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Predicting the critical pre-aging time in ECAP processing of age-hardenable aluminum alloys

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ABSTRACT

Age-hardenable Al alloys may be successfully processed by equal channel angular pressing (ECAP) at room temperature, if the processing is carried out immediately after water quenching from the solution treatment temperature. It is important to estimate the critical time for any age-hardenable alloys, since after this time, ECAP processing will cause catastrophic cracking or segmentation at room temperature. In this study, ECAP processing was carried out on two age-hardenable Al alloys (2014 and 7075) at room temperature. The results demonstrated that the critical time could be predicted successfully by using tensile test curves related to different times after quenching. It is also shown that room temperature ECAP processing of these materials for more than a single pass is not possible and causes damage. However, a single pass will have significant effects on the strength of the material.

1. Introduction

Equal channel angular pressing (ECAP) is the most well developed of all potential severe plastic deformation (SPD) processing techniques, and is an effective tool for achieving grain refinement [1]. The yield strength of the material increases significantly after the first ECAP pressing, but further increase in strain induced by subsequent passes gradually increases the yield stress. On the other hand, the elongation to failure decreases considerably after the first pass and remains almost the same after subsequent passes [2].

In the last decade, many metals and alloys have been successfully processed by the ECAP technique but some materials such as hexagonal closed-packed (HCP) alloys and age-hardenable aluminum alloys are difficult to process by ECAP at room temperature. The formation of precipitates in the solution treated age-hardenable Al alloys leads to loss of deformability of the material [3]. So, segmentation or cracking of the material is expected. These problems may be avoided by increasing the processing temperature [4–12]. However an increase in the pressing temperature leads to a larger grain size and also enhances additional precipitation, and may lead to overaging of the material. Recently, Chinh et al. [3] developed a strategy for the processing of age-hardenable alloys by ECAP at room temperature and found that ECAP processing may be conducted successfully, without the formation of catastrophic cracking or segmentation, if ECAP processing is performed immediately after quenching from the solution treatment temperature or at least within a very short pre-aging time.

The objective of the present investigation is to develop a strategy to be used in ECAP processing to predict the "critical time" after quenching, in which ECAP processing may be conducted successfully without cracking or segmentation. The experiments were carried out using two age-hardenable alloys: a commercial Al 7075 and Al 2014 alloy which consists of lower amounts of alloying elements and leads to lower precipitation kinetics. The alloys were processed by ECAP at different pre-aging times up to the critical time in which cracking begins to occur. Also, in order to find a relationship between the tensile test curves and the critical time, tensile test was conducted at different times after solution treatment. The results demonstrated that the steps induced by the dynamic strain aging (DSA) disappear from the tensile test curve after a definite time, which is the same as the "critical time" after which ECAP processing cannot be conducted successfully. The results also showed that the second room temperature ECAP pass could not be processed, because DSA occurred after the first pass.

2. Experimental materials and procedures

2.1. Sample preparation

The chemical compositions of aluminum alloys 2014 and 7075 are given in Table 1. The ECAP facility had an internal angle of 90° and an angle of 20° at the outer arc of the curvature at the intersection of two parts of the channel, as indicated in Fig. 1, thereby giving an imposed strain of ~1 on each pass [1].

Polytetrafluoroethylene (PTFE) tape was used as the lubricant. The pressing was conducted at room temperature and constant displacement rate of 0.2 mm s^{-1}. The pressing force was monitored during ECAP. The samples were machined for ECA

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Table 1
Chemical compositions in wt.% of the alloys 2014 and 7075 used in this study.

Alloy	Cu	Mg	Zn	Si	Mn	Fe	Al
2014	3.85	0.82	0.02	0.43	0.67	0.25	Balance
7075	1.68	2.80	6.00	0.10	0.23	0.31	Balance

pressing with diameters of 10 mm, and were subjected to ECAP in two different states:

State A: the specimens were annealed for 3 h at 495 °C and 480 °C for alloy 2014 and 7075, respectively and furnace cooled prior to ECAP processing.

State B: the specimens were annealed for 3 h at the temperatures mentioned above, and then quenched in water (WQ). These samples were naturally aged at room temperature for different periods of time prior to being processed by ECAP, in order to estimate the critical time in which cracking begins to occur.

Repetitive pressings were undertaken on furnace cooled specimens up to a total of four passes, equivalent to a strain of ∼4, with the samples rotated about their longitudinal axes by 90° in the same sense between consecutive passes in the processing route designated B_c [1].

2.2. Tensile test

Cylindrical tensile specimens were machined from as-ECA pressed specimens. When considering the time spent during ECAP processing of quenched samples and further machining to prepare a tensile specimen, tensile test could be performed 30 min after quenching. Therefore, for comparison, the as quenched specimens with no further processing were also tested after 30 min of room temperature aging. In addition the furnace cooled specimens were tensile tested after pressing for four passes. The dimensions of these tensile specimens are given in Fig. 2.

Also, some specimens were determined to be tested at different times after quenching without any subsequent pressing to estimate the relation between the obtained tensile curves and the critical time.

All of the tensile tests were performed at room temperature using an Instron type testing machine operating at a constant rate of crosshead displacement with an initial strain rate of 2×10^{-3} s^{-1}.

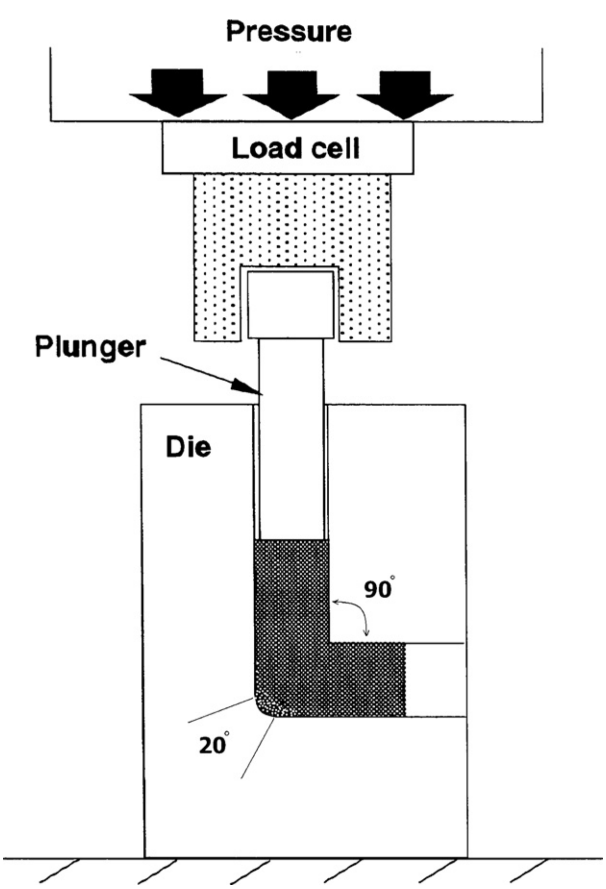

Fig. 1. Schematic illustration of used ECAP facility.

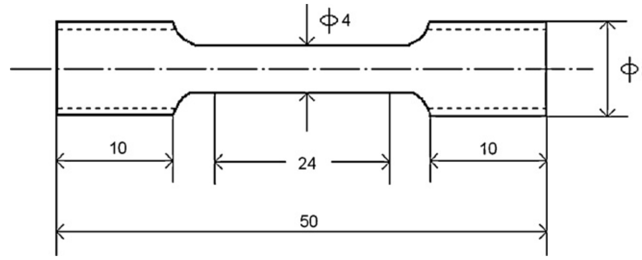

Fig. 2. Dimensions of tensile specimens (mm).

2.3. Microstructure characterizations

The fracture surfaces of the tensile test specimens and the segmented specimens were characterized by a Cambridge-S360 type scanning electron microscope (SEM) equipped with EDX.

3. Results and discussion

3.1. ECAP processing

In order to find the critical time, ECAP processes were carried out at room temperature on the specimens after natural aging for different periods of time. Samples of Al 2014 alloy were ECAP pro-

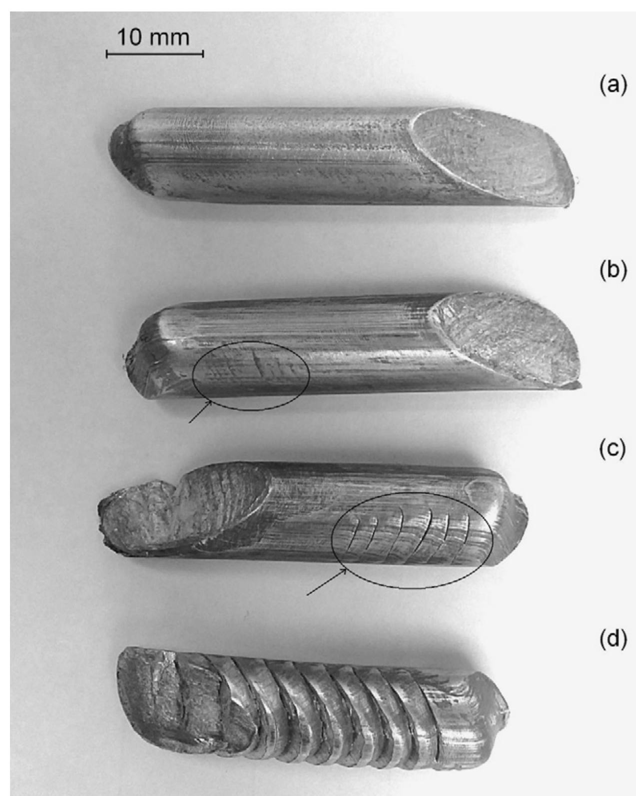

Fig. 3. The surface topography of the ECAPed Al 2014 alloy processed for one pass after quenching; ECAP conducted after (a) 30 min of natural aging, (b) 45 min of natural aging, (c) 50 min and (d) 180 min of natural aging.

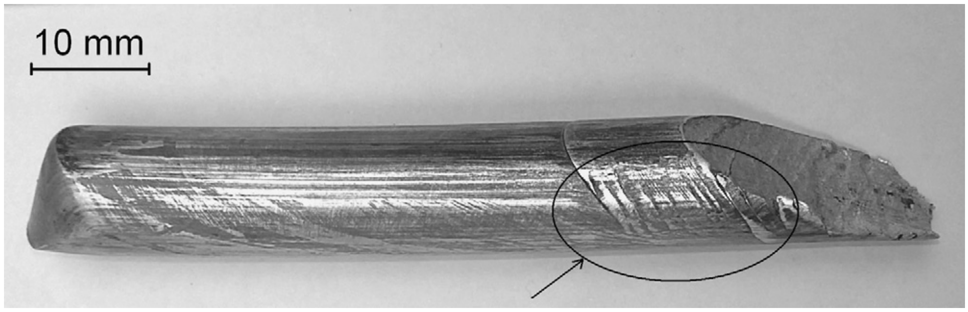

Fig. 4. An Al 2014 alloy, ECAPed until half length immediately after quenching and another half processed after one hour of room temperature natural aging.

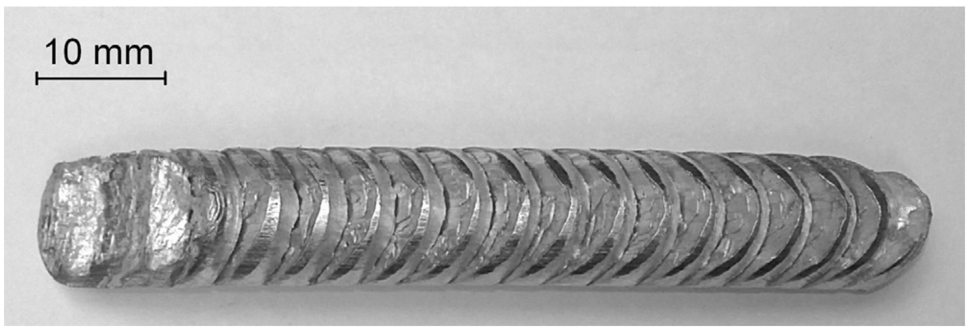

Fig. 5. The Al 2014 specimen, conducted to the second pass of ECAP processing in supersaturated state.

cessed successfully in the first 45 min of natural aging, but after that, cracking and segmentation begins to occur, as shown in Fig. 3.

Also, one specimen was ECAP processed until half length after a short pre-aging time, and another half was pressed after one hour. The first ECAPed half of the specimen was processed successfully, but the other half cracked (Fig. 4). This result confirms the effect of precipitation in crack formation during ECAP processing, too. Therefore, these cracks are not due to the shear bands, which are always formed in ECAP processing, but are the consequence of the presence of precipitates combined with shear bands.

Several specimens were processed by ECAP immediately after quenching. The specimens were then machined and conducted to the second ECAP pass. The second pass was carried out about 20 min after quenching, and catastrophic cracking occurred, as shown in Fig. 5.

When the solution treated material was subjected to imposed strain, the DSA resulted in formation and the growth of precipitates, leading to loss of formability of the material. Therefore the second pass could not be performed. This type of cracking can also be seen when the material is subjected to ECAP processing after a long room temperature natural aging (Fig. 3d). Thus a major reason of catastrophic cracking during the second pressing is a higher dimension of precipitates as a consequence of DSA, and strain hardening is a minor effective factor. Fig. 6 shows the force-displacement curves of the processing material during the first (a) and the second pass (b). The force-displacement curve of the first pass was smooth, because the material flowed continuously without segmentation. But during the second pass, the material could not flow as the precipitates were strong enough to suppress the dislocation motion. So the load rises up until cracking occurs and the material flows

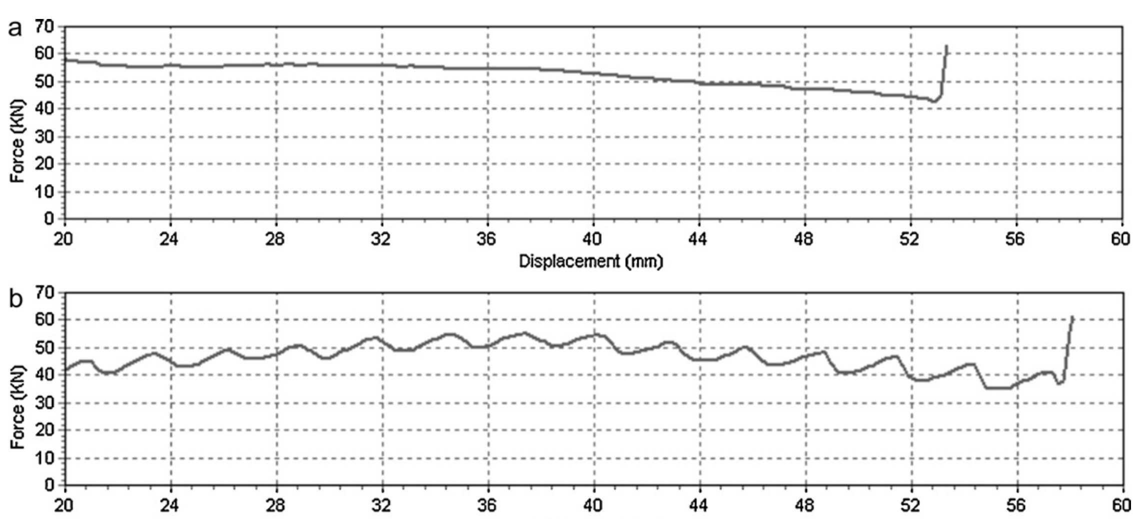

Fig. 6. Force-displacement curves of Al 2014 alloy during first (a) and second (b) ECAP pressing.

Fig. 7. Samples of Al 7075 alloy, ECAPed after (a) less than 3 min and (b) 10 min of natural aging.

by segmentation and the load decreases. Repeating this cycle gives the curve shown in Fig. 6b.

ECAP processing of furnace cooled specimens was performed successfully for four passes. Thus strain hardening cannot be considered as a major reason of failure during the second pressing of solution treated material. ECAP processing of used commercial Al 7075 alloy was different. Also, when ECAP was processed immediately after quenching, the samples cracked. This is shown in Fig. 7.

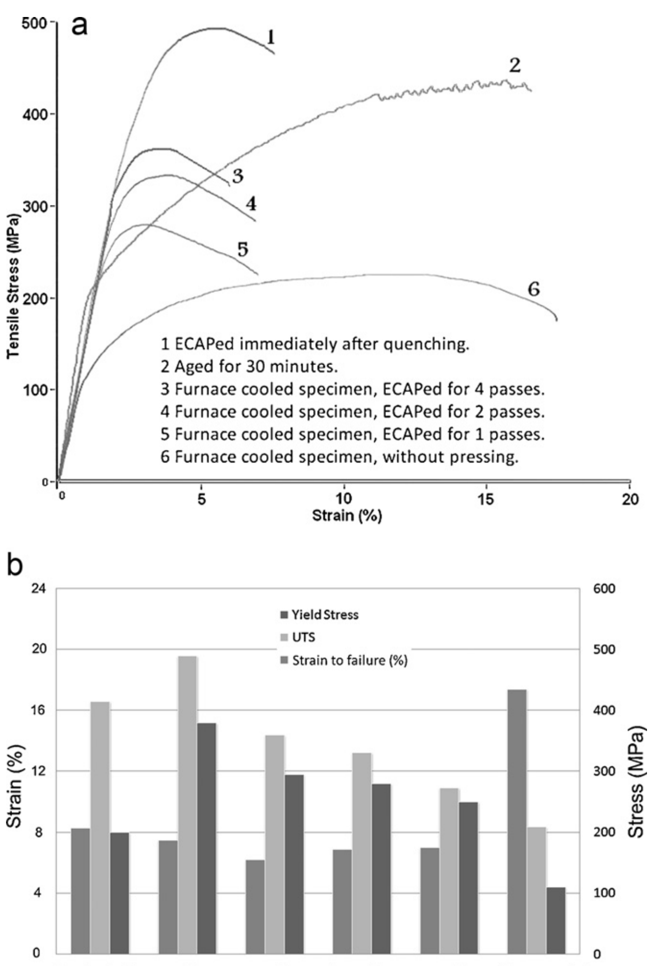

Fig. 8. (a) Tensile curves of Al 2014 ECAPed specimen in supersaturated state, aged specimen and furnace cooled specimen without pressing and ECAPed furnace cooled specimens and, (b) tensile properties of the specimens.

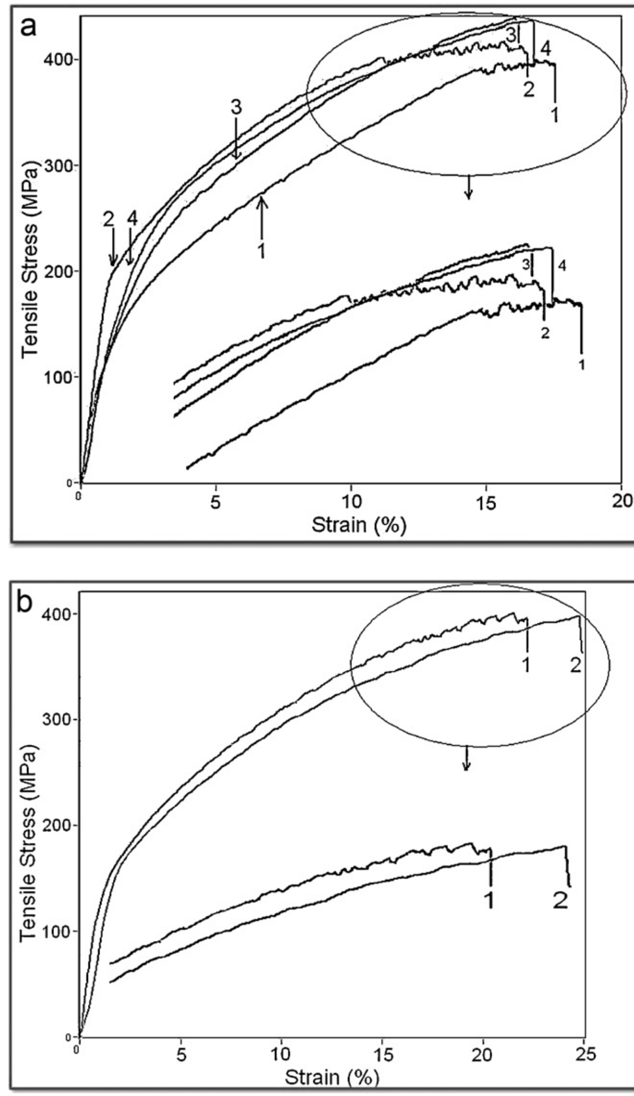

Fig. 9. Tensile curves of specimens after different aging times. (a) Al 2014 alloy after 15, 30, 45 and 60 min of natural aging, illustrated in curves 1, 2, 3, and 4, respectively and (b) Al 7075 alloy. The tests were performed immediately after quenching (curve 1) and 3 min after quenching (curve 2).

But the furnace cooled samples of the alloy were ECAP processed successfully for four passes.

The Al 7075 alloy has greater amounts of alloying elements. Thus the kinetics of precipitation is faster than the Al 2014 alloy. Therefore the precipitates form and grow rapidly to the size that suppresses moving dislocations hence cracking occurs during ECAP. The critical time will be reduced for this composition but by decreasing the alloying elements, critical time will increase.

3.2. Tensile behavior

ECAP processing of samples required about 10 min. Considering the time spent for machining, the tensile test was carried out 30 min after quenching. The engineering stress–strain curves of the ECAP processed 2014 Al alloy, unECAPed but water quenched alloy after 30 min of natural aging, and unECAPed and ECAPed furnace cooled alloy are shown in Fig. 8.

The curves show that processing in a supersaturated state (curve 1) leads to the highest yield strength and the ductility of the material has no significant reduction compared with the furnace cooled specimen, ECAPed for 4 passes. The yield strength achieved

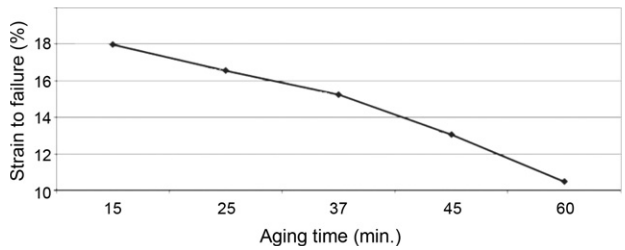

Fig. 10. Reduction of ductility by increasing aging time in Al 2014 alloy.

was 378 MPa which is 52% higher than the yield strength of furnace cooled ECAP processed after single pass that was 248 MPa (curve 5) and 90% higher than the yield strength of unECAPed water quenched material that was 200 MPa (curve 2). Considering unECAPed furnace cooled material as the reference material, there is an increase of 140 MPa and 90 MPa in yield strength, which is achieved by one pass of pressing of the furnace cooled specimen and natural aging without any subsequent processing, respectively. Using the superposition rule, and by subtracting these two values from an increase of 270 MPa in yield strength achieved by processing in a supersaturated state, the significant effect of DSA in the strengthening of the processed material can be found. The strain induced by ECAP processing results in the formation of finer and more homogenous precipitates that prevent the dislocation motion. Also, the increase of dislocation density during ECAP processing is another strengthening mechanism. The incorporation of these two mechanisms leads to significant strengthening.

The yield strength achieved by a single pass of room temperature ECAP of the solution treated material is higher than the furnace cooled material after 4 passes. This result clearly shows the role of precipitation hardening in the strengthening of ECAPed material in the supersaturated state.

ECAP processing showed that the critical time was about 45 min for Al 2014 alloy and that the Al 7075 alloy had a few minutes of critical time. Tensile tests were performed after different aging times. The curves are shown in Fig. 9.

The serrated yielding in supersaturated Al alloys can be seen at the first stages of aging, but by spending time, the steps gradually disappear from the tensile curve. The time at which the serrated yielding vanishes is named "t^*". Tensile curves of the Al 2014 alloy show that after about 45 min, the steps disappear. So the t^* is the same as the critical time achieved by ECAP process-

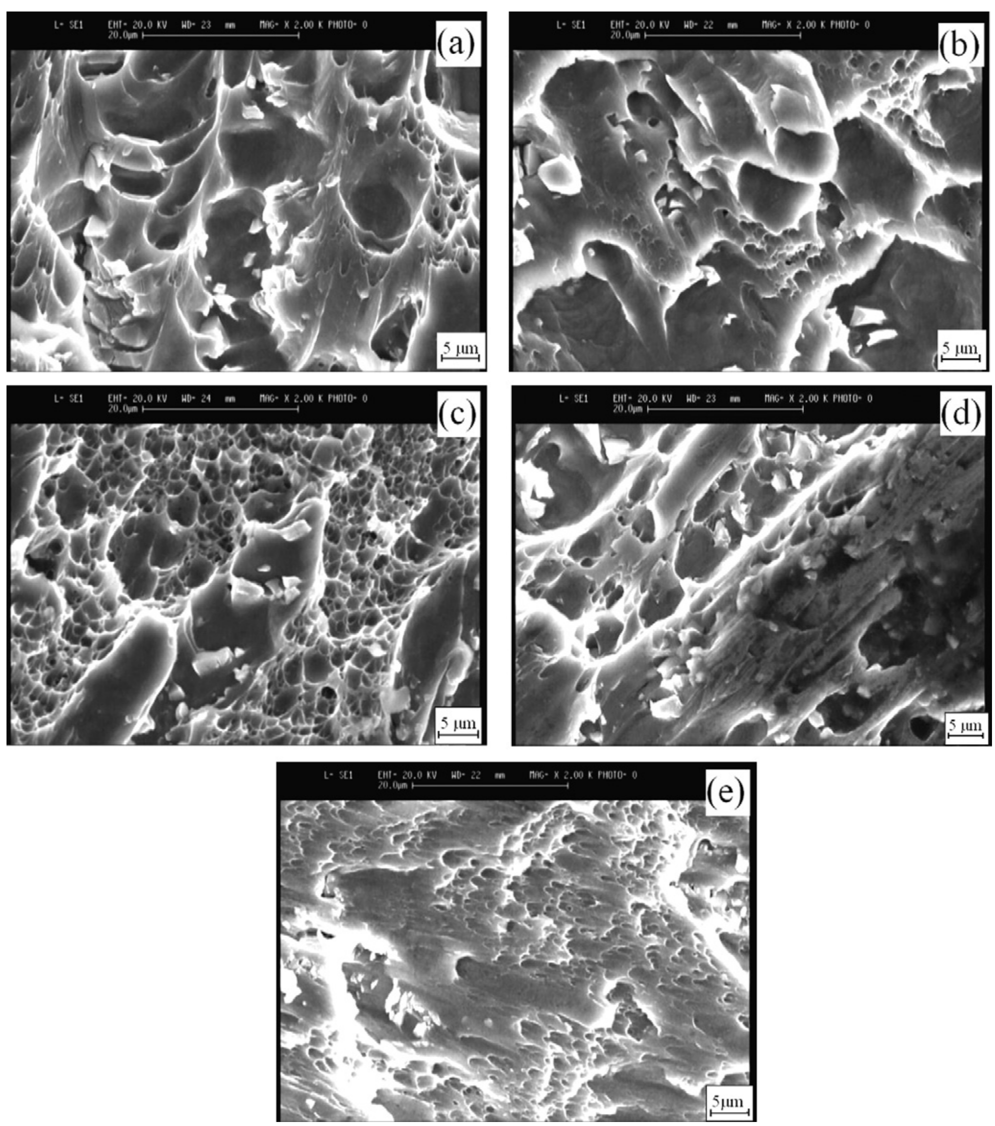

Fig. 11. Fracture surfaces of Al 2014 alloy after natural aging for (a) 15 min, (b) 30 min, (c) 45 min, (d) 60 min, and (e) is the fracture surface of ECAPed alloy immediately after solution treatment.

ing. The same experiments were performed for the Al 7075 alloy and similar results were obtained. The serrated yielding of this alloy can be seen by the first few minutes of aging time. Also, the critical time obtained from the ECAP processing was about zero for this alloy. So there is a correlation between the tensile behavior of the material and successful room temperature ECAP processing.

The occurrence of serrated yielding is due to the interaction of diffusing solute atoms with moving dislocations. A high concentration of solute atoms leads to stronger pinning of dislocations, so a higher degree of serrated yielding will occur. Over time, the formation of precipitates will decrease the concentration of solute atoms. After t^*, the concentration of solute atoms is lower than a specific value, so the serrated yielding does not occur. Also the structure of the material will have a transition from supersaturated state (sss) to sss + GP zones, and the ductility and formability of the material decreases, as shown in Fig. 10. After t^*, room temperature ECAP processing of age-hardenable alloys tends towards cracking, which is due to the loss of sufficient deformability.

Differences between alloying systems lead to different serrated yielding appearance periods. For the alloy with a higher concentration of alloying elements, the steps vanish at the earlier stages of natural aging. Consequently, the Al 7075 alloy had a much lower period of t^* than the Al 2014 alloy. This is related to the faster kinetics of precipitation in the alloy with a high amount of alloying elements. Therefore, the alloy with the higher amount of alloying elements will have a lower critical time.

The tensile curve of the ECAPed Al 2014 in supersaturated state has no serrated yielding. Therefore there is no "t^*", thus the second ECAP pass could not be performed. DSA results in the formation and growth of significantly more precipitates. So the concentration of solute atoms diminishes from the specific value and serrated yielding does not occur.

3.3. Microstructural characterization

Fig. 11 shows the micrographs of fracture surfaces where (a), (b), (c) and (d) are the solution treated Al 2014 alloy with different natural aging times, and (e) is the ECAPed alloy immediately after quenching. In Fig. 11a the sample was aged for 15 min before tensile testing. Large dimples are observed in the fracture surface of this sample which is related to ductile fracture. The fracture surface of the 30 min natural aged sample shows a more brittle fracture (Fig. 11b) in contrast to 15 min aging. The fracture surface of the sample aged for 45 min shows many homogenous fine dimples. In this state, the centers of formation of the dimples are mostly rough particles of intermetallic phases which are created by the cutting mechanism. This result demonstrates that after 45 min of natural aging, the transition from supersaturated state (sss) to sss + GP zones occurs and the precipitates are strong enough to suppress the dislocation motion. After 60 min of aging (Fig. 11d), the fracture surface clearly changed to the brittle mode. The smooth surface of the ECAPed material demonstrates the brittle fracture as seen in Fig. 11e.

Fig. 12 shows the micrograph of the ECAP fracture surface of the Al 2014 alloy, processed after 3 h of natural aging. Fig. 12b is the higher magnification of the circled area shown in Fig. 12a. The P2 pointed particle in Fig. 12b seems to be a precipitate in the matrix, pointed by P1. It can be observed clearly from the image that the crack initiation was due to the precipitates.

When the specimen is ECAPed after the critical time, DSA brings about larger particles. Therefore, dislocation motion is suppressed and material deformability decreases, which leads to crack initiation and failure of the material during ECAP processing.

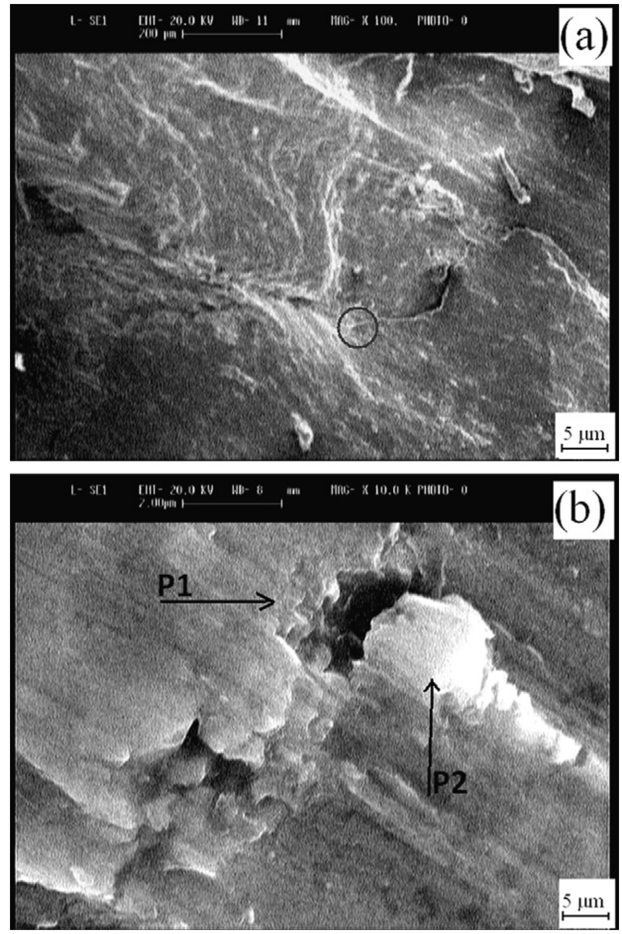

Fig. 12. (a) Fracture surface of ECAP fractured Al 2014 specimen, (b) higher magnification of the circled area.

4. Summary and conclusions

1. Experiments were conducted on two supersaturated aluminum alloys in order to develop a process to find the critical ECAP pre-aging time. The results show that ECAP can be performed successfully before the time at which serrated yielding disappears and after that, cracking and segmentation begins to occur.
2. DSA and precipitates are the main factors leading to crack initiation, incorporated with shear bands.
3. A higher concentration of alloying elements leads to lower critical time.
4. The results reveal that room temperature ECAP processing of age-hardenable alloys in the supersaturated state leads to a higher strength and optimum ductility compared with processing in the furnace cooled state.

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