Microstructure and Corrosion behavior of wrought AZ80 Mg alloys after the combined processes of ECAP and Hot Rolling

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Abstract. In the current work, wrought AZ80 Mg alloys were subjected to equal channel angular pressing (ECAP) followed by hot rolling (HR). Then, combined effects of ECAP and HR on microstructure and electrochemical corrosion behavior were investigated in 3.5wt.% NaCl solution. The microstructure and corrosion morphology study was made through optical and scanning electron microscope respectively. The results show that the use of hot rolling after ECAP significantly decreases the grain size compared to as-received and ECAP-4 pass processed Mg alloys. In addition, electrochemical impedance spectroscopy and potentiodynamic polarization results have shown that the hot rolling of AZ80 Mg alloy after ECAP exhibited lower corrosion current and higher corrosion resistance is due to fine grain microstructure and continuous and uniform distribution of secondary phases. This was evidently observed during this study.

Keywords: AZ80, Corrosion, Nyquist plot. ECAP, Hot-Rolling.

1. Introduction
Wrought Mg alloys generating an extensive interest in the area of current research. It offers low density, high specific strength, higher vibration absorption capacity and radiation shielding. Among that magnesium alloys is majorly used in automobile, aircraft and defence applications owing to its lower density. But Mg alloys have lower tensile strength and corrosion resistance properties [1]. Therefore, many researchers worked on equal channel angular pressing of Mg alloys to improve the mechanical properties and corrosion resistance. Lifei Wang et al. [2] have discussed the effect of grain size on mechanical properties after ECAP. The study showed that higher grain refinement was exhibited higher tensile strength. M. Eddahbi et al. [5] compared the microstructure, texture, and the thermal stability of an AZ31 magnesium alloy processed through ECAP and large strain hot rolling. As a result, the homogenous and slightly coarse microstructure obtained through ECAP, with an average grain size of \~7 µm. high strain hot rolling exhibited an average grain sizes nearby 3 µm, but it was quite heterogeneous. Moreover, the microstructure of material is thermally stable at 250°C up to annealing time of almost 10³ min. J.A. del Valle et al. [4] studied the effect of grain size reduction on work hardening and ductility in magnesium alloys after ECAP processing and rolling. The study reveals grain size reduction increases the work hardening capacity. Shahriar Koleini et al. [6] revealed that the corrosion tendency of as-received Mg–1Ca was remarkably decreases by hot rolling process due to the grain refinement. Fuyong Cao et al. [8] investigated the effect of hot rolling on the corrosion behaviour of Mg–X alloys (X = Gd, Ca, Al, Mn, Sn, Sr, Nd, La, Ce, Zr or Si) by mass loss tests in 3.5wt.% NaCl solution. From the it was observed that corrosion rates for all Mg–X alloys except Mg-0.1Zr and Mg-0.3Si reduced after hot rolling, attributed to fine-grained Mg alloys having a homogeneous microstructure, and uniform distribution of secondary phase (β) particles. G.F. Lima et al. [10] studied the hydrogen storage capacity of pure magnesium after the combined processes of ECAP and cold-rolling. The study revealed that the magnesium processed by ECAP with cold rolling technique produced the faster hydrogen absorption ability when compared to the ECAP processed
samples. Many authors worked on microstructure and tensile behavior of ECAP pressed magnesium alloys and corrosion behavior of hot-rolled magnesium alloys. But, it was noticed that very less literature available on corrosion behavior of Mg alloys after ECAP and ECAP with the rolled condition. In this study, made an attempt to study the microstructure and corrosion activities of as-received, equal channel angular pressing and subsequent hot rolling at 523K for further improvement of corrosion resistance.

2. Experiments
A wrought AZ80 Mg alloys, with a chemical composition (wt.%) of 91.5 Mg, 8 Al, 0.5Zn was used as the processing material. The Mg alloys were homogenization treated at 400 °C for 18 h and furnace cooled. The equal channel angular press consists of an Mg billet being pressed through a die, which contains two channels intersecting at die channel and corner angle, $\phi = 110^\circ$ and $\psi = 30^\circ$ respectively, route Bc is used as ECAP processing route, where the Mg sample would be rotated 90 degrees CCW direction between any two pressing processes [3]. During the ECAP pressing process, the sample of 16mm diameter, 80mm length was used and pressed with a ram speed of 1 mm/s and molybdenum disulphide (MoS$_2$) was used as a lubricant to minimize the friction between the billet and the ECAP-die. Further, hot rolling was performed on ECAP-4P sample with a 75% area reduction at a hot rolling temperature of 250°C. Thereafter, electrochemical corrosion performance of wrought AZ80 magnesium alloys in as-received, ECAP-4P and ECAP-4P followed hot rolled sample was investigated. Which was carried out using electrochemical corrosion analyzer, model: Gill AC-1684, supplied by Tech-science Pvt Limited, Pune (India) with an auxiliary electrode (AE) graphite (Gr) and the reference electrode (RE) saturated calomel electrode (SCE). 1cm$^2$ area of the working electrode (AZ80 alloy) was exposed to the 3.5wt.% NaCl solution. Then, microstructure and corrosion morphology study were done through optical microscopy (BIOVIS material plus) and Scanning Electron Microscopy (Model: JEO JSM–638OLA from JEOL, USA) operated at 30kV.

3. Results and discussion
3.1 Microstructure Evolution
Figure 1 represents the microstructure of as-received, ECAP-4P and ECAP-4P followed by hot-rolled wrought AZ80 Mg alloy. The alloy consists of $\alpha$-Mg and $\beta$-$\text{Mg}_{17}\text{Al}_{12}$ secondary phase, essentially distributed along the grain boundaries presented in Fig. 1 (a) and (b) [7,12]. An average grain size of the as-received sample was 90µm. Fig. 1 (b) presents the fine-grain microstructure of AZ80 Mg alloy after 4th pass of ECAP, here the average grain size was reduced to 88% compared to a the as-received alloy. Further, In Fig. 1(c) observed elongated grains along the rolling direction, the accumulation of dislocation density was increased after 75% area reduction of ECAPed Mg alloy and it is difficult to identify the single grain as shown in Fig.1 (c). Also, the hot rolling of ECAPed Mg alloys revealed the uniform distribution of secondary phase which is depicts in Fig. 1 (c).

![Figure 1](image_url) 
Figure 1. Optical image of wrought AZ80 Mg alloys a) as-received b) ECAP-4P c) ECAP-4P and hot rolled at 523K
3.2 Corrosion Behaviour

Unprocessed and processed samples were subjected to electrochemical impedance spectroscopy (EIS) test to investigate the corrosion behavior of wrought AZ80 Mg alloys. Figure 2 shows the Nyquist plots of specimens which were immersed in 3.5 wt.% NaCl solution. The diameter of arc in EIS plot is associated to the corrosion resistance of the specimen. Which signifies larger the arc higher the corrosion resistance vice versa [7,9]. From figure 2, it was seen that there is a second arc at the end of the capacitive arc of ECAP-4P followed by HR sample in the plot which shows an inductive mode initiated due to the breakdown of protective passive layer as shown in Fig.2. The diameter of capacitive arcs increased for ECAP-4P and ECAP-4P followed by HR compare to as-received sample. In figure 3 decrease in corrosion rate and an increase of charge transfer resistance was observed for ECAP processed specimen due to the occurrence of equiaxed ultra-fine grains and uniformly dispersed secondary particles. The crucial reason for the improvement in corrosion resistance of fine-grained materials is attributed to an enhancement in protective layer formation. On the other hand, the charge transfer resistance of the ECAPed sample is inferior to that of ECAP followed by hot rolling. Considerably higher value of Rct was found in ECAP followed by hot rolling as associated to ECAPed and as-received sample. The ultrafine-grained material provides better protection which prevents rupture of Mg(OH)2 layer which decreases the corrosion rate and also Mg17Al12 phases act as a barrier for the initiation of corrosion. A similar observation was made by Gopi K R et al. [11]

![Figure 2. Nyquist plots of unprocessed and processed samples](image-url)
3.3 Corrosion Morphology

Samples are investigated in 3.5 wt.% NaCl solution during electrochemical corrosion analysis, then the corrosion products are removed to observe the corrosion mechanism. Fig. 3 (a), (b) and (c) shows the surface morphology of as-received, ECAP-4P and ECAP-4P followed by hot rolling at 523K respectively. Fig 3 (a), as-received alloy have shown abundant corrosion pits along the grain boundaries. In addition, in comparison with Fig 3 (a), the surface area of corrosion pits was reduced in as-processed alloy shown in Fig 3 (b) and (c). This indicates that secondary beta phases (β-Mg17Al12) distributed after processing. Which reduces the number of cathodic cites, as a result, improves the corrosion resistance [9, 12].

Figure 3. SEM images of the corroded surface a) as-received b) ECAP-4P c) ECAP-4P and hot rolling at 573K
4. Conclusion
The current study reveals the differences in microstructure and basic corrosion behavior of wrought AZ80 Magnesium alloy of as-received, after ECAP and ECAP followed by subsequent hot rolling. Corrosion analysis was carried out by utilizing electrochemical corrosion technique. Based on the results and discussion the following conclusion were drawn.

1. Thermo-mechanical treatments by equal channel angular pressing followed by hot rolling were employed to achieve ultra-fine grain structure (11μm–2μm) in comparison with the as-received specimen (90 μm).
2. Potentiodynamic polarisation investigation showed that the corrosion rate of as-processed samples (ECAP and ECAP followed by HR) were lower than the un-processed samples.
3. The corrosion results confirm that hot rolling after ECAP is an effective technique to produce corrosion resistant material.

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