



Effect of cryorolling and annealing on recovery, recrystallisation, grain growth and their influence on mechanical and corrosion behaviour of 6082 Al alloy



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H I G H L I G H T S

- The 6082 Al alloy process through cryorolling and followed by annealing.
- The recovery, recrystallisation and grain growth explained through EBSD, TEM and DSC.
- The recovery and recrystallisation occurs between 110 and 250 °C and 250–300 °C and beyond that grain growth started.
- Cryorolled samples have excellent mechanical property after annealing at 150 °C.
- Cryorolled samples annealed at 350 °C shows excellent corrosion resistance.

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In the present study, recovery, recrystallisation, grain growth, mechanical, and corrosion resistance of 6082 Al alloy subjected to cryorolling (CR) and annealing (AN) treatment were investigated. CR was performed to produce ultrafine grained structure and subsequently, AN treatment was given to investigate its influence on recovery, recrystallisation, and grain growth. The recovery and recrystallisation occurs between 110 and 250 °C and 250–300 °C respectively, where as grain growth starts from 300 °C due to insoluble dispersions (AlMn & AlMnSi) and Mg₂Si precipitates in the alloy as evident from Differential scanning calorimetry (DSC), Electron back scattered diffraction (EBSD), hardness and Transmission electron microscopy (TEM) results. CR 6082 Al alloy has showed increased hardness of 120 VHN and ultimate tensile strength of 353 MPa. Annealing of CR 6082 Al alloy at 150 °C exhibited maximum hardness, ultimate tensile strength (UTS) and ductility of 127 VHN, 362.5 MPa, and 11%, respectively. CR of the 6082 Al alloy has produced high density of dislocation and increase in grain boundary area, which enabled the formation of dense oxide film on surface, enhancing its corrosion resistance. CR samples annealed at 350 °C shows excellent corrosion resistance as compared to as deformed Al alloy.

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1. Introduction

The demand for 6XXX Al alloys has been ever growing in automotive, aero space, and marine structures due to their high specific strength, improved mechanical and corrosion resistance

properties [1,2]. Forty six percentages of Al alloys are currently used for making sheets and plates in various structural applications [3]. The 6082 Al alloy is used for fabricating hot extruded automotive parts due to its sufficient plasticity for extrusion, high ample strength, excellent weldability, formability and machinability [4]. Mechanical properties of Al alloys can further be enhanced by grain refinement. There are several severe plastic deformation methods used for producing ultrafine grain structure in the bulk alloys [5–7]. It is well reported in literature that accumulative roll bonding and equal channel pressing require very high true strain to produce

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ultrafine grains in the bulk alloys [8,9]. The requirement of large plastic strain for the formation of ultrafine grain is a demerit of these processes as it poses significant challenges in tool design and die geometries. To obviate these limitations, cryorolling has been identified as a potential technique to produce ultrafine grains as it requires less plastic strain [7]. UFG and nanostructured alloy produced are severely deformed structures, which restricts dislocation movement and hence ductility is reduced [10]. Large plastic deformation causes structural inhomogeneity in the alloy making it thermally unstable UFG structure [11,12]. UFG materials exhibit high driving force for grain growth compared to bulk materials [13]. Thermally stable UFG alloys with their high diffusion rate at high temperatures, show super plasticity at high strain rate [14,15]. 6XXX Al alloys are precipitation hardenable alloys, thus during annealing precipitates emerge out from Al-matrix and provide strength to material by grain boundary pinning [16].

The effect of annealing on UFG Al alloys has been investigated by various researchers [16–18]. Panigrahi et al. [16,17], reported that during annealing of 6063 and 7075 Al alloys, recovery, recrystallisation and precipitation occurs simultaneously, leading to a thermally stable structure up to 250 °C. DSC and TEM have been used to study the precipitation kinetics and thermal stability of UFG alloys [19,20]. The effect of heating rate and processing (CR & CR + Warm rolled) on precipitation of 6061 Al alloy has been investigated using DSC [21]. The precipitation kinetics in Al alloys is influenced by cold rolling [22] as the generations of defects like dislocation enhances precipitation by facilitating the nucleation events [21–23] and subsequently its evolution has been shifted to low temperature side as compared to solution treated (ST) Al alloy. DSC, TEM and EBSD are used to investigate the recovery, recrystallisation and grain growth in Al alloys.

The corrosion resistance was significantly influenced by ultra fine/nano grain structures of the alloy as reported in the literature [24,25]. Abdulstaa et al. [26], have reported that UFG 1050 Al alloy has promoted the formation of denser oxide film on its surface to protect against corrosion. Corrosion resistance of UFG Al alloys can be significantly improved by annealing at optimum temperature as reported in the literature [27]. The literature on recovery, recrystallisation, grain growth, mechanical, and corrosion behaviour of UFG 6082 Al alloy processed by CR is scanty. Therefore, the present work has been focused to study thermal stability, strength, and corrosion resistance of Al 6082 subjected to CR and post-annealing treatment. DSC, EBSD and TEM were used to characterize the recovery, recrystallisation, grain growth, of CR 6082 Al alloy and electrochemical measurements were made to understand its corrosion behaviour. The hardness and tensile tests were made on the alloy to substantiate the influence of CR and annealing treatment on the mechanical properties.

2. Experimental

The wrought (cast & rolled) 6082-T6 Al alloy has been procured from Hindalco Industries Ltd, Mumbai, INDIA. The composition of 6082 Al alloy (in wt%) was 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. Laboratory rolling mill has been used to process the alloy under cryogenic conditions. The initial sample size of $45 \times 30 \times 10 \text{ mm}^3$ was solution treated (ST) at 540 °C for 24 h to get homogenized grain structure followed by water quenching at room temperature. The ST sample was then ground using conventional grinder to remove the oxide films. The sample was then dipped in liquid nitrogen for 15 min to ensure the uniform temperature throughout its cross section. The sample was then cryorolled by laboratory rolling mill with 110 mm roller diameter and 8 RPM roller speed. A true strain of 0.061 was given during rolling in each

pass (0.6 mm thickness reduction) so as to achieve a total strain of 2.3 in 15 passes. The final dimension of sample after cryorolling was $450 \times 30 \times 1 \text{ mm}^3$ as shown in Fig. 1. To study the precipitates in ST, CR and CR+AN samples, X-ray diffraction (XRD) was carried out at 4°/min from angle 30° to 70°. The sample for XRD investigation was taken out from cryorolled sheet as shown in Fig. 1 and the same sample was used for EBSD measurement.

DSC measurements were carried out by using a Perkin Elmer Paris Diamond instrument. The samples were prepared by punching a disc of 5 mm having 30 mg weight. The inert environment has been maintained in furnace with help of nitrogen gas, flowing with a rate of 20 ml/min. All samples were tested from 0 to 450 °C temperature range and test was performed twice to ensure reproducibility. Before reloading a sample, furnace was cooled down to –5 °C for 3 min. Solution treated pure aluminium was used as reference material to study precipitation behaviour of ST and CR samples of 6082 Al alloy with heating rate of 10 °C/min using DSC. To study the effect of annealing on precipitate evolution, DSC was performed on CR+AN (150–400 °C) samples at heating rate of 10 °C/min. Recovery and recrystallisation peaks of CR samples were obtained during DSC runs by taking ST 6082 Al alloy as a reference sample. The baseline correction was performed to isolate the heat effects of transformation reactions corresponding to specimen.

To investigate the recovery, recrystallisation and grain growth, EBSD scan was made at a step size of 0.2 µm on Technai FESEM. The EBSD data was first cleaned by providing a grain tolerance angle 5° and grain size 2 (grain having two step size considered as grain). Subsequently, IPF (Inverse pole figure orientation map) image was extracted from EBSD data. To find out the grain growth in CR+AN samples, partition of IPF image was made by giving condition; Grain orientation spread <1° & grain size >4 µm. The grain boundary angle super imposed on image quality map of CR and CR+AN samples is used to estimate the fraction of low and high angle grain boundaries in CR+AN samples. The grain boundaries from 1.5 to 10° and 10–180° are taken as low and high angle grain boundaries, respectively, in the present study.

Transmission Electron Microscopy (TEM) sample were prepared by thinning the sample up to 0.1 mm thickness as per the standard procedure [21]. 3 mm diameter disc punched from the above sample thickness has been subjected to twin jet electro polishing, at –20 °C with 30 V DC power current for preparing very thin sample. Etchant solution containing 20% perchloric acid and 80% methanol was used. TEM characterization of the thin sample is performed by using FEI TECNAI 20 operated at a voltage of 200 KV.

To study the effect of annealing temperature on recovery, recrystallisation, grain growth and mechanical property, set of CR samples were subjected to a wide range of annealing temperatures ranging (CR+AN) from 150 °C to 400 °C for 1 h. Hardness testing and tensile testing of ST, CR, and CR+AN samples were carried out at room temperature. Bulk vicker hardness testing has been done using 10 kg F load with a dwell time of 15 s. Averages of eight readings were taken to get hardness. Ultimate tensile strength (UTS) test has been performed by S-Series, H25K-S tensile tester

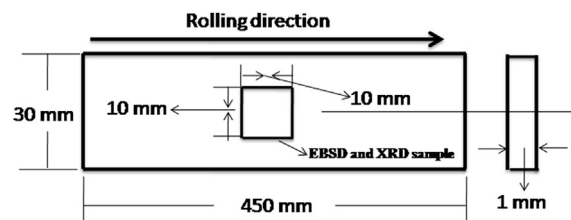


Fig. 1. Final dimension obtained after Cryorolling.

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