amorphous and initially crystalline states. It has been established that the amorphization of a crystal structure in the material under study is mainly the result of the deformation dissolution of small crystals. In turn, the nanocrystallization of the amorphous state has been determined by the superposition of different channels of the dissipation of the elastic energy injected into the material in the course of severe deformation. It has been concluded that the development of individual shear bands and their branching, the accumulation of an excess free volume, and the presence of direct and reverse thermoelastic martensitic transformations are among the most probable ways of dissipation.

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Microstructure evolution in Cu-Cr-Zr alloy during extensive cold rolling

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Structural changes in Cu-Cr-Zr alloy were studied under extensive cold rolling strains. Two sets of samples were used. The samples of Cu-Cr-Zr alloy were solution treated and then a part of samples was subsequently aged at 450°C. The cold rolling was carried out at an ambient temperature up to total strains above 4. The microstructure evolution during the process of intensive plastic deformation was characterised by the development of ultra-fine grain/subgrains with the transverse (sub) grain size of about 100 nm. Cold rolling to the relatively low strain resulted in elongation of initial grains along the rolling direction. Further deformation to total strain above 4 led to increasing the density of microshear bands and the development of nano-scale grains. The effect of initial microstructure on the structural mechanisms and the kinetics of grain refinement during large strain processing are discussed in some details.

Atomic level investigation of initiation of accommodation processes at triple junctions in nanocrystalline materials

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Grain boundary sliding (GBS) is one of the dominating processes of high-temperature plastic deformation of small grain size polycrystals. Deformation of polycrystals depends not only on the GBS but also on the degree of shear accommodation in the triple junctions (TJ). The role of TJ in the development of the plastic deformation is typically studied using model tricrystals [1-4]. On the other hand, in the study of TJ behavior in nanocrystalline materials computer simulation methods become increasingly important. Molecular dynamics method is an efficient tool in the study of polycrystals at atomistic level. Computer simulations enable us to estimate qualitatively the processes accompanying plastic deformation in two-dimensional [4-6] and quasi-three-dimensional crystal models [7-10].

In the present work by means of molecular dynamics simulation, self-organization processes during plastic deformation of nanostructure was carried out and the results were compared to the results of experimental study of accommodation observed during high-temperature creep of model tricrystals. Fairly good qualitative agreement was found between the accommodation processes observed experimentally for tricrystals and numerically for a polycrystal with nanosize grains. With the help of simulations the early stage of development of accommodation was investigated. It was established that GBS and accommodation processes evolve simultaneously at rather high external stress level used in the simulations. Emission of lattice dislocations from TJ and grain boundary ledges takes place not only due to accommodation of shear incompatibility in TJ but also due to relaxation of internal stress at concentrators. Cooperative migration of grain boundaries in TJs having the shape of spiral was revealed.

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Fracture surface morphology of titanium alloy with different prehistory of structure

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The fracture surface morphology of impact specimens of the (α+β) - titanium alloy was investigated. The specimens was manufactured by pressure welding of sheets with nano- and microcrystalline (NC and MC) structures with average grain size 0.2 μm and 3 μm, respectively. As a result of pressure welding the average grain size in the sheets with initial NC structure has increased to ~2 μm (MC type I) and in the sheets with initial MC structure has increased to ~4 μm (MC type II). The microstructure and the fracture surface of type I and II sheets were investigated by using the scanning electronic microscope «JXA-6400». The casual secant method (CSM) was used for definition of the average grain size. The image analysis system (IAS) was used for size distribution definition of the fracture cells [1, 2]. The histograms of size distribution have shown that the average size of cells and α-grains in both types of sheets are similar. The fracture of sheets occurs mainly on grain boundaries, i.e. has intercrystalline mechanism. Meanwhile, the histogram range corresponding to big fracture cells in type I sheet is wider than in type II sheet. In type I sheet the maximum size of fracture cells amount to ~13 μm that corresponds to the group from ~7 grains. In type II sheet the maximum size of fracture cells amount to ~16 μm that corresponds to the group from ~4 grains. The revealed difference in the fracture surface morphology can be connected with prehistory of structure of compared sheets.

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Effect of heat treatment on nanostructuring in high-strength aluminum alloy by severe plastic deformation

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Effect of prior heat treatment on nanostructuring in high-strength 7475 Al alloy, subjected to high-pressure torsion at ambient temperature up to strain of ~7, was investigated. The alloy was

(i) solution treated and water quenched; and then

(ii) artificially aged

before high-pressure torsion. In distinction with the state (i), the state (ii) was characterized by the presence of nanoprecipitates of strengthening η′- phase in aluminium matrix.

Relatively uniform nanocrystalline structures can be produced by high-pressure torsion in both the alloy states accompanied by significant increase in its hardness with a factor of about 1.8. In the as-quenched alloy (state (i)), hardness increases discontinuously in the earlier stages of deformation followed by saturation at higher strains. In contrast, in the aged state (ii), hardness growths continuously with almost constant hardening rate in the strain interval investigated.

The initial alloy state significantly affects the morphology and characteristics of the microstructure induced by high-pressure torsion. Namely, a mixed (sub)grain/cellular structure, which is composed by both equiaxed and elongated crystallites separated by high-density dislocation subboundaries, is developed in the aged state (ii), whereas severe plastic deformation of the as-quenched material (i) leads to formation of more equiaxed and coarser crystallites with more equilibrium boundaries.

Influence of initial phase structure on the microstructure transformations taking place during severe plastic deformation are discussed in detail. It is concluded that the presence of the metastable η’-phase precipitates in the deformation structure of the preliminary aged alloy may delay the formation of equiaxed nanoscale grains even at large high-pressure torsion strains due to inhibition of dynamic recovery and homogenization of dislocation slip.

Microstructure and mechanical properties of copper after ECAP and cold rolling

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Ultrafine grained (UFG) materials have been subject of serious interest due to their outstanding mechanical properties. Severe plastic deformation (SPD) methods are capable of producing bulk UFG materials. Among these methods equal channel angular pressing (ECAP) is one of the most attractive [1]. Relatively large scale rods with rather homogeneous structure could be produced by ECAP. These rods are often subjected to rolling in order to obtain UFG sheets [2, 3]. However, influence of rolling on microstructure and properties of ECAPed metals hasn't been studied properly. That's why in current work we were compared microstructure and mechanical properties of commercial purity copper after 1, 2, 4 or 10 ECAP passes by route B_C and after ECAP with subsequent cold rolling. Strain imposed during one ECAP pass was equal to 1.15, the resulting rolling strain was about 2.2. Specific attention has been paid to microstructural characterization in 3 dimensions by electron backscattered diffraction (EBSD) analysis utilizing field emission gun scanning electron microscope (FEG - SEM) in order to obtain complete qualitative and quantitative understanding of structural features.

Microstructure and properties of copper after ECAP with various passes numbers are extensively studied [4, 5]. Our data was in good correlation with the results of previous investigations and hence wouldn't be described here in details. It should be noted that increase of strain imposed during ECAP resulted in structure refinement and strengthening of copper. Structure was rather equiaxed with fraction of high angle boundaries (HABs) about 50 % after 10 passes.

Microstructure after rolling consisted from flattened in normal direction grains. They were elongated in rolling direction. Thickness of these grains decreased with increase in ECAP passes numbers and was about 0.3 μm after 10 passes and rolling. Inside grains well developed fragmented structure was found after 1 or 2 ECAP passes and rolling. Rolling after higher number of ECAP passes resulted in less prominent fragmented structure, especially after 10 passes. Fraction of HABs varied significantly in different sections: it was approximately the same in sections perpendicular to rolling direction and transverse direction but much lower in section perpendicular to normal direction. For example after 10 ECAP passes and rolling these values were 70-74 % and 45 % respectively.

Dependence of strength on ECAP passes number was similar in both cases of ECAP and ECAP with subsequent rolling. Strength increased rapidly after first and second passes and reached saturation after forth pass. Rolling after ECAP significantly increased strength by 80 – 120 MPa. Thus U.T.S. after 10 ECAP passes was equal to 403 MPa and after 10 passes and rolling - to 485 MPa. Such strengthening was accompanied by loss of plasticity. Elongation to failure after ECAP was about 10 – 13% and after ECAP and rolling - about 5 – 8%. It should be noted that after rolling dependence of elongation on passes number was very similar to those for strength, i.e. it increased with increase in passes number. Correlation between microstructure and mechanical properties will be discussed.

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Structure and properties of ultra-fine grained Cu-Cr-Hf alloys prepared by severe plastic deformation

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The severe plastic deformation (SPD) of low-alloying Cu-alloys that is used in order to receive a submicrocrystalline structure and improve strength characteristics of these alloys, especially the fatigue, was studied in various works [1-3]. In [4] the high-pressure torsion (HPT) method was used for Cu-Cr-alloys with the different content of Cr that allowed to raise their thermal stability. The aim of the present study is to investigate the structure and properties of ternary Cu-0,66 %Cr-0,89 %Hf alloy (in mass %) received by HPT and the effect of the initial state on the thermal stability of this alloy. The alloy was prepared by arc melting in a purified argon atmosphere. The ingot of the alloy was subjected to hot forging. The forged rods 12 mm in diameter were cut into discs (0,6 mm thick) to be subjected to heat treatment (HT) in accordance with the two following regimes: cold water quenching from 900ºC (2-h holding) – regime HT1, and annealing at 600ºC for 2 h and cooling in the air – regime HT2. SPD by HPT was performed under 4 GPa pressure at room temperature using the Bridgman anvil. The true strain was ε = 4,8 (5 revolutions). The microhardness and the electrical resistivity were measured on the samples with different initial states after subsequent heating, in the temperature range from 50 to 550ºC (step of 50ºC). The microstructure of the alloy was studied at all stages of experiment using as-cast, forged, quenched and annealed samples and ones subjected to HPT. The investigation of the microstructure of samples showed the presence of the inclusions of Cr-phase (almost purity Cr) and Hf-phase (Cu_5Hf) in the structure. These phases dissolved into Cu-solid solution at the heating before quenching, and precipitated from the supersaturated solid solution after annealing at 600ºC. After HPT the structure of the alloy has been significantly refined. The Cu_5Hf inclusions, which are not dissolved after the heat treatment, has been broken, whereas the Cr-inclusions are not deformed. The changes of the microhardness (H_V) of alloy in the regimes HT1 and HT2 were identical, but the absolute values were rather large in the initial quenching state (regime HT1) than in the initial annealing one (regime HT2). It is likely to be related to the peculiarities of a dislocation structure. The precipitation of Cr and Hf particles from supersaturated solid solution on dislocations during SPD leads to a higher strengthening. In both cases the microhardness increases and reaches its maximum in the temperature range from 350 to 450ºC. Then H_V began to decrease more sharply up to 550ºC. At the same time, the electric resistivity (ρ) after HPT was considerably lower in the initial annealing state (~ 2 time) than in the initial quenching state in the consequence of the decomposition of the solid solution at 600ºC. At first, the electrical resistivity in its initial annealing state sligtly rises from 3,67 to 4,51 μO·cm as the heat treatment temperature is increased up to 300ºC, then above this temperature ρ is decreased. The electrical resistivity of the sample in the initial quenching state subjected to HPT decreased progressively from 9,06 to 3,45 μO·cm as the heat treatment temperature is increased up to 550ºC. This fact is indicated on the decomposition of the solid solution into all temperature range. Thus, enough high thermal stability of the Cu-0,66 %Cr-0,89 %Hf alloy subjected to HPT was reached in both states: initial quenching and initial annealing. The use of HPT and subsequent annealing for the alloy allowed to obtain quite a favorable combination of the properties at the 450ºC, when the microhardness remains high and the electrical resistivity decreases to almost the initial value, which the alloy posesses without HPT (Table).

Table 1. H_V u ρ of alloy Cu-0, 66 %Cr-0, 89 %Hf in different states

Alloy	HT	H_V, MPa			ρ, μO·cm		
		Before HPT	HPT	HPT+ heating at 450ºC	Before HPT	HPT	HPT+ heating at 450ºC
Cu-0,66%Cr-0,89%Hf	1	1383±65	2368±19	**3177±60**	7,89±0,18	9,06±0,28	**4,06±0,16**
	2	1230±57	2050±39	**2510±50**	3,67±0,15	4,51±0,14	**3,45±0,19**

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Nanocrystalline S304H austenitic stainless steel subjected to multiple forging

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A Super304H austenitic stainless steel, Fe – 0.1%C – 0.12%N – 0.1%Si – 0.95%Mn – 18.4%Cr – 7.85%Ni – 3.2%Cu – 0.5%Nb – 0.01%P – 0.006%S (all in mass %), with an average grain size of about 10 μm was used as the starting material. The steel samples were annealed at 1100°C for 30 min and then subjected to multiple forging at ambient temperature. Rectangular samples with initial dimension of $10\times12.2\times15$ mm^3 were cut for multiple forging tests. The multiple forging was carried out by means of multi-pass compressions at room temperature with consequent change of the loading direction in 90 degrees. The samples were forged under a strain rate of 10^{-3} s^{-1} to a strain of ~0.4 in each pass followed by water quenching. It was shown that the multiple forging resulted in significant strengthening. The hardness of 6 GPa and the yield strength of 2000 MPa were achieved after total strain of 4. The strengthening during the multiple forging was accompanied by the formation of almost equiaxed nanocrystalline structure with an average grain size of about 50 nm. The softening behavior of the nanocrystalline samples was studied by means of isochronal annealing at temperatures of 500° to 700°C for 30 min. The operative mechanisms of recovery and recrystallization in the nanocrystalline steel and their effect on the mechanical properties are considered.

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Influence of alumina particles on grain size in nanostructured composite manufactured by ARB process

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In the present work, the accumulative roll bonding process was performed up to thirteen cycles at room temperature for manufacturing the nanostructured aluminum/15 vol.% alumina composite strip. TEM investigation confirmed that the development of UFGs by ARB process involves severe shear deformation, pinning of the grain boundaries by alumina particle and particles of oxide film, and continuous recrystallization. The process starts with a low angle boundaries formation, then shear mechanism is introduced and further grain refinement occurs up to 13th cycle. The mean grain size after the thirteenth ARB cycle of MMC strip was around 100 nm (Fig. 1).

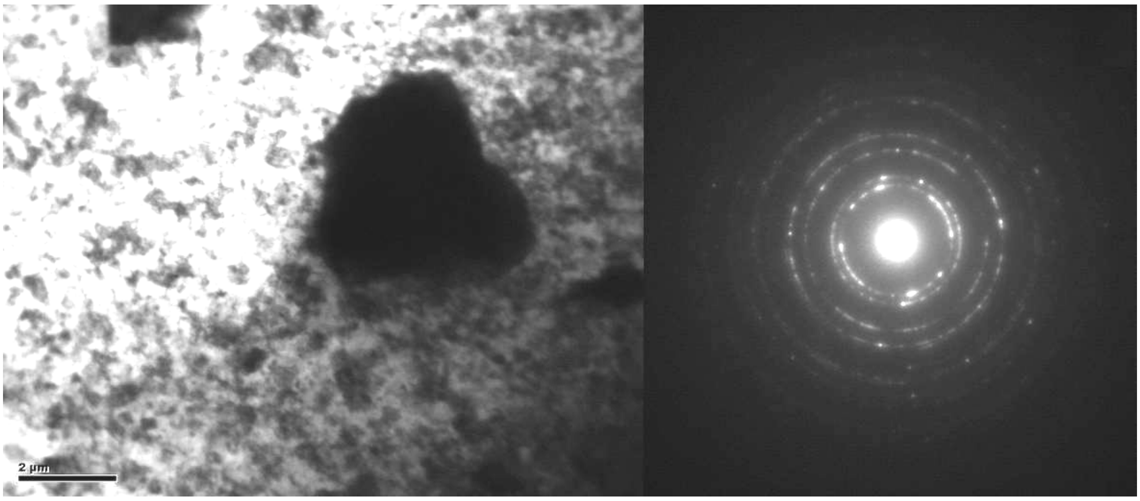

Fig. 1. TEM micrograph and the matching SAD pattern from RD–TD plane of ARBed composite strip with 15 vol.% reinforcement after thirteen cycles.

The findings also revealed that the presence of large particles and the specific structure of the deformed matrix in the vicinity of the particles can be responsible for the PSN of recrystallization [1]. The crystallite or subgrain sizes of ARBed pure aluminum and aluminum/alumina composite calculated by the Williamson–Hall technique were 150 and 63 nm, respectively. The produced MMC exhibited a higher hardness than the annealed and ARBed pure aluminum strips so that the hardness values of the aluminum/15 vol.% alumina composite were 5 and 1.3 times higher than those of the annealed and ARBed pure aluminum, respectively (Fig. 2).

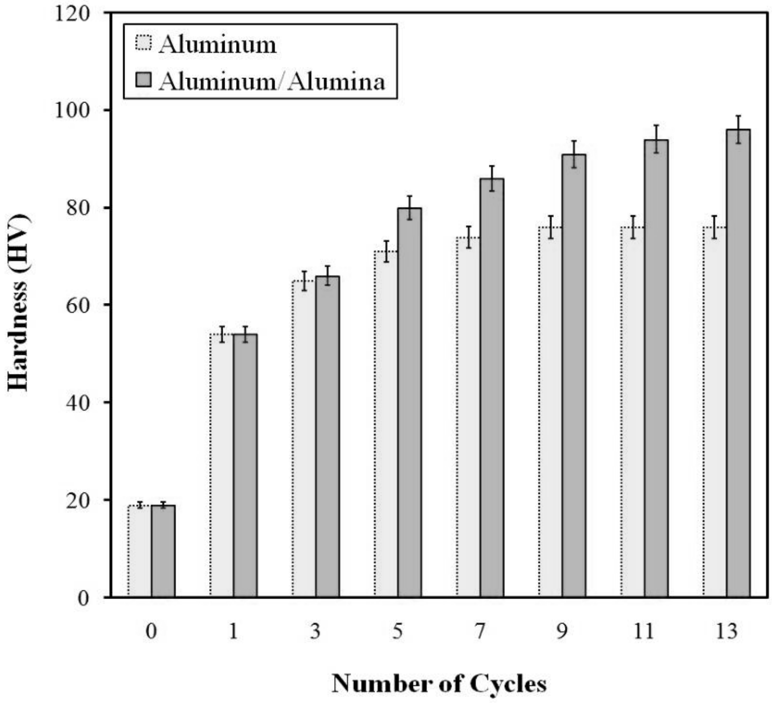

Fig. 2. Variation of hardness versus number of ARB cycles (strain) for reinforced and unreinforced AA1100 alloy

After the first cycle, hardness rapidly increased, then dwindled and finally saturated by further rolling. This saturation takes place because the materials reach the steady state density of dislocation determined by a balance between dislocation generation during plastic deformation and annihilation in the dynamic recovery and continuous recrystallization [2,3].

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The evolution of structure and mechanical properties of Hadfield steel single crystals under cold rolling

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Hadfield steel is an austenitic manganese steel that possesses high strain hardening, which is predominantly attributed to the formation of twin boundaries at room temperature under tension and compression. Plastic deformation by twinning may play a crucial role in structural refinement of the materials. Hence, Hadfield steel is an interesting material for studying hardening under severe plastic deformation conditions because quick fragmentation by twinning and a rapid increase in the dislocation density are expected to occur at room temperature.

The microstructure and mechanical properties of <001> and <111> Hadfield steel (Fe-13Mn-(1.0÷1.3)C wt.%) single crystals were investigated under cold rolling using optical, transmission electron microscopy, X-ray diffraction and measuring of microhardness. Rolling was conducted at room temperature up to 5-75% reduction.

The evolution of mechanical twinning was observed to set-in at the early stages of rolling of the single crystals of both orientations (ε=5%). Increasing the strain produced an increase in the density of the deformation twins and the number of active shear systems. After strain $\varepsilon > 40\%$, the initial twin net was destroyed, the twin boundaries were distorted, and shear bands (SB) were observed. Moreover, there was orientation dependence of mechanical twinning formation in specimens after repolishing and etching. Under rolling up to 10-20% reduction we observed more homogeneous net of twins in <001> single crystals as compared with microcrtucture of <111> single crystals. In <111> single crystals «rough cells» of twins formed at first. Walls of this «rough cells» consisted of separate thin twins, and inside of this «rough cells» the thin twins of several active shear systems were observed.

Using transmission electron microscopy, either twinning or a high density of glide dislocations was observed, which formed a cellular dislocation arrangement. The twins were thin, and the distance between twin boundaries varied from 5 nm to 100 µm, and the twins often did not grow in width. Instead, the increase in the fraction of the twinned volume was mainly due to the nucleation of new thin twins.

The main hardening results from the interaction between multiple twinning and dislocation slip. The distortion of the twin traces and the formation of steps on the boundaries of the "matrix-twin" are the results of the interaction of several twin systems with each other and with dislocations from different slip systems. Because the twinned volume and the volume in the SBs differ in their crystallographic orientation from that of the matrix, different twin systems can be activated inside those volumes. As a result, twins and SBs are often internally twinned, which should also contribute to internal stresses and provide for a further increase in the strength of the steel.

Cold-rolling reduces the size of the coherent scattering regions (CSR) and increases the microstrain of the crystal lattice, $\Delta d/d$, where d is the interplanar spacing. Even at lower degrees of strain (ε=20-30%), the size of the CSR indicated the formation of an ultrafine-grained state with undistorted fragments of the microstructure 10-20 nanometers in size.

Cold-rolling caused a high strain hardening. Microhardness rapidly increase as compared with initial value (H_μ=2.4 GPa). Rolling up to a strain of ε=30% gives values of microhardness H_μ=5.0 GPa. After strain $\varepsilon > 60\%$, microhardness change slowly (H_μ=6.5-7.0 ГПа).

Thus, substructure in Hadfield steel single crystals under cold rolling at room temperature determines predominantly by formation of thin micro- and nano- twins in several systems. Mechanical twinning is an effective mechanism for the hardening of Hadfield steel because the boundaries of this special type are difficult to degrade. A net of special boundaries (twins) during the early stages of deformation create an ultrafine-grained microstructure without the need for extremely high degrees of deformation.

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Protective coatings on samples made of ultrafine-grained titanium alloy

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Titanium and titanium alloys are considered as one of the most widespread structural materials due to their high strength-to-weight ratio and corrosion resistance. They are widely used in aerospace and engineering industries.

The problem of enhancement of service properties, life time and reliability of items and constructions imposes new requirements to structural titanium materials. In recent years ultrafine-grained structure formation in Ti alloys by severe plastic deformation (SPD) techniques has been widely used to increase properties of these alloys. Ultrafine-grained Ti alloys are highly promising for production of gas-turbine engine parts, in particular rotating blades of low-pressure compressors. Application of nanostructured ingots during shaping of blades enabled to carry out isothermal forging in the conditions of low-temperature superplasticity, i.e. at temperatures of 600…650^{o}C. Firstly, such a temperature range allows using cheaper materials for a die-set and reducing energy resources. Secondly, the thickness of the oxide surface layer of an ingot decreases in this temperature range that reasonably reduces the extent of subsequent mechanical processing.. However, protective coatings, in particular made of TiN, are conventionally used in such items operating in extreme conditions. But in respect to UFG Ti alloys the application of protective coatings has not been investigated thoroughly.

In the present work the following was investigated:

1) The structure of TiN coating applied on the surface of initial coarse-grained and ultrafine-grained titanium alloy VT-6 by ion-plasma method.

2) Effect of coating and ion implantation on the change in the UFG structure of the alloy VT-6.

Two techniques were used to form a UFG structure:

1) ECAP (equal-channel angular pressing)

2) HPT (high pressure torsion)

The UFG structure: a) after HPT; b) after ECAP is shown in Fig. 1.

a) *b)*

Fig. 1. The UFG structure: a) after HPT; b) after ECAP

The formation of a UFG structure can be observed in both states, but after HPT a mean grain size is approximately 150 nm, and after ECAP it is about 250 nm.

The electron microscope investigations were also used for studying a coating structure. It has been established that TiN coatings have also a UFG structure, whereas the features of this structure were sensitive to the alloy micro structure. The hardness of the alloy was measured after coating application and according to these measurements the hardness increases near the coating. The effect of coating process on the alloy structure is confirmed by these data and requires further investigations.

Therefore, the coating application on UFG titanium alloy has a number of features related to interaction of coating and base material.

Acoustic emission (AE) source location methods based on wavelet transform

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In this paper, the acoustic signals (Fig.1) are analyzed using the method of wavelet transform (WT), by which the arrival time differences of one frequency component in one mode are obtained.

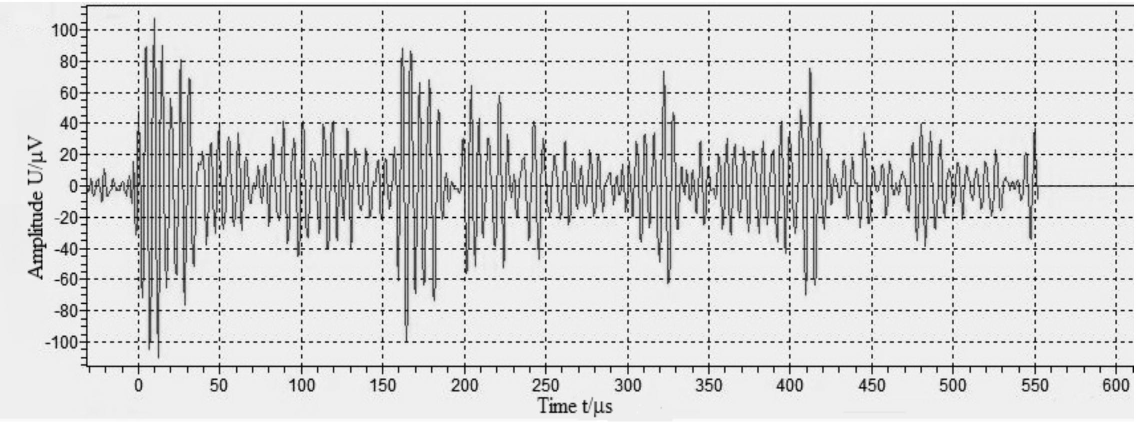

Fig. 1. Wavaform of lead break AE signals received sensor

The (WT) has been introduced to the time-frequency representation of transient waves propagating in a dispersive medium. For wave propagation in dispersive media, the accuracy of source location can be improved by using the time differences of different frequency components in one mode. J. Jingpin, W.Bin and H. Cunfu [1] applied the WT to an AE source location using plate waves. For its good resolution both in time and frequency domains, the Gabor wavelet has been used to the analysis of AE signal. It has been shown that one frequency component in the output signal can be extracted from the amplitude of the envelope of WT:

$$|WT_u(x,t)| = \sqrt{2a}\left|\hat{\psi}_g(a\omega_c)\right|\left[1+\cos\left(2\Delta kx - 2\Delta\omega b\right)\right]^{1/2}$$

Where $|WT_u(x,t)|$ is the magnitude of WT of signal x, $\hat{\psi}_g(\omega)$ is the Fourier transform of the basic wavelet, the parameters a and b stand for scale and shift of the basic wavelet, k is the wavenumber corresponding to frequency ω. Its methods indicate that the magnitude of WT takes its maximum value at $f = {}^{1}/_{a} = {}^{\omega_c}/_{2\pi}$ and $b = ({}^{\Delta k}/_{\Delta\omega})x = {}^{x}/_{c_g}$. Therefore, the location of the peak of magnitude on (a, b) plane corresponds to the arrival time $b = {}^{x}/_{c_g}$ of the wave on the frequency of $f = {}^{\omega_c}/_{2\pi}$.

In this technique, by the WT of acoustic signals, the arrival times of one frequency component in one mode can easily be obtained.

Further development of this technique will have to concentrate on experiments conducted on complex structures and the determination of the direction of the acoustic source and sensor source in one sensor source location scheme. It is believed that further investigation into the possibilities of these techniques will lead to improve analysis and calculated quantitative AE results.

References:

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The effect of equal channel angular pressing on structure and mechanical properties of low-carbon Fe-Mn-Ti-V-C and Fe-Mo-Nb-V-C steels

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The influence of equal channel angular pressing (ECAP) on mechanical properties and structure of low-carbon steels 10G2FT (Fe-1.12Mn-0.08V-0.07Ti-0.1C, wt.%, quenched from 1180°C after 30 min.) and 06MBF (Fe-1Mo-0,1V-0,1Nb-0,06C, quenched from 920°C after 0.5h and annealed at 670°C for 1 hour) was investigated. The ECAP was performed by route Bc, Φ=120°, for 4 passes at 400°C for 10G2FT steel and for 6 passes at 300°C for 06MBF steel.

After quenching, a structure of 10G2FT steel was martensitic one with thickness of plates ~ 0.15 μm (Fig.1a). In steel 10G2FT, there were Fe_3C carbides located along the boundaries of martensitic plates and fine-dispersed VC, TiC (~5-10 nm) and large spherical Fe_3C (~60 nm) carbides inside of grains. Steel 06MBF in initial state was composed of martensitic plates (thickness ~0.4 μm) and a globular ferrite (~0.8 μm) (Fig.1c). Structure of 06MBF steel enclosed coarse carbides of Fe_3C, M_6C, V_8C_7 (~90 nm) which are situated predominantly on the boundaries of grains and martensitic plates, and particles of Fe_3C (15-20 nm) inside of grains were revealed.

An ultrafine-grained structure (UFG) with the average grain size of 300 nm was formed in both steels after ECAP (Fig.1b, d) (SAED from the areas of 1,4 μm^2 (fig.1 b) and 0,5 μm^2 (fig.1d)). The SAED pattern showed that ECAP resulted in milling of initial structure. After ECAP, the reflections became more diffused compared to the initial state. The extinction contours in grains existed (Fig.1b, d) because of grains are in non-equilibrium state. ECAP provided a strong fragmentation ether initial structure or carbides. The carbides of Fe_3C (~ 35nm), $M_{23}C_6$, M_6C in steel 10G2FT and ultra-fine particles of Fe_3C (~ 2-3 nm), Fe_3C, V_8C_7 (~ 70 nm) in steel 06MBF were observed.

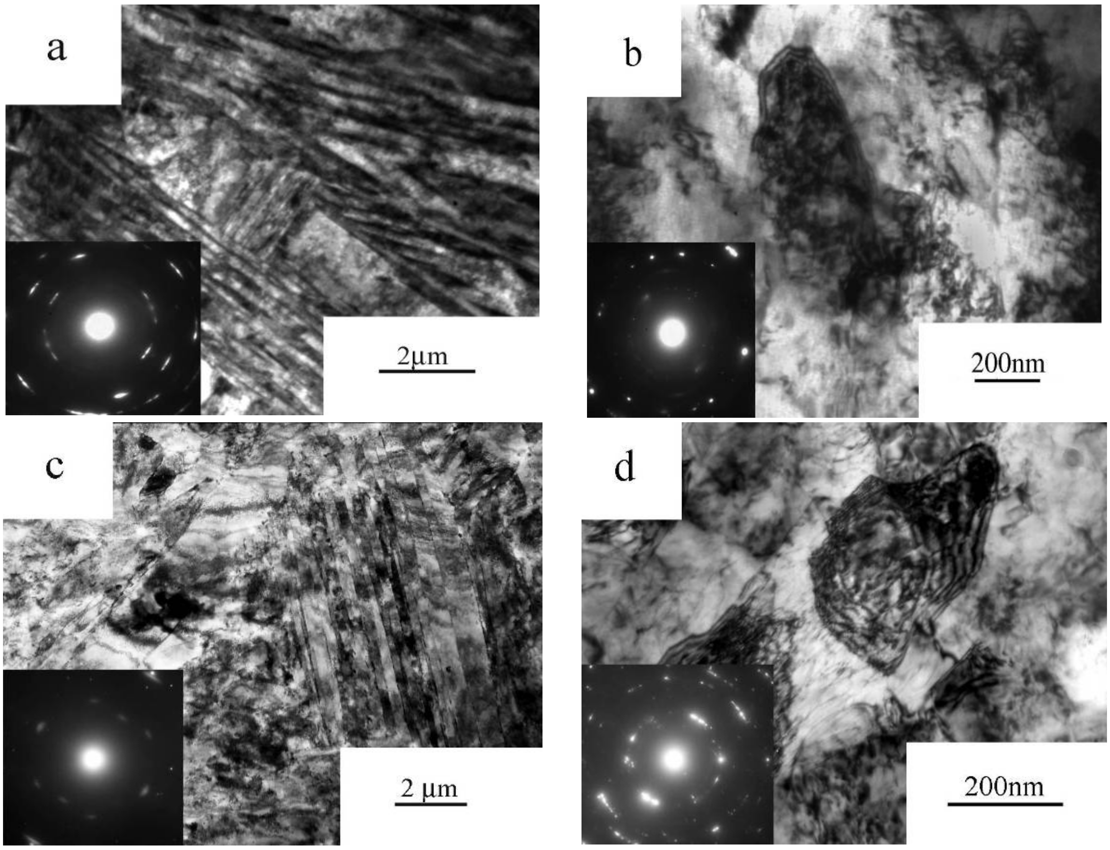

Fig. 1. TEM microstructures of the low-carbon steels 10G2FT (a, b) and 06MBF (c, d) before (a,c) and after ECAP (b, d).

The increasing of steel microhardness after ECAP was found out. The microhardness of both steels after ECAP is 3.9 MPa in spite of differences in initial values of mechanical properties (3,7GPa for 10G2FT and 2,1GPa for 06MBF). Steels possess thermal stability of microhardness and ultrafine-grained structure up to 500°C.

We can conclude that ECAP resulted in formation of UFG structure (300 nm) with a system of fine carbides which is of great importance for increasing of mechanical properties and thermal stability of steels investigated.

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Thermal stability of ultrafine-grained structure processing of 18-10 stainless steel using high-pressure torsion

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The wide application of stainless steels is restricted by their non-sufficient strength properties. Ultrafine-grained structure processing (UFG) (D_3 <10 μm) is one of the most efficient method for improving these properties in traditionally heat-strengthened chromium-nikel alloys. Our former studies dealing with properties of submicrocrystalline corrosion stable austenitic steels processed by multiple step forging under conditions of cryogen temperatures confirmed this opportunity.

The most efficient method for UFG structure processing bulk billets of metals and alloys is severe plastic deformation (SPD). Torsion under pressure is a method providing processing marginal states in alloys due to high strains.

The present work considers the results of UFG structure thermal stability studies. Ultrafine-grained (UFG) states was obtained in popular 18-10 stainless steel by severe plastic deformation (SPD) by means of high-pressure torsion (HPT). This research is devoted to applicability borders definition of that UFG stainless steel. By complex research critical temperature was determined. At the heating more higher critical temperature unique properties and structure are lost. It is shown that critical temperature not depend on HPT temperature and equal 500°C at the both HPT temperatures (20º C and 400º C).

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Consolidation of 2 vol. % carbon nanotube reinforced Al matrix nanocomposites via equal channel angular pressing

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Keywords: *ECAP, SPD, CNT, Composite, Aluminium matrix.*

Carbon nanotubes (CNTs) have been considered as an ideal reinforcement to improve the mechanical performance of monolithic materials. However, the CNT/metal nanocomposites have shown lower strength than expected. In this study, the CNT reinforced AL matrix nanocomposites were fabricated by ECAP. ECAP (equal channel angular pressing), the most promising method in SPD, was used for the CNT-Al powder consolidation. The powder ECAP processing with one, two, four and eight route C passes was conducted at room temperature. The effect of ECAP on consolidation behavior of powder, microstructure and mechanical properties of subsequent compacts in comparison to conventional direct extrusion (DE) is presented. It was found by mechanical testing of the consolidated 2 vol. % CNT-Al that high mechanical strength could be achieved effectively as a result of the Al matrix strengthening and improved particle bonding during ECAP. It is confirmed that the key issue to enhance the strength of CNT/metal nanocomposite is homogeneous distribution of CNTs.