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Mechanical properties and pitting corrosion behavior of Al5085 alloy processed via equal channel angular pressing (ECAP)

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ABSTRACT

In this paper, the impact of the number of passes of equal channel angular pressing (ECAP) on mechanical properties and corrosion behavior of Al5085 alloys in an HNO3 solution is investigated. After a single pass of ECAP, the ultimate tensile strength (UTS) was increased noticeably from 275 MPa to 342 MPa. Also, the yield strength (YS) and microhardness were improved from 82 MPa to 118.5 MPa and 94 HV to 136 HV, respectively. After the second pass all UTS, YS, and microhardness increased by 13.5%, 17.8%, and 11.7% compared to the first pass. Meanwhile, the elongation to failure was reduced by 15% and 7.5% after one and two passes of the ECAP, respectively. According to scanning electron microscope (SEM) micrographs, unlike the unprocessed sample in which there is no evidence of pitting corrosion occurrence, after one pass ECAP, the pitting corrosion occurred through all the surfaces of the sample. Also, there is no evidence of pitting corrosion after two passes of ECAP.

Keywords: Equal channel angular pressing (ECAP), Corrosion, Pitting corrosion, mechanical properties, severe plastic deformation (SPD).

1. Introduction

Aluminum-magnesium alloys (5xxx series) in which Mg is the main alloying element exhibit the highest strength among non-heat treatable Al alloys [1-3]. Al-Mg alloys could be used as a replacement to steel in the automotive industry and vessels due to their high strength to cost, high strength to weight, oxidation resistance, and good formability [4]. This series is known as marine alloys which are used in ships and radar boats because of theirs high corrosion resistance in seawater [5, 6]. Since ultra-fine grained (UFG) Al 5xxx shows a good combination of mechanical and corrosion properties, many efforts have been made to produce it. Severe plastic deformation (SPD) is one of the most operant methods to produce UFG and NG materials [7, 8]. The mechanical properties of SPD processed materials are enhanced through grain refinement, grain size reduction, and increasing in dislocation density [7, 9-14]. Equal channel angular pressing (ECAP) is one of the most favorable SPD methods that was invented in 1977

by Segal [15]. As a result of passing through the angular channel, a certain amount of plastic strain is introduced to the sample, which is influenced by die parameters. In this regard, a strain of ~1.15 is applied to the sample in each pass of the ECAP by the intersection angle of 90° [16].

Another key property of structural materials is corrosion resistance which is influenced by material type, microstructure, and corrosive media [17]. Internal stress, which is created as a result of nonuniform thermomechanical processing could be considered as another effective parameter that influences on corrosion resistance. From the perspective of thermodynamics, owing to the presence of a high fraction of grain boundaries and a higher amount of internal energy in UFG material compared to coarse-grained (CG) materials, the latter has a lower tendency to dissolve in aggressive environments [18] which were reported by many researchers [19-21]. However, many studies reported contradictory results that shows reducing the grain size leads to better corrosion resistance [22-24]. To explain and clarify these results, Ralston and Birbilis [25] categorized the effect of grain size on corrosion resistance with respect to the dynamic polarization curve into three modes. First; active mode, in which reducing the grain size reduces the corrosion resistance, second; The passive mode has the opposite trend of active mode and third; the active/passive mode in which acts like active mode at active areas and vice versa. Since SPD methods have major effects on grain size, texture, microstructure, and internal stress of processed material, numerous research studies have been done on the corrosion properties of SPDed samples [17, 18, 25]. In this regard, the stress corrosion cracking (SCC) behavior of Al-Mg alloy is investigated by several researchers, which happens along the grain boundaries owing to the presence of stress and a susceptible environment (e.g. solution containing Cl-) [26]. Asiful et al. [27]

studied the Al5083 alloy electrochemical behavior in a 3.5% sodium chloride solution after the ECAP process. The shape of the achieved potentiodynamic polarization curve shows active mode with respect to Ralston and Birbilis categorization [25] where the corrosion resistance increases.

As far as the authors are aware, there is currently no published paper that examines how ECAP affects the corrosion behavior of Al5085. Therefore, the equal channel angular pressing was conducted in two consecutive passes on aluminum 5085 alloys to investigate its impact on mechanical properties and corrosion behavior.

2. Experimental procedure

Al5085 alloy with a length of 75 mm and a diameter of 15 mm was used as a starting workpiece. The ECAP process using a die as illustrated in Fig. 1a was implemented to refine the microstructure of the sample. ECAP routes are categorized into three classes. In route A, the sample has no rotation between consequent passes. In route B, the sample rotates by 90 after each pass, and in route C the sample rotates 180 after each pass [17, 27, 28]. In this study, two passes of ECAP have been performed on the sample using route C. To investigate the mechanical properties of ECAPed samples, a tensile test was carried out by a 5 Ton SANTAM STM-50 machine at a strain rate of at room temperature. The Vickers microhardness measurements were conducted by an indenter load of 200 g for 10 s using Koopa hardness tester.

To study the corrosion response of initial and ECAPed samples, the following procedure was performed. At first, the samples were immersed in a 5% sodium hydroxide (NaOH) solution at 80 for 60 seconds. Then, the samples were immersed in concentrated HNO₃ at room temperature for 30



Fig. 1- a) The ECAP die and b) unprocessed, one pass and two-pass ECAPed samples.

seconds. A scanning electron microscope (SEM) 10 KV FEI Nova NanoSEM450 was used to investigate the surface characteristics. Furthermore, an SEM equipped with energy dispersive X-ray spectroscopy (EDS) was employed for elemental analysis. Also, JMatPro software was used to calculate stable and metastable phases.

3. Result and discussion

The unprocessed, one-pass, and two-passes ECAPed samples are depicted in Fig. 1b. Fig. 2a illustrates the engineering stress-strain curves at room temperature for the initial sample, the one-pass, and the two-pass ECAPed sample. In addition, Fig. 2b shows the change of UTS, YS, and elongation percentage after each pass of the ECAP process. As it is obvious YS and UTS increase remarkably after the ECAP process and performing more passes leads to further increase in both of them. The similar results were reported by other studies [29,30].

Based on Fig. 2a and 2b, performing one pass ECAP leads to an increase in UTS from 275 MPa to 342 MPa and YS increases from 82 MPa to 118.5 MPa. Also, the second pass ECAP process are improved UTS and YS to reach 388 MPa and 139.6 MPa, respectively. This enhancement of mechanical properties after conducting the SPD method happens due to two main reasons of work hardening and grain refinement [31]. Work hardening is considered the main strengthening mechanism in plastically deformed metals and alloys. This mechanism increases the strength due to multiplication, movement, and interaction of dislocation. Performing the SPD generates random dislocations inside the grains and consequently increases the UTS and YS noticeably. Grain refinement is another important mechanism, which affects the mechanical properties. According to the well-known Hall-Petch relationship, reducing the grain size causes an increase in strength [17] due to the high fraction of grain boundaries which acts as a strong obstacles against dislocation movements. Besides these mechanisms, in Al 5xxx, Mg alloying element as a solute atom causes more strengthening due to the solid solution strengthening mechanism. It should be noted that the rate of strengthening by SPD decreases by increasing the number of passes. This matter happens as a result of the balance of generation and annihilation of dislocations [17]. The same trend has been reported by several researchers [32,33]. As can be seen in Fig. 2a and b, after performing ECAP, due to the absorption of dislocations into the grain boundaries, the probability of the recovery process increases [34] and consequently, the strain hardening reduces, which is a usual phenomenon for SPDed samples. This low strain hardening capability of UFG materials produced by SPD methods causes earlier necking compared to coarse grain ones [35]



Fig. 2- a) Engineering stress-strain curves of the initial, one-pass, and two-pass ECAP processed samples, b) UTS, YS, and elongation of tensile samples.

Table 1- Mechanical properties of the unprocessed, one-pass, and two-passes ECAP processed sample

	Ultimate tensile strength (MPa)	0.2% proof stress (MPa)	Elongation %	Vickers hardness (HV)	Hardness error value* %
Initial	275	82	23.5	94	3
One pass	342	118.5	20	136	2
Two-pass	388	139.6	18.5	152	2

and finally, the ductility of the material reduces. According to Fig. 2a and 2b, the elongation has been reduced from 23.5% to 20% and 18.5% after one and two passes of the ECAP, respectively.

According to table 1, after one pass of the ECAP process hardness of the material increases significantly and further improvement is achieved after two passes of ECAP. This improvement could be related to work hardening, an increase in the dislocation density, grain refinement, sub-grains generation, and a high fraction of grain boundaries. As it is demonstrated in table 1, the microhardness has reached 136 HV from 94 HV after one pass ECAP. Performing the second pass increases the hardness by ~61.7% from the initial one and the hardness of 152 HV resulted. The sharp increase after the first pass could be attributed to higher strain/work hardening compared to the second pass of the ECAP process. It is observed that the hardness trend is in good agreement with the UTS and YS trend as mentioned above. Also, table 1 summarizes mechanical properties changes.

Fig. 3 indicates the SEM micrograph of the

initial and ECAPed samples after dipping in HNO_3 solution, by various magnifications. As can be seen, there is no evidence of pitting corrosion happening on the surface of the initial sample (Fig. 3a).

However, performing one pass ECAP on the sample accelerates the pitting corrosion sharply. This pitting corrosion could be the result of the existence of an intermetallic phase inside the Al matrix as suggested by Sharma and Ziemian [36]. Alloying elements in aluminum alloys could act as anode or cathode concerning the Al matrix which causes positive or negative effects on corrosion resistance. In other words, alloying elements are categorized into two classes. Type A shows anodic behavior concerning the Al matrix, and type C acts as a cathode and causes the dissolution of the Al matrix. According to Fig. 4, the Al-Mn intermetallic phase existed in the SEM micrograph of the onepass ECAPed sample before and after the corrosion test. As can be seen in Fig. 4b, the pitting corrosion mainly occurred around the Al-Mn intermetallic phase which is nobler than the Al matrix (type C). The same phenomenon was reported by Son et al.



Fig. 3- SEM micrograph before and after dipping in HNO₃ solution for a) unprocessed sample, b) one pass ECAPed sample, and c) two pass ECAPed sample (AI-Mn intermetallic phases are pointed with yellow arrows).

[37] which investigates the pitting corrosion of CP Al and Al 5052 alloy subjected to ECAP. They reported that in the case of Al 5052, the pitting corrosion happens mainly around Si particles despite Al-Fe intermetallic compound existence.

Also, Fig. 3c indicates performing two-pass ECAP enhances the corrosion resistance of the Al5085 sample and there is no evidence of pitting corrosion on the surface of the sample. This matter could be the result of two main reasons: first, intermetallic compounds break down by further straining of ECAP. As Miyamoto [18] suggested, this breakdown prevents intermetallic phases to act as nucleation sites; so pitting corrosion improves. Second is the state of residual stress. As the amount of residual stress changes the free energy state of the material, it affects the work function of the surface. It should be noted that the tension state of residual stress which causes the formation of cracks and defects on the passive film has a negative effect on corrosion resistance [25]. Also, Tanski et al. [38] reported that routes A and Bc of ECAP cause only tension residual stress but on the other hand route C leads to both tension and compressive residual stresses due to the rotation of the sample by 180° after each pass.

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4.conclusions

The effect of grain refinement by the ECAP process on mechanical properties and corrosion behavior of Al5085 alloy in HNO₃ solution was investigated. It was seen that after the first pass UTS, YS, and microhardness increases significantly by 24%, 44.5%, and 45%, respectively from the initial one. Also, after the second pass UTS, YS, and microhardness reached 388

MPa, 139.6 MPa, and 152 HV respectively. The elongation of unprocessed, one-pass processed, and two-pass processed samples was 23.5%, 20%, and 18.5%, respectively. It has been shown that

pitting corrosion resistance reduces after the first pass and many small pits have been formed on the surface of the sample.

Also, small pits mainly formed around Al-Mn intermetallic phases. There is no sign of pitting corrosion happening after performing the second



Fig. 4- SEM micrograph of one-pass ECAPed sample a) before dipping in HNO3 solution and b) after dipping in HNO₃ solution, c) EDS spectra elemental analysis, and d) calculated stable and metastable phases by JMatPro software. (Al-Mn intermetallic phases are pointed with yellow arrows).

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