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INVESTIGATION OF THE PRECIPITATION MECHANISM IN THE SEVERELY DEFORMED AND AGED 2024 Al-ALLOY

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ABSTRACT

Isothermal age hardening process at 190°C was applied to the two 2024 Al-alloy specimen groups consisting of the samples severely deformed by one-pass equal channel angular pressing (ECAP) and the samples without pre-deformation. The morphology, size, distribution of precipitates and the hardness values at different levels of aging were determined by using transmission electron microscopy and micro hardness. It was observed that, in comparison with the solutionized-aged specimens, ECAPed samples reach the peak-hardness remarkably earlier during aging, and they have smaller and well-distributed precipitates.

Keywords: *Severe plastic deformation, Aging, Al 2024, Precipitate*

AŞIRI PLASTİK DEFORMASYON VE YAŞLANDIRMA İŞLEMLERİ UYGULANAN 2024 Al-ALAŞIMINDA ÇÖKELTİ OLUŞUM MEKANİZMASININ İNCELENMESİ

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ÖZET

Eş Kanallı Açısal Pres (EKAP) yöntemiyle tek paso aşırı deforme edilen ve herhangi bir deformasyon uygulanmayan 2024 Al-alaşımı numunelerden oluşan iki deney grubuna, 190°C'de farklı sürelerle yaşlandırma işlemi uygulanmıştır. Elde edilen numunelerde, geçirimli elektron mikroskobu ve mikro sertlik ölçümleri yapılmıştır. Kırınım paternleri ve koyu alan analizi yardımıyla farklı yaşlanma basamaklarında, çökeltiler boyut, şekil ve dağılım yönünden karşılaştırmalı olarak incelenmiştir. Deforme edilmeden yaşlandırılmış numunelerle karşılaştırıldığında, EKAP ile deforme edildikten sonra yaşlandırılmış numunelerde çökelti boyutlarının çok daha küçük olduğu ve yaşlandırma sırasında daha kısa sürede maksimum sertliğe ulaşıldığı gözlemlenmiştir.

Anahtar Kelimeler: *Aşırı plastik deformasyon, Yaşlandırma, Al 2024, Çökelti*

1. INTRODUCTION

Al 2024 alloys are commonly used in aerospace industry due to their light weight mechanical properties [1]. They are age-hardenable Al alloys with 3.8-4.9 wt% Cu and 1.2-1.8 wt%Mg as major alloying elements with minor constituents of Fe, Mn, Zr and Cr [2]. Severe plastic deformation [3] and age-hardening [4] are two most-common ways to improve the mechanical properties of this alloy system. Equal channel angular pressing (ECAP), one of the most applicable and practical severe plastic deformation techniques, has been considered to be a short cut to obtain higher strength by introducing high density of defects. Attributed to high ductility of aluminum alloys and low cost of the process, ECAP is well suited for AA2024 alloys as compared to other metallic alloy systems [5,6]. Although ECAP system is simple and easy to apply, the mechanisms involved are matter of question. Since excess amount of deformation induced defect structure above a certain limit, general principles of cold deformation do not easily apply in ECAP. The high level of deformation by ECAP may lead to multiply the vacancies and dislocations especially during the first pass. The defects induced by ECAP have significant importance on aging behavior [7]. Several studies have discussed the mechanism of precipitation and the sequence of the transformation [1,8-10] in AA 2024. The most common sequence was found to be:

α (Saturated Solid Solution) ➔ Guinier-Preston-Bagaryatsky zones (GPB) ➔ S″➔S′➔S

There are still unclarified aspects of aging of aluminum alloys. For example, a recent study claiming that S″ phase is just a variant of S′ [10] and yet another study investigating the mystery of GPB zones and the effect of vacancies on precipitation [3]. When combined with the severe plastic deformation conditions of ECAP, kinetics of aging process even gets more complicated. Studies on ECAP and aging relationship on Al alloys showed that precipitates are more evenly distributed and their sizes are considerably smaller if ECAP was applied after the aging process [11,12]. Some other studies have stated that the optimum aging temperature should be relatively low on the order of 80°C yet some other claim the higher temperatures i.e. above 100 °C are more practical and suitable for an effective aging process [11,13]. In the light of literature, it is obvious that there is a need to understand the precipitation of Al 2024 alloy followed by ECAP and the present study aims to investigate ECAP-aging relationship at some extent. The change in precipitate morphology, distribution, and aging characteristics with and without plastic deformation is investigated by using various transmission electron microscopy techniques.

2. EXPERIMENTAL PROCEDURE

Two group of Al 2024 samples were solution heat treated for 1 h at 495°C and then quenched to 0°C salty ice-water mixture. ECAP die of 120° was used for 18 mm diameter sample for a single pass. A sample at the center of the specimen was cut at an angle of 46° to observe the behavior at the same shear region. Interrupted aging was applied to both ECAPed and only solutionized samples in oil bath at 190°C for 5-65 min and 1-24 h, respectively. Micro-hardness tests were carried out at Shimatzu HMV-2 test machine. TEM samples were taken for peak aging and over aging times of both samples. Samples that belongs to, solutionizing+11.5 h aging and solutionizing+ECAP+59 min aging, (peak-aging for just solutionized and ECAP processed Al 2024, respectively) were thinned down to below 100μm, and through dimpling a 10 μm center was obtained. Etchant of 25% nitric acid, 75% methanol was used at -30°C for electro-polishing. TEM was performed using a JEOL JEM2100F. Dark field (DF), bright field (BF) images coupled with selected area diffraction patterns (SADP) were collected at various stages of aging.

3. RESULTS AND DISCUSSION

The micro-hardness tests show that the peak aging time for solutionized sample is 11.5 h as compared to 1 h for ECAP processed sample as seen in Figure 1. A drastic drop in peak aging time, as low as one tenth of just solutionized Al 2024, has been observed. The reason of increased phase transformation kinetics may be attributed to the level of defect both in vacancies and dislocations. Pre-peak aging can be considered as 9 h for just solution heat treated and 50 minutes for the ECAP processed samples. When phase transformation is considered this decrement should also be leading to differences in the precipitates themselves. To compare the transformation stages, the TEM images of the precipitates are presented in Figures 2 and 3, for 9 h and 50 minutes pre-peak aging times corresponding to the solutionized and ECAPed Al 2024, respectively. The precipitates are observed as long needles in the solutionized Al 2024 whereas in the ECAP processed one, the precipitates are smoother in shape and much smaller in size.

In Figures 4 and 5, precipitates are observed for peak aged Al 2024 alloy of solution heat treated and ECAPed samples, respectively. In the solutionized sample with no plastic deformation, the length of the precipitates is found to increase and in ECAPed sample well distributed relatively small precipitates are observed. The comparison of images indicates that rather than a growth of already existing precipitates, nucleation of newly formed precipitates are observed since the size range of precipitates remains the same as pre-peak aged sample. When the size ranges are compared for solution heat treated sample an increase from 300 nm to 500 nm is observed for the length of the precipitates. On the other hand for ECAP processed alloy the precipitate lengths remain in the range of 15-30 nm. The reason for the change in the distribution and sizes of the S precipitates is probably the ease of precipitation due to the increased nucleation sites for precipitation from the defects within the crystal structure (i.e. dislocations). It can be concluded that ECAP process yields to not only finer precipitate size, but also a more homogeneous distribution of the precipitates in Al 2024 alloy as shown in Figure 5.

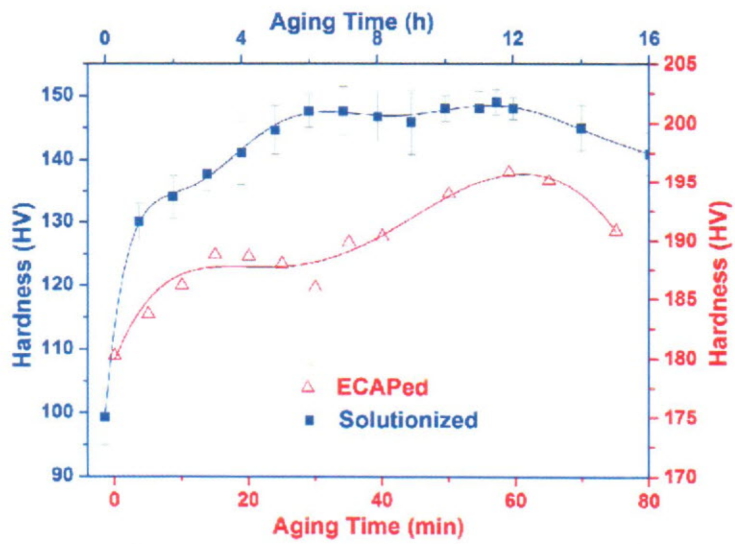

Figure 1. Time vs. micro-hardness results of interrupted aging curves for solution heat treated and solution heat treated with 1 pass ECAP processed Al 2024 alloy at 190°C. The peak aging time for solution heat treatment is 11.5 h, solution heat treatment and ECAP is 1 h.

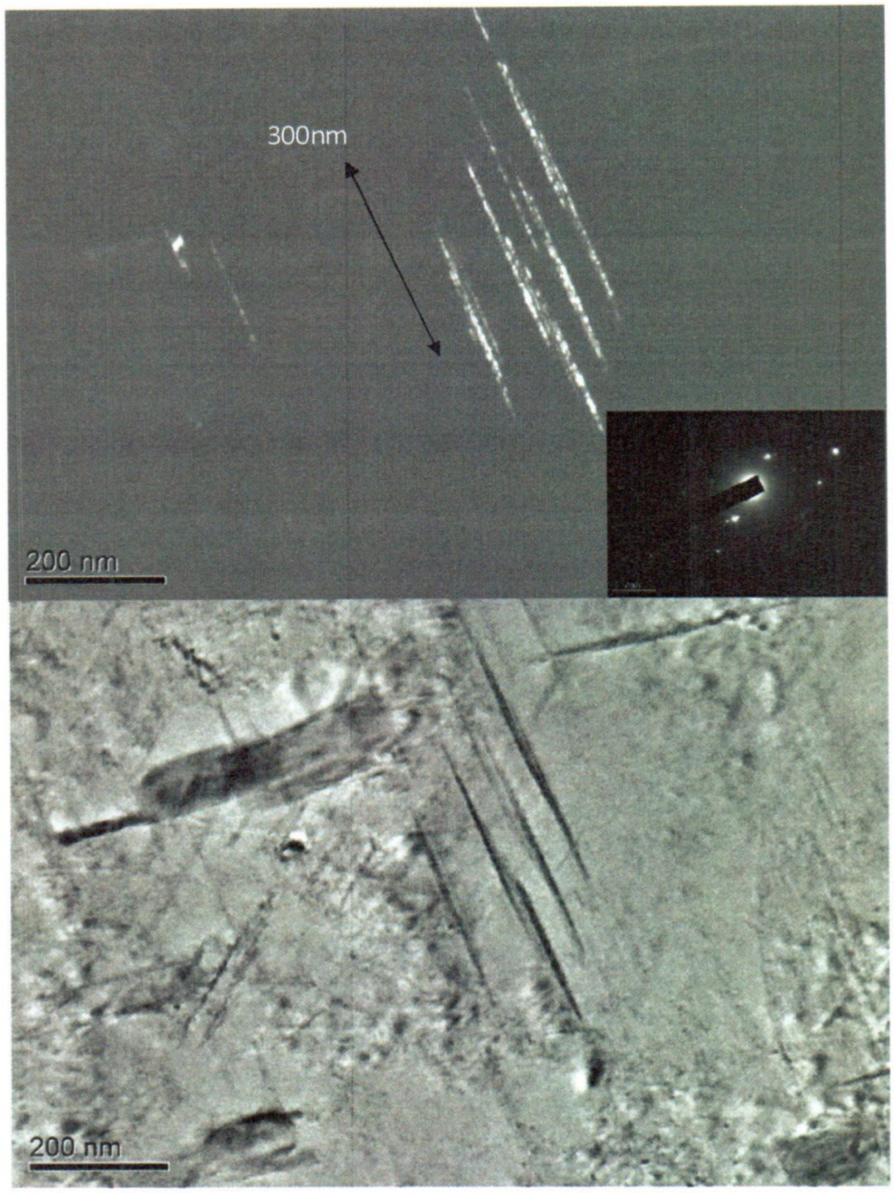

Figure 2. Electron microscope images Al 2024 alloy solution heat treated at 495 °C for 1 h, quenched and aged at 190°C for 9 h in oil bath.
9 hours is pre-peak aging time.
(a) dark field, (b) bright field.
(b) The needle-like features are the precipitates of S′ or S phase of $CuMgAl_2$.

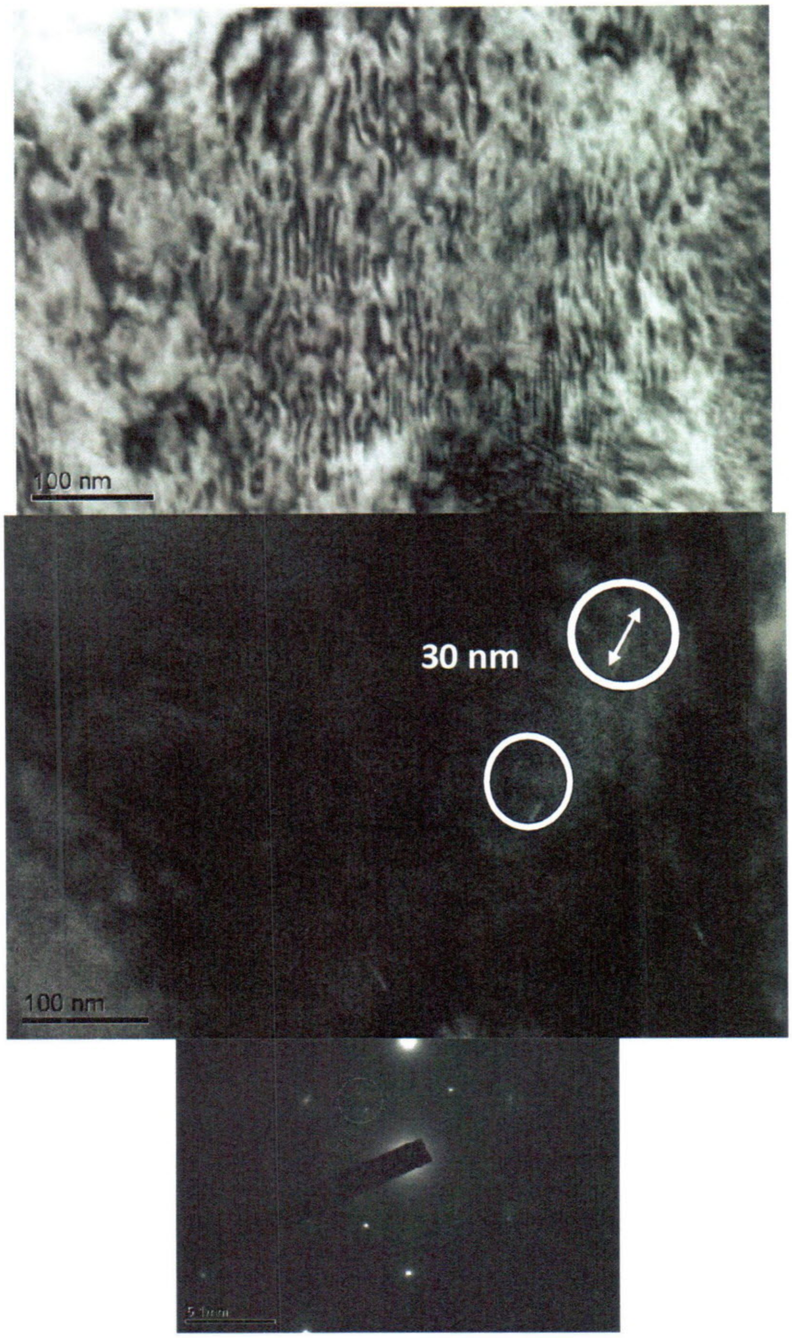

Figure 3. Electron microscope images Al 2024 alloy solution heat treated at 495 °C for 1 h, quenched and ECAP processed then aged at 190°C for 50 minutes in oil bath.
50 min is pre-peak aging time
(a) dark field, (b) bright field, (c) SADP.
The circled particles are precipitates of S′ or S phase of $CuMgAl_2$.

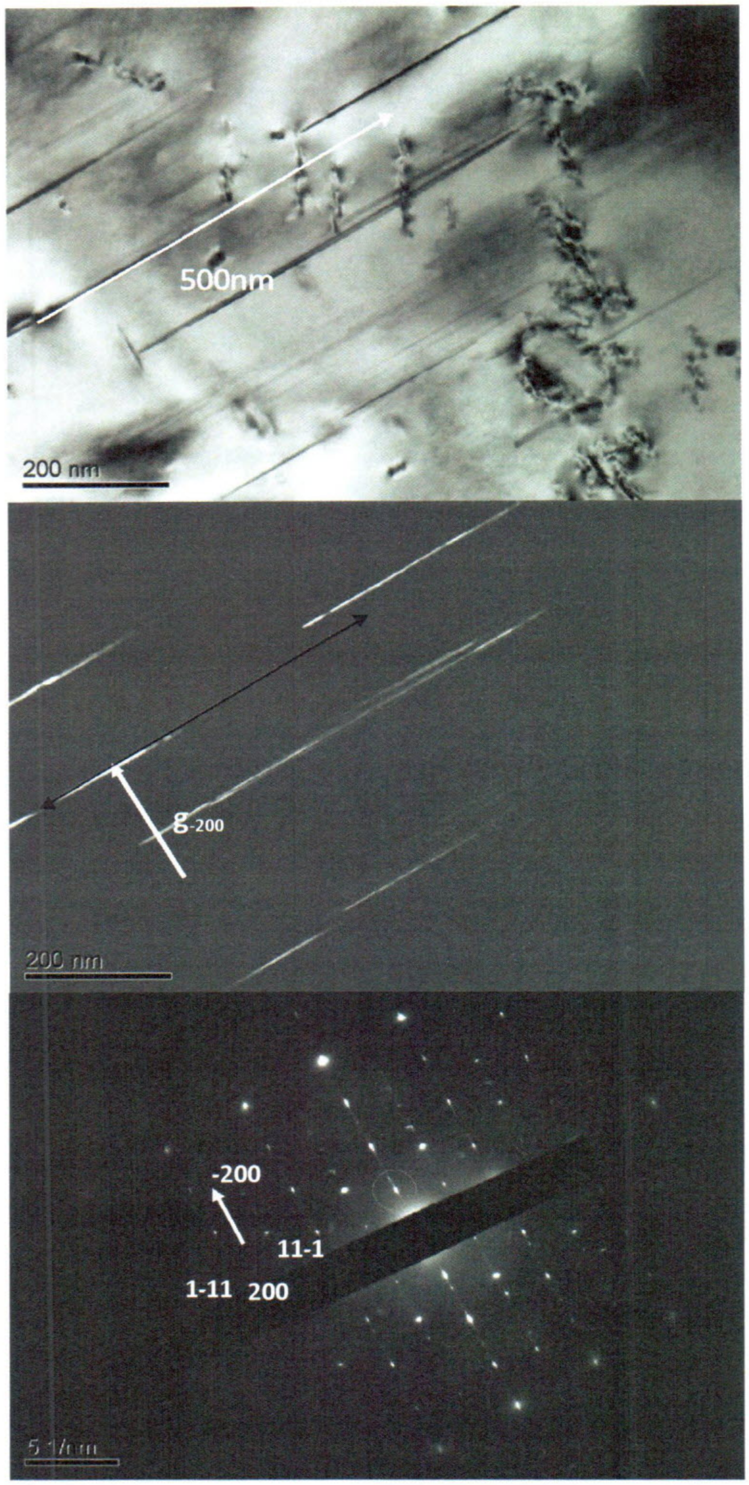

Figure 4. Electron microscope images Al 2024 alloy solution heat treated at 495 °C for 1 h, quenched and aged at 190°C for 11.5 h in oil bath. 11.5 h is peak aging time.
(a) dark field, (b) bright field.
The needle-like features are the precipitates of S′ or S phase $CuMgAl_2$.

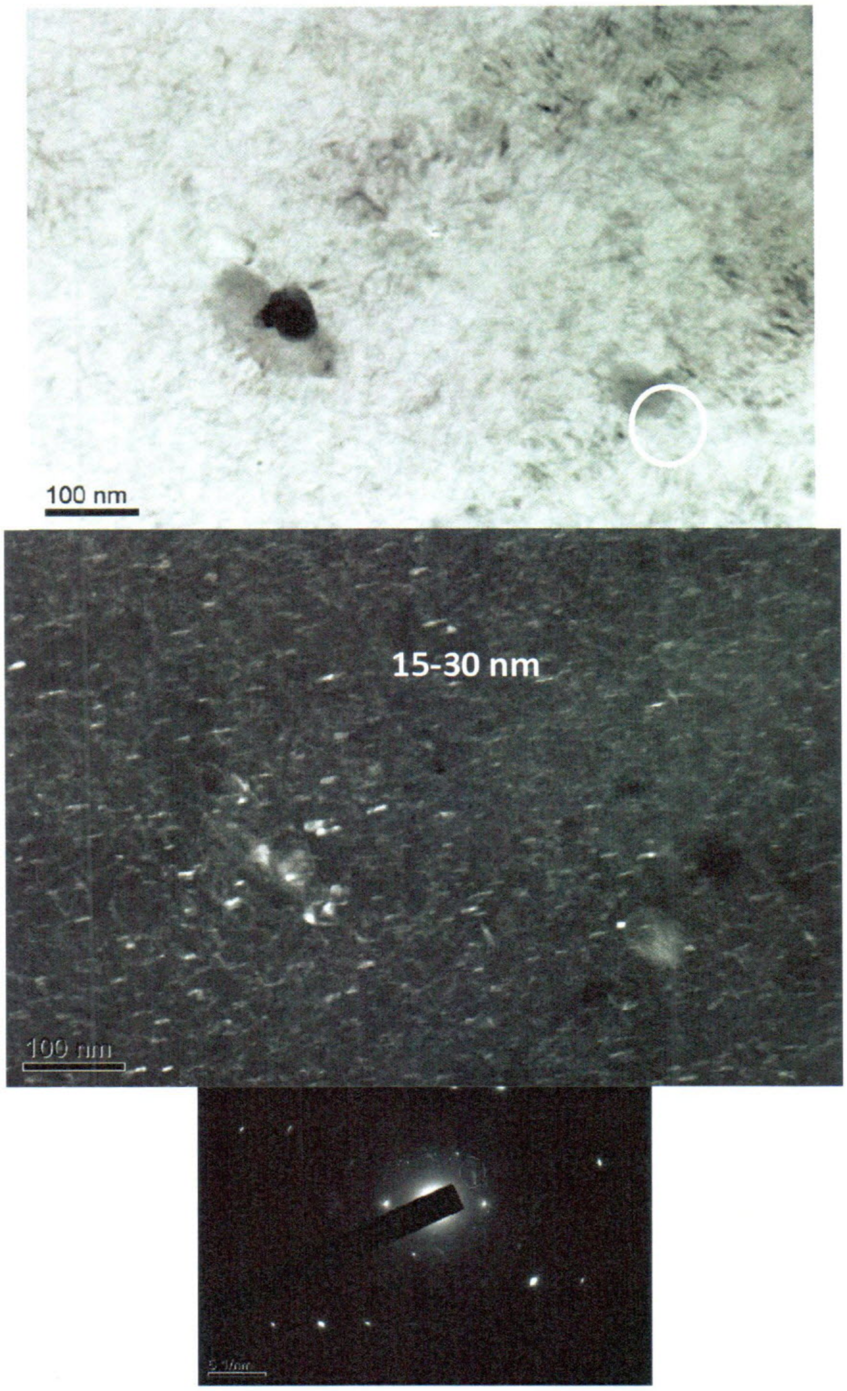

Figure 5. Electron microscope images Al 2024 alloy solution heat treated at 495 °C for 1 h, quenched and ECAP processed then aged at 190°C for 60 minutes in oil bath.
60 min is peak aging time.
(a) dark field, (b) bright field, (c) SADP.
The circled particles are precipitates of S′ or S phase of $CuMgAl_2$.

4. CONCLUSIONS

Age hardening process at 190°C was applied to the two 2024 Al-alloy specimen groups consisting of the samples severely deformed by one-pass equal channel angular pressing (ECAP) and the samples without pre-deformation. The morphology, size, distribution of precipitates and the hardness values at different levels of aging were determined by using TEM and micro-hardness tests. The followings can be concluded from the results of this specific study.

ECAPed specimens were found to reach the peak-age before the solutionized and aged specimens with smaller and well distributed precipitates. Peak aging time at 190°C of ECAP processed Al 2024 is 1 h whereas for solution heat treatment it is 11.5 h.

Regarding the dark field images of S phase precipitates

- The precipitates observed after pre-peak aging for ECAPed sample is finer in size and smoother in shape as compared to pre-peak aged solutionized samples;
- Precipitates in peak-aged samples are 500 nm in size in solely solution heat treated specimens whereas they are 15-30 nm and well distributed in ECAPed Al 2024;
- ECAP process results in a finer distribution and smaller precipitates in Al 2024 alloy.

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