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Effect of deformation temperature on precipitation, microstructural evolution, mechanical and corrosion behavior of 6082 Al alloy

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Abstract: The influence of cryorolling (CR), room temperature rolling (RTR) and post annealing on precipitation, microstructural evolution (recovery, recrystallisation and grain growth), mechanical and corrosion behavior, was investigated in the present work. The precipitation kinetics and microstructural morphology of CR, RTR, and post annealed samples were investigated by differential scanning calorimetry (DSC), transmission electron microscopy (TEM), and electron back scattered diffraction (EBSD) to elucidate the observed mechanical properties. After annealing at 200 °C, UTS and hardness of CR samples (345 MPa and HV 127) were improved as compared to RTR samples (320 MPa and HV 115). The increase in hardness and UTS of CR samples after annealing at 200 °C was due to precipitation of β'' from Al matrix, which imparted higher Zener drag effect as compared to RTR samples. The improvement in corrosion and pitting potentials was observed for CR samples (−1.321 V and −700 mV) as compared to RTR samples (−1.335 V and −710 mV). In CR samples, heavy dislocation density and dissolution of $Mg_4Al_3Si_4$-precipitates in the Al matrix have improved corrosion resistance of the alloy through formation of protective passive layer and suppression of galvanic cell, respectively.

Key words: aluminum alloy; cryorolling; mechanical property; corrosion potential; precipitation

1 Introduction

Al−Mg−Si alloys are widely used as structural materials in the aerospace and automobile industries due to their high specific strength, corrosion resistance, and fracture strength [1]. According to Hall−Petch relation, ultrafine grained materials with the grain size ranging from 100 nm to 1 μm, exhibit very high strength and hardness but low ductility as compared to their bulk counterpart. The combination of high strength and ductility could be achieved in the ultrafine grained (UFG) materials with bimodal or multimodal microstructure features as reported in the literature [2]. Severe plastic deformation (SPD) processing has recently emerged as a novel approach to produce UFG and nanostructures (NC) in metallic materials. Several SPD techniques such as equal channel angular pressing (ECAP), high pressure torsion (HPT), accumulative roll-bonding (ARB), friction stir processing (FSP), room temperature rolling (RTR) and cryorolling (CR) are used to produce nanocrystalline and ultrafine grains in the bulk materials [3]. Among these techniques, CR has emerged as a new technique to produce UFG at relatively low true strains [4]. Cryorolling enables effective suppression of dynamic recovery, leading to very high dislocation density in the deformed materials, which subsequently facilitates formation of sub grain structures followed by UFG in the Al alloys as reported in the literatures [5−10].

The 6082 Al alloy is an age hardenable alloy and contains Mg and Si as major alloying elements. These elements are dissolved into the Al matrix at high solution treatment temperature (550 °C, 24 h). The precipitate formed in 6082 Al alloy was identified as Mg_2Si as reported in the earlier literature [11] but recent advanced characterization studies have revealed its chemical

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composition as $Mg_4Al_3Si_4$ [12,13], which imparts thermal stability to the alloy [14,15]. Cryorolling of the 6082 Al alloy easily suppresses any local heating and Mg/Si particle remains dissolved in the Al matrix during processing. The deformed Al alloys upon the post annealing treatment show high strength and ductility as compared to the bulk Al alloys [16−20]. The effect of deformation temperature on precipitation sequence, mechanical and corrosion behavior of 6082 Al alloy is limited in the literature. Although thermal stability of the UFG Al alloy was investigated, recovery, recrystallisation and grain growth of age hardenable bulk Al alloys subjected to thermo mechanical processing at room and cryogenic temperatures have not been subjected to detailed studies so far [21−26]. In the present work, mechanical and corrosion behaviors of CR and RTR 6082 Al alloy were studied and correlated with microstructural evolutions such as grain size and dislocation density. The effect of working temperature on $Mg_4Al_3Si_4$-precipitate evolution in the alloy was investigated. Annealing of the deformed 6082 Al alloy was performed between 100 and 400 °C to substantiate the influence of CR and RTR on recovery, recrystallisation and grain growth. The dislocation density and precipitation evolution and morphology were characterized by using X-ray diffraction (XRD), DSC and TEM. The recovery, recrystallisation and grain growth of the deformed Al alloy were investigated by using DSC, EBSD and TEM.

2 Experimental

The 6082-T6 (cast & rolled) Al alloy was procured from Hindalco Industries Ltd., Mumbai, India. The chemical composition (Al: 96.31, Si: 1.35, Mg: 0.67, Mn: 0.72, Fe: 0.248, Zn: 0.12, Cu: 0.062, Cr: 0.0698, Ti: 0.045 and remaining impurity, mass fraction, %) was confirmed by X-ray fluorescence (Rigaku supermini200). After solution treatment (550 °C, 24 h) [24], CR and RTR (unidirectional rolling) were carried out by using laboratory rolling machine up to true strain of 1.38. To maintain the working temperature, samples were dipped (during processing) in liquid nitrogen and water during CR and RTR, respectively.

Tensile samples were prepared according to sub size-ASTM E8M standards (gauge length: 26 mm, length of reduced section: 33 mm, length of grip section: 30 mm, width: 6.25 mm, width of grip section: 10 mm, radius of fillet: 6.25 mm, thickness: 6.25 mm). The test was performed on S-series, H25K-S tensile tester at room temperature. For each condition, six tensile samples were prepared to average out the tensile properties. For hardness measurements, at least 8 readings were taken and the average hardness value was reported. The mechanical polishing of samples, prior to mechanical testing, was carried out by using 320, 800, 1200, 1500 and 2000 grit emery papers followed by cloth polishing.

The cyclic polarization tests were conducted by using a Gamry potentiostat (interface 1000) in 3.5% NaCl environment to investigate the corrosion property of CR and RTR samples. To remove the oxide layer from the surface of the samples, potentiostatic test was performed before the cyclic polarization test with following conditions: initial φ of 0 V, initial time of 10 s, final φ value of 5 V, final time of 100 s, sample periods of 1 s, limiting J to 800 mA/cm^2, sample area of 0.785 cm^2, initial delay of 3600 s. The conditions chosen for cyclic polarization tests are as follows: initial delay of −0.5 V, apex φ of 1.5 V, final φ of 0 V, forward scan of 5 mV/s, reverse scan of 2.5 mV/s, sample periods of 1 s, apex J of 10 mA/cm^2, sample area of 0.785 cm^2, initial delay of 300 s. At least six readings were taken for each condition to ensure reproducibility of the results.

In EBSD sample to remove stress generated on surface due to cutting, mechanical and cloth polishing followed by electro-polishing were performed at 11 V for 90−120 s at −30 °C. Further, fine cloth polishing with colloidal silica was carried out to relieve remaining stresses from the surface. The electro-polishing conditions consist of Aplab 3010 DC power supply, solution (20% perchloric acid + 80% methanol), and reference electrode (stainless steel). The polishing of samples (3 mm in diameter and 0.1 mm in thickness) for TEM study was performed by twin-jet polisher as per the standard procedures.

For DSC measurement, 30 mg disc (5 mm in diameter and 0.6 mm in thicknesses) was prepared by mechanical polishing followed by using punch of 5 mm diameter. To investigate the precipitation effect of deformed Al alloy, pure Al of 30 mg disc was used as a reference sample, whereas to identify the recovery and recrystallisation effect in CR and RTR alloys, ST 6082 Al alloy was used as a reference sample during a DSC run. The DSC scan was carried out at a heating rate of 15 °C/min from 0 to 500 °C.

3 Results and discussion

3.1 X-ray diffraction

The XRD patterns of CR, CR followed by annealing, RTR and RTR followed by annealing are shown in Figs. 1(a) and (b), respectively. The formation of ($Al_{12}(Fe,Mn)_3Si$ and $Mg_4Al_3Si_4$)-phase and $Al_{12}(Fe,Mn)_3Si$-phase, was observed in CR and RTR samples, respectively. $Mg_4Al_3Si_4$-precipitate has dissolved into the Al matrix after ST treatment as explained in our earlier work [27]. In RTR sample, due to local heating or dynamic recovery during rolling,