Original Article

Effects of hot forming cold die quenching and inter-pass solution treatment on the evolution of microstructure and mechanical properties of AA2024 aluminum alloy after equal channel angular pressing

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ABSTRACT

The aim of this research is to investigate the effect of hot forming cold die quenching (HFQ) equal channel angular pressing (ECAP) on the microstructural evolution and mechanical properties of AA2024 aluminum alloy. For this purpose, solution treated AA2024 aluminum alloy was initially ECAPed in HFQ and water quenched (WQ). Microstructural evolutions, dislocation density, and mechanical properties were investigated after up to 3 (WQ-ECAP) and 5 (HFQ-ECAP) passes of deformation. Intermediate solution treatment was conducted after each ECAP pass. The samples underwent aging treatment after deformation. Large amount of shear banding was observed in samples after the first ECAP pass which was reduced in the successive passes of ECAP through intermediate solution treatment. Tensile properties and microhardness enhanced by increasing number of ECAP passes. However, after the fifth pass of HFQ-ECAP, sudden decrease in mechanical properties was observed. Partial decomposition of supersaturated solid solution was found to occur at higher strains. Mechanical properties and deformability were improved in the HFQ-ECAP samples with respect to WQ-ECAP.

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1. Introduction

AA2024 is a heat treatable aluminum alloy of 2xxx series exhibiting high strength, reasonable ductility and acceptable corrosion resistance [1,2]. Due to its high strength to weight ratio, this alloy is heavily being utilized in automotive and aerospace industries [1,2]. Wide range of applications have created motivation in research to improve the strength and ductility of this alloy. For example, several severe plastic deformation (SPD) processes and different ageing procedures have been applied on AA2024 alloy in recent years [3–13].

Due to its high strength and presence of low melting point (LMP) phases which limit the hot deformation temperature of the alloy, there are many difficulties and complexities in pro-
duction of AA2024 aluminum alloys. In addition, the rate of age hardening of the alloy in room temperature is very high which extremely limits the room temperature deformation of the alloy in solution treated state. It is well-known that precipitates of age-hardened aluminum alloys cause deformation deficiency under high strain [14]. This problem has been addressed by dynamic strain aging (DSA) and shear banding [7,14,15]. DSA combined with shear banding can produce coarse precipitates that tremendously reduce ductility and workability of the material [15]. In recent years, several new methods have been developed for the deformation of heat treatable alloys to acquire maximum possible benefits [16,17]. For example hot forming and cold die quenching (HFQ) was introduced in 2008 [17] to profit from hot deformation along with high cooling rate [18,19].

Equal channel angular pressing (ECAP) considered to be the most utilized severe plastic deformation (SPD) technique that results in remarkable mechanical properties. ECAP provides fairly complete homogenity in the final product [20] exhibiting uniformly-distributed fine grained structure [21]. Various ECAP studies have been reported for AA2024 alloy in different initial states including fully annealed [22], aged, over aged [11], and solutionized conditions [7,8,10]. Among ECAPed AA2024 alloys processed with different initial conditions, solutionized samples have shown remarkable properties [7,15,22]. For example, Kim et al. [10] and Kotan et al. [8] respectively investigated the influence of warm and room temperature ECAP of solution treated AA2024 alloy. It was found that sound ECAP processing of AA2024 alloy was only possible for one pass while the second pass resulted in surface damage [7,10]. Indeed, to the best of authors knowledge, ECAP processing of solutionized AA2024 alloy beyond one pass has not been successful. Similar difficulties have been reported in room temperature ECAP processing of several other age-hardenable aluminum alloys [7,10,14,15].

There are two general methods to successfully ECAP the age-hardened alloys in supersaturated solid solution. Materials may deform successfully by ECAP at higher temperatures. Disadvantages of this approach include the formation of extra-large precipitates, recrystallization and grain growth leading to the reduction in mechanical properties [14,15]. Alternatively, ECAP can be conducted at room temperature followed by the quenching of solution treated samples, where the GP zones and other strengthening precipitates are not widely formed. In recent years, room temperature ECAP of age-hardenable alloys has been studied in detail due to minimized grain growth, limited recovery, and the presence of higher dislocation density [7,23,24]. For example, several studies have reported significant improvement in the strength of pre-ECAP solution treated AA2024 alloy followed by age treatment at 100 °C instead of conventional aging at higher temperature ranges of 170–190 °C [7,10]. Aging treatment at 100 °C has been described as an appropriate temperature for ECAPed age hardenable aluminum alloys because of its improved controllability with good impression on both ductility and strength [7,10,25–27]. The dislocation density enhancement during ECAP process provides more precipitation sites to produce smaller precipitates.

So far, many studies have been conducted on the HFQ processing of heat treatable aluminum alloys and several other alloys [3,5,18,19,28–31]. It is found that by adopting an appropriate design strategy of the HFQ process, the number of components and their sizes can be reduced, thereby lowering the cost and weight. Moreover, further research is being carried out to examine the applicability of this method to form pieces with complex configuration and significant strength [19]. Attempts are indeed made to optimize the existing HFQ techniques to be incorporated with several deformation methods [16,32,33]. However, the HFQ processing has been mostly applied to sheet materials while limited number of studies are reported for bulk materials [5,19]. It should be noted that these authors investigated the effect and feasibility of combining HFQ process and ECAP for one pass. In this study, HFQ-ECAP process used as a combined method to examine simultaneous benefits of HFQ process and room temperature ECAP on the microstructural development and mechanical property improvement of AA2024 alloy at higher passes. Additionally, deformation was performed along with an intermediate solution treatment before each ECAP pass, to increase workability of the alloy to withstand further deformation. The purpose of this study was to determine the effect of HFQ-ECAP approach on the evolution of microstructure, particle distribution, and tensile properties. For comparison, few samples are quenched in water after solution treatment prior to ECAP to discuss the differences.

### 2. Experimental procedure

Hot extruded rods of commercial AA2024 aluminum alloy with 32 mm in diameter were received alongside the chemical composition disclosed in Table 1. Cylindrical specimens with pre assumed 15.9 mm diameter and average length of 100 mm were machined from the rods. Meanwhile for HFQ specimens, applied diameter was 15.7 mm that increased to 15.9 mm after heat treatment. An ECAP die made of hot worked tool steel (H13) with two 16 mm wide channels intersected at 90° and an outer curved corner angle of 22° was utilized. ECAP was conducted at a ram speed of 1 mm/sec. Since sticking occurred and resulted in ram bending and surface failure of the deforming samples during HFQ-ECAP, high-temperature resistant colloidal graphite-based grease was applied as lubricant.

In order to ensure that no effects of previous extrusion deformation disturb the evolution of microstructure during ECAP, a high temperature solution treatment at 495 ± 2 °C was chosen. This temperature was the maximum allowed temperature without dissolving the low melting point (LMP) phases during solution treatment. Initial investigations demonstrated that the samples are not fully recrystallized at solution times of less than 24 h. Therefore, all samples were solution treated at 495 ± 2 °C for 24 h, prior to ECAP. Room temperature ECAP and hot forming cold die quenching (HFQ) ECAP was performed on the samples. Therefore, the samples were

<table>
<thead>
<tr>
<th>Element</th>
<th>Al</th>
<th>Cu</th>
<th>Mg</th>
<th>Mn</th>
<th>Fe</th>
<th>Si</th>
<th>V</th>
</tr>
</thead>
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<tr>
<td>Wt.%</td>
<td></td>
<td>1.24</td>
<td>0.43</td>
<td>0.19</td>
<td>0.11</td>
<td>0.02</td>
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quenched in water prior to ECAP (WQ-ECAP specimens) or inserted into the die orifice to perform HFQ-ECAP. In HFQ, ECAP begins instantly followed by the insertion of workpiece into the entrance channel of the die. As it was observed, neither of the WQ-ECAP nor HFQ-ECAP samples can resist the second pass of deformation, the samples underwent intermediate annealing at 495 ± 2 °C for 30 min (or intermediate solution treatment) between successive ECAP passes. Deformation was repeated until the samples were fractured at 4 and 6 passes for WQ and HFQ trials, respectively. Route Bc, i.e., 90 degrees rotation between each pass of deformation, was used in this research due to its better effects on producing finer grains with homogenous microstructure [34]. Aging treatment was conducted at 100 °C for 25 h. Some samples were kept un-aged for comparison purposes. All samples whether aged or none aged were held at −18 °C before mechanical testing and microstructural observations.

Samples were extracted for microstructural analysis along the longitudinal axis parallel to ECAP direction from central regions. After cutting, the samples were ground and mechanically polished using emery papers and 0.3 μm MgO particles, respectively. Eventually Barker’s reagents (4 mL HBF₄ 48 % and 200 mL distilled water) were prepared for electro-etching of samples operated at 20 V DC for 50 s. Microphotographs were captured by means of HVU17Z-HR3-TRF-P optical microscope operated using polarized light.

By utilizing the plain surface of the samples that was ground and polished, X-ray diffraction analysis was performed on a DRON-8 diffractometer using Cu Kα radiation at 40 kV and 30 mA with a scan rate of 0.05 °/s. The well-known Williamson-Hall approach plus Rietveld technique were utilized to calculate the average dislocation density of specimens. First of all, average domain size (Dᵥ) and microstrains (ε) were estimated through Williamson–Hall relationship [35] based on the slope and ordinate intersection of the line plotted using following Eq. (1) [36]:

$$\beta \cos \theta = \frac{\lambda}{D_v} + 4\epsilon \sin \theta$$  \hspace{1cm} (1)

Where $\beta$, $\theta$, and $\lambda$ are the full-width at half maximum (FWHM), the Bragg’s angle of the peak, and the wavelength of X-ray, respectively. At the end, dislocation density was calculated by Rietveld technique using equations below [36]:

$$\rho_d = \frac{3}{D_v}$$ \hspace{1cm} (2)

$$\rho_b = \frac{\eta^2}{b^2}$$ \hspace{1cm} (3)

$$\rho = (\rho_d \cdot \rho_b)^{1/2}$$  \hspace{1cm} (4)

Where $\rho_d$ and $\rho_b$ are dislocation density due to domains and dislocation density due to the microstructure, respectively while $b$ is the Burger’s vector and $\rho$ is the dislocations density. It should be noted that, for the initial solution treated sample with equiaxed grains, measurement of the dislocation density by the Williamson-Hall method is not possible, thus the dislocation density is considered to be $10^6$ cm⁻² for this sample.

Tensile and microhardness testing were conducted to measure the effect of processing parameters on the hardness, strength, and ductility of the samples. Tensile tests were conducted on sub-sized samples with dimensions according to ASTM-E8 standard. According to the standard, the characterizing sizes such as gage length, width, and thickness were 12.5, 3 and 2 mm, respectively. The samples were extracted along the length of the ECAPed specimens and from the central part of the deformed bar. Santam universal testing machine was used at a crosshead speed of 0.5 mm/min at room temperature. The Microhardness tests were performed using a SHAAB-M5 microhardness machine under 100 g load for 15 s on the longitudinal direction of the deformed sample. Microhardness results were reported as average measurements of at least 5 points.

3. Results and discussion

3.1. Initial microstructure

The AA2024 samples to be studied in this investigation were extracted from hot extruded rods. In order to ensure that the previous deformation did not affect the evolution of microstructure, the samples were recrystallized. However, recrystallization needs temperatures as high as 550 °C which is above the softening temperature (505 °C [37]) of low melting point (LMP) phases in this alloy that should be avoided. Therefore, the maximum allowed temperature was chosen 10 °C below 505 °C, i.e., 495 °C. Grain structure of the sample after the solution treatment for 30 min at 495 ± 2 °C is shown in Fig. 1(a). It can be seen that the grain structure is elongated and not recrystallized. Increasing the annealing time to 1, 2, 6, 12 and 18 h resulted in partially recrystallized microstructures among the elongated grains. For example, Fig. 1(b) shows the initial microstructure after 6 h of annealing time. However, by increasing the solution treatment time to 24 h, recrystallized and equiaxed microstructure is achieved as displayed in Fig. 1(c). Therefore, all samples were solution treated for 24 h to ensure that the effects of prior deformation are annihilated and would not significantly affect the evolution of microstructure during ECAP.

Application of solution treatment prior to precipitation hardening is a routine production process for 2xxx series aluminum alloys. Coarse pre-existing precipitates may dissolve during solution treatment. However, this has negligible effect on intermetallic particles. Distribution of second phase particles after solution treatment and water quenching is shown in Fig. 1(d). As the dissolution time was long enough, i.e., 24 h, the soluble particles are likely to be dissolved and the observed particles may be considered as insoluble. Two types of particles are observed in this image. One includes coarse gray particles in the size range of 1–10 μm. Furthermore, smaller spherical particles are also observed in black. The primary particles are aligned along the initial extrusion direction, during the production steps of the alloy. It is clear that this alignment remains unchanged after the solution treatment.
3.2. Microstructural features during ECAP

3.2.1. Evolution of grain structure

Evolution of grain structures during HFQ- and WQ-ECAP are exhibited in Figs. 2 and 3, respectively. Large numbers of shear bands are observed in the microstructures of the samples after the first ECAP pass, as observed for HFQ-ECAP and WQ-ECAP in Figs. 2(a) and 3(a), respectively. These images are captured from the central regions of the ECAP samples where the density of shear banding is higher. This is due to the greater effect of deformation and higher strain imposed onto the sample in the central regions [8,38,39]. Consequently, significant grain refinement is expected at these regions. These shear bands are extended into few adjacent and dynamically recrystallized (DRX) grains as observed in the HFQ sample. However, such DRX grains are not observed in the WQ-ECAP sample where the shear banding is more severe and at higher density. Shear banding is an indication of localized deformation and can result in the fracture with further deformation. Since shear banding is more severe in WQ-ECAP sample, the probability and severity of fracture is larger, as was observed by the fracture of WQ samples at lower passes.

After the intermediate solution treatment for 30 min at 495 ± 2 °C, recrystallization may not occur considering the fact that it was not recrystallized at the same conditions in the solution treatment, as observed in Fig. 1(a). However, one may note that this image was captured after annealing of a hot extruded sample. While the temperature is low and the amount of deformation is high during ECAP, recrystallization is likely to occur and being observed. This leads to finer grain structure which can afford more deformation with less shear banding. Figs. 2(b) and 3(b) show the microstructure of HFQ-ECAPed and WQ-ECAPed specimens after second pass of ECAP, i.e., HFQ2 and WQ2 samples. The fact that the elongated grains are significantly finer than those in the one pass deformed samples, denotes the recrystallization of the samples during intermediate annealing treatment. Shear bands are observed in both samples, however, the density has significantly reduced. This can be attributed to more capacity of the sample with fine-grain structure for uniform deformation and fewer shear banding as a sign of localized deformation.

Further deformation with the application of intermediate annealing resulted in additional grain refinement. This can be observed in Figs. 2(c) and 3(c) which show the microstructures of the HFQ3-ECAP and WQ3-ECAP samples. According to the micrographs, the samples contain finer grain structure with reduced number density of shear banding. After applying the intermediate solution treatment of the fourth and the fifth ECAP passes under the HFQ-ECAP process, the grain refinement continued, as being observed in Figs. 2(d) and (e). Less shear banding is observed in these samples. As a conclusion, it should be noted that by considering the Figs. 2 and 3, it’s clear that the presence of shear bands are decreased after using consecutive ECAP passes. This phenomenon can be clarified by the several main conditions which promote shear banding. Indeed, possibility of shear banding increases by enlargement.
of grain size prior to deformation, presence of high amount of solute atoms, applying high extent of strain and deformation at low temperatures [40]. Presence of intermediate solution treatment after first ECAP pass eliminates the present shear bands and grain refinement through static recrystallization. Thus reducing of grain size and high quantity of grain boundaries, limit the promotion of shear banding. This trend continues after each intermediate solutionizing and successive passes. There are various studies which have reported this effect [40,41]. Moreover, amount of solute atoms i.e. alloying elements after intermediate solutionizing is lower compared to the initial solutionizing treatment due to the shorter time. According to other reports [14, 15], presence of inferior amount of solute atoms results in less DSA content and shearable precipitates in heat treatable aluminum alloys. Therefore, little strain localization and shear banding occurs [7].

It is worth noting that the recrystallized morphology of workpieces after intermediate solution treatment are represented in Fig. 4. Figs. 4(a) and (b) demonstrate microstructure of samples after intermediate solution heat treatment between passes number of 1, 2 and 2, 3, respectively. These microstructures are common in HFQ-ECAP and WQ-ECAP samples that deformed successfully until third pass. Further, intermediate solution treated morphology of HFQ-ECAPed samples between passes number of 3, 4 and 4, 5 are illustrated in Fig. 4(c) and (d), respectively. On the other hand, occurrence of intermediate annealing and partial recrystallization of microstructure can reduce the mechanical and microstructural benefits of SPD to the some extent. However utilizing intermediate solutionizing in this study is essential. First of all, intermediate solutionizing dissolves all GP zones and strengthening precipitates that are generated dur-
Fig. 3 – Evolution of grain structure of AA2024 alloy during WQ-ECAP (a) 1, (b) 2 and (c) 3 passes.

ing and following ECAP in time intervals between successive ECAP passes. These precipitates cause the workpiece of the next ECAP pass to be damaged if the intermediate solutionizing is not used, as explained in other studies [7,10,14,15]. Secondly, intermediate solutionizing is needed to dissolve alloying elements and excessive precipitates to generate a supersaturated solid solution that is necessary to gain the advantages of aging treatment after each ECAP pass. Thirdly, the heat treatment between passes is needed to increase the workability and continuing ECAP up to the highest possible quantity. Fourthly, combination of intermediate annealing and ECAP resulted in finer and more homogenized grain structure in successive passes which was unattainable through one pass ECAP. Moreover several other benefits of intermediate annealing is reported in literature. For instance Wei et al. [42], compared the deformation of workpieces with and without intermediate annealing and elucidated improved homogeneous deformation of samples with intermediate annealing.
In addition, Suzuki et al. [43] indicated the effect of multiple deformation stages and intermediate heat treatment of an aluminum alloy on further homogenous distribution of secondary particles that in turn results in smaller recrystallized grains. In another study Chang et al. [44], concluded that intermediate annealing between consecutive ECAP passes of pure aluminum leads to proper ductility without sacrificing tensile strength due to the increased amount of high angle boundaries.

3.2.2. Evolution of second phase particles
Distribution of second phase particles after HFQ-ECAP and WQ-ECAP are shown in Figs. 5 and 6, respectively. It can be seen in Figs. 5(a) and 6(a) that after first pass of ECAP, initial coarse gray intermetallic particles are fragmented and redistributed. It can be seen that the number density of small particles has increased with respect to the initially solution treated samples that may be attributed to particle fragmentation and re-distribution. The particles seem to be slightly round, which may have been resulted from fragmentation, compared to the initial particle morphology. Fragmentation and re-distribution of particles may be due to shear deformation during ECAP [11]. Furthermore, finer black particles are found at higher density and in reduced size. Figs. 5(b) and 6(b) show the respective microstructures of HFQ2 and WQ2 samples which were ECAPed for the second pass after intermediate solution treatment. The coarse gray intermetallic particles are more dispersed. In addition, the alignment of these particles along the initial extrusion direction is destroyed. The average size of the intermetallic particles has decreased and number density has increased. Indeed, the large gray particles are fragmented. Moreover, fine black particles are better distributed in the sample. After intermediate solution treatment, third deformation pass was conducted and the microstructures of HFQ3 and WQ3 are presented in Figs. 5(c) and 6(c), respectively. The intermetallic gray particles are more dispersed while larger particles are still present. Indeed, particles are becoming smaller and more spherical with increasing pass number. Smaller gray and finer black particles are also found to be more dispersed all over the samples.

As previously mentioned, the WQ-ECAP samples failed after third pass of deformation. However, the HFQ-ECAP samples withstand the failure upto sixth pass of deformation. Fig. 5(d) refers to the microstructure of the HFQ4 sample. In this sample, particles continue to refine where the initial coarse gray particles gradually transform into small spheres with more uniform distribution throughout the sample. After the intermediate solution treatment and fifth HFQ-ECAP pass, as demonstrated in Figs. 5(e) and 5(f), a large accumulation of intermetallic particles and coarse deposited particles are observed. These compounds have been distributed throughout the sample with a great volume fraction. Formation of these particles can be attributed to the decomposition of the supersaturated solid solution due to various deformation and heating cycles. The reduction in mechanical properties due to the decomposition of the supersaturated solid solution in aluminum alloys has been reported in other studies [45–47].
The microstructure of the HFQ5-aged sample is presented in Figs. 7(a) and 7(b). It is clear that after aging, the number density of coarse particles is reduced and the distribution is improved. Large percentage of intermetallic particles are deposited at the grain boundaries and in some cases within the grains. As reported in several studies about aluminum alloys, it seems that this phenomenon is related to decomposition of the supersaturated solid solution thru increasing equivalent strain [45-47].

3.2.3. Dislocation density
X-ray diffraction patterns of the samples, with and without aging treatment, are shown in Fig. 8. The α-aluminum phase was the only detected phase. After deformation, the peak intensity was altered, which is probably due to the network defects and distortions [48]. However, after aging treatment the peak intensities partly increased because of recovery. The Williamson-Hall approach [35] and Rietveld technique [36] were used to calculate the dislocation density.
Values of dislocation density in the samples are presented in Fig. 9. Significant increase in dislocation density was observed after an ECAP pass in WQ and HFQ specimens. This may be related to the activation of the multiplication mechanisms in dislocations and their interactions with defects or with each other that leads to severe work hardening. Owing to the presence of alloying elements, soluble precipitates, and enriched super saturated solid solution, the dislocation density of the WQ-ECAP sample was expected to be superior. While, the maximum dislocation density was obtained after the first deformation pass of the HFQ-ECAP sample. The reason for this phenomenon is not clear. However, the presence of additional strain in the warmer central region of the HFQ compared to the surface parts maybe the cause of this discrepancy. It should be noted that although the sample is warmly deformed, due to the very short time of deformation, extensive recovery is not expected to reduce dislocation density.
After aging treatment, the dislocation density of HFQ1-aged sample did not change much, indicating a slight recovery of the sample during aging. Dislocation density of the HFQ2 sample was significantly reduced compared to the HFQ1. The main reason for this reduction is the intermediate solution treatment before the second pass of deformation that greatly lowered the dislocation density due to the recrystallization. It is true that the HFQ2 sample undergoes similar deformation as HFQ1 after intermediate annealing, however, the existence of finer grain structure results in more dislocation annihilation by grain boundaries leading to reduced dislocation density. Dislocation density of the sample does not change much after aging, which corresponds to slight recovery during aging. After the third pass deformation, the dislocation density increased despite the recrystallization occurrence due to the intermediate solution treatment. This trend was also observed in the HFQ4 sample, nevertheless a sharp decrease in the dislocation density was detected in the HFQ5 sample. In line with the variations in the distribution of second phase particles, this can be attributed to the decomposition of solid solution in the form of large deposits. In the case of aged samples, a similar trend is observed. Indeed, in all three HFQ3, HFQ4 and HFQ5 samples, the dislocation density has decreased after aging treatment. In addition, the reduction in dislocation density has steadily increased from HFQ3-aged to HFQ5-aged, indicating a greater recovery effect during aging by increasing the number of passes.

WQ1, which was water quenched prior to ECAP, has the highest dislocation density among the WQ samples. In as-deformed state, the dislocation density in WQ2 and WQ3 samples is slightly lower than WQ1. This may be attributed to the application of intermediate annealing which resulted in grain refinement. In finer grain state, the grain boundaries may play as sinks to dislocations lowering the dislocation density in the deformed state. Examining the dislocation density of the WQ-ECAP aged samples also indicates a decrease in the dislocation density of samples after the aging process, which is more pronounced by increasing the number of passes. In addition, the sinking role of grain boundaries remains during aging whereby the fine grained samples, i.e., WQ2 and WQ3, show significant reduction in dislocation density during aging treatment.

3.3. Mechanical properties

3.3.1. Hardness

Average Vickers microhardness of HFQ-ECAP and WQ-ECAP samples in aged and non-aged conditions are shown in Fig. 10.
In addition, the microhardness of the samples without deformation are also presented. For this purpose, the values of hardness are presented after quenching and after aging treatment at 100 and 190 °C for 25 and 8 h, respectively. Average Vickers hardness of the samples after solution treatment and quenching was 128.53 HV, which increased to 137.57 and 145.81 HV after aging at 100 °C and 190 °C, respectively. Among the deformed samples, it can be seen that the hardness of the WQ-ECAP samples is relatively higher than the HFQ specimens. This may be attributed to the lower quenching rate applied to the HFQ samples in comparison with the WQ samples. This results in a less matrix enrichment and solution hardening during quenching with fewer efficient precipitation hardening during aging treatment.

Hardness of deformed samples increased compared to the undeformed ones. Increasing the hardness after ECAP is due to the dislocations density rise after deformation and also dislocations interaction with solute atoms or each other during indentation. By performing the intermediate solution treatment and the second ECAP pass, hardness was increased. Maximum hardness of all samples was observed in WQ1 and WQ2 due to the presence of more enriched super saturated solid solution in water quenched samples and the greater effect of aging treatment. Highest microhardness of the HFQ-ECAP samples is observed in HFQ1. With the application of intermediate solutionizing and further ECAP, hardness can be considered unchanged or slightly reduced in HFQ2, HFQ3 and HFQ4. However, the sharp reduction in hardness of HFQ5, as described in microstructural observations, can be ascribed to the decomposition of the supersaturated solid solution at high deformation and heating cycles [45–47].

Hardness is found to increase with aging treatment after deformation in both WQ and HFQ samples. This is due to the formation of fine and coherent precipitates in the sample that interrupt the normal movement of dislocations [13]. Consequently, dislocations have to cut these precipitates or circumvent them resulting in an increased hardness value [13]. Increase in hardness in HFQ samples by applying aging treatment indicates the efficiency of the HFQ process to replace the WQ prior to aging. The hardness of the HFQ1-aged sample is slightly higher than HFQ2-aged and HFQ3-aged samples. As the reduction in hardness in HFQ5-ECAP sample was attributed to the decomposition of alloying elements from solid solution, the increase in hardness after aging may be due to the dissolution of some of the decomposed particles and residual deposits formed during the decomposition of the super saturated solid solution. In addition aging results in the formation of strengthening precipitates based on the part of super saturated solid solution that was not decomposed.

### 3.3.2. Tensile properties

Engineering stress-strain curves of the un-deformed samples in as-quenched and aged conditions are exhibited in Fig. 11. After aging, the flow stress, yield stress (YS), and ultimate tensile strength (UTS) increased with respect to the solution treated and quenched sample. Uniform elongation (eu) and
In fact, during high temperature aging, precipitates form at lower density and higher growth rate leading to non-uniformly distributed coarser precipitates. Such distribution of precipitates results in localized deformation and reduced ductility. However, when aging is conducted at lower temperature, finer precipitates form with improved distribution. Precipitates can play a strain hardening role to postpone necking and result in enhanced ductility provided that they are well distributed.

Since highly uniform and fine precipitates are expected to form during aging treatment at 100 °C, higher strength may be expected compared to the sample aged at 190 °C. However, it can be seen that the enhancement in strength is not pronounced at lower aging temperature. Therefore, it may be concluded that aging did not continue to the maximum hardness and strength at 100 °C. These heat treatment conditions are selected based on previous investigations [7,10,25] due to the fact that this treatment is going to be applied on severely deformed samples, as well. When the deformed samples are considered, increasing the aging temperature or time may cause few problems and should be avoided. Indeed, the diffusion rate increases, by increasing dislocation density due to deformation, would result in increased precipitates size with reduced strength. In addition, extensive recovery or slight recrystallization may occur, which can greatly affect the achievement of desired mechanical properties [25]. Therefore, aging at 100 °C for 25 h was preferred for strengthening of the deformed samples.

Effects of ECAP deformation on tensile properties of HFQ-ECAP and WQ-ECAP samples are shown in Fig. 12. As displayed in this figure, after one pass deformation, YS and UTS are increased, which is consistent with the results obtained by other researchers in ECAP of solution treated AA2024 alloy [7–10]. Comparison of tensile curves before and after aging shows that the application of aging treatment leads to increase in flow stress, YS, and UTS as well as great improvement in elongation. This behavior may be attributed to the dislocation annihilation occurred by the recovery during the aging process. Indeed, improvement in ductility can be credited to the reduction of dislocation density during aging treatment,
which creates more space for further deformation and postponed necking and fracture. As the strength increases with aging while decreases due to the recovery \cite{7}, the net effect of these parameters determines the trend of strength variation during tensile testing.

It can be seen in Figs. 12(a) and (b), that similar to the observations of hardness, HFQ5-ECAP sample shows significant reduction in YS, UTS, and elongation when compared with all other specimens. This is probably due to the decomposition of the supersaturated solid solution, especially into the grain boundary regions in addition to reduced dislocation density. As the intermediate annealing is performed at high temperature, such decomposed particles may be of extremely coarse size which do not contribute in strengthening but may cause softening due to matrix depletion. After aging (HFQ5-aged sample), the UTS is relatively and the elongation is largely increased compared to the none-aged HFQ5 sample. This could be due to the dissolving of the decomposed particles, especially deposited particles on the grain boundaries and possibly owing to the precipitation of the strengthening precipitates during aging treatment.

Comparison between the tensile test curves of HFQ and WQ samples shows that the YS of the HFQ-ECAP samples is lower than the WQ-ECAP samples. This is in line with lower observed hardness values in HFQ in contrast to the WQ samples. However, the UTS and elongation of the HFQ-ECAP samples are higher than WQ-ECAP ones. This reflects the high efficiency of the HFQ-ECAP process to improve the mechanical properties of the AA2024 aluminum alloy. It is clear that the WQ samples with more efficient cooling, i.e., water quenching, would possess higher fraction of alloying elements in solution and consequently more efficient solution treatment than the HFQ sample that are cooled in the die. Accordingly, higher strength and aging response is expected in WQ samples. Therefore, in order to interpret such improvement in UTS and ductility, it should be noted that these two parameters are directly governed by the work hardening capacity of the alloy. Indeed, if the alloy is extensively work-hardened, it will show a postponed necking and consequently increased elongation and UTS. During HFQ process, fewer dislocations are generated since the sample is deformed at relatively higher temperature, certainly higher than that of the WQ samples. Therefore, lower dislocation density is generated in HFQ samples that leaves more room for work hardening and consequently postpones necking.

Fig. 12 – Engineering stress-strain curves of ECAPed samples in (a) HFQ-ECAP process, (b) HFQ-ECAP process with aging treatment. (c) WQ-ECAP process and (d) WQ-ECAP process with aging treatment.
In addition, one may notice that in the HFQ samples, the YS is not significantly affected by aging treatment while in the WQ samples, the YS reduces during aging. This is in line with the fact that the extent of reduction in dislocation density is less inclusive in HFQ samples than the WQ samples. Therefore, YS, as a significantly affected characteristic by dislocation density, is slightly altered.

Another interesting point which is observed in both the tensile and hardness results is the slight variation in properties with increasing the number of ECAP passes. Such behavior of slight variations with increasing number of passes is a property of AA2024 alloy, as has been broadly observed by other researchers. Indeed, various studies [11,22] have shown that the effect of grain size refinement should be examined along with the presence of precipitates and other factors such as dislocation density [8]. For example, Horita et al. [22] studied the effect of several ECAP passes on mechanical properties of annealed AA2024 samples and showed no significant change in YS with the slight change in UTS and εf in different strains up to equivalent strain of four. Furthermore, Mao et al. [13] studied the effect of several ECAP passes on mechanical properties of aged and over aged AA2024 specimens and exhibited the very negligible change in mechanical properties up to equivalent strain of 4 followed by no change or even slight decrease in some cases up to equivalent strain of 8. On the other hand, due to the intermediate solution treatment between the deformation passes, partial recrystallization and extensive recovery may be expected. This is more likely to occur in the samples with significant deformation. Consequently, no substantial increase in hardness and tensile strength is expected with increasing the number of passes.

4. Conclusions

In this research, effects of equal channel angular pressing (ECAP) on the evolution of microstructure, particle distribution, hardness, and tensile properties of AA2024 aluminum alloys was investigated. Prior to ECAP, the samples underwent two different quenching cycles, i.e., water quenching (WQ) and hot forming cold die quenching (HFQ). According to the results of this investigation, the following conclusions were made:

1. Whether in WQ or HFQ state, severe shear banding in the first pass and through thickness cracking resulted in fracture at the second pass. Utilization of intermediate solution treatment between ECAP passes resulted in successul deformation of up to 3 and 5 passes of ECAP in WQ and HFQ modes, respectively.
2. ECAP led to the refinement and improved distribution of particles in the sample. In addition, grain refinement occurred and consequently, less shear banding was observed in the successive passes of ECAP when compared with the first pass.
3. Hardness and tensile strength (YS and UTS) increased significantly after the first pass of ECAP and remained almost unchanged in the successive passes. This was attributed to the applications of intermediate solution treatments which may have resulted in recrystallization prior to successive ECAP passes.
4. Aging treatment resulted in enhancement in hardness, strength, and ductility. Improvement of ductility was attributed to occurrence of partial recovery leading to the reduction in dislocation density during aging.
5. HFQ-ECAP process requires lower pressing load when compared to WQ-ECAP but yields similar mechanical properties.

Conflict of interest

There is no conflict of interest.

REFERENCES

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