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Effect of deformation temperature on mechanical properties of ultrafine grained Al–Mg alloys processed by rolling

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ABSTRACT

Aluminum–Magnesium (Al 5083) alloy was subjected to cryorolling (CR) and cryorolling followed by warm rolling (WR) in order to investigate the changes in mechanical behavior and microstructure evolution in the present work. Al alloy specimens were first cryorolled up to 50% thickness reduction followed with warm rolling at 100 °C, 145 °C, 175 °C and 200 °C till to achieve total 90% thickness reduction. The final microstructure of all conditions were analyzed and compared through transmission electron microscopy (TEM), Electron back scattered diffraction (EBSD), and X-ray diffraction (XRD) techniques to investigate the effect of WR deformation temperatures on mechanical properties. The mechanical behavior of the processed samples were evaluated through hardness and tensile tests performed at room temperature. An increase in yield strength (522 MPa), ultimate tensile strength (539 MPa) and ductility (6.8%) was observed in WR specimens at 175 °C, hardness also increases to (146 HV) as compared to CR samples. These samples were annealed in temperature range from 150 °C to 300 °C to investigate their thermal stability. The CR samples exhibited severely deformed structure with high dislocation density network while cryorolled followed by warm rolled (WR) samples has shown formation of ultrafine grains associated with dynamic recovery. At elevated temperature of 200 °C, WR samples showed decrease in strength accompanied with increase in elongation due to dominant dynamic recovery effect led to reduction in dislocation density.

1. Introduction

Metals and their alloys with ultrafine grained (UFG) microstructure exhibit excellent combination of strength, and toughness as compared to bulk material. Therefore, production of ultrafine grained (UFG)/nanostructured material has attracted significant research interest worldwide. Different severe plastic deformation (SPD) techniques such as accumulative roll bonding (ARB) [1,2], multiaxial forging (MAF) [3,4], equal channel angular pressing (ECAP) [5–8], high pressure torsion (HPT) [9], repetitive corrugation and straightening (RCS) [10], are being explored since last decade to produce UFG/ nanostructured materials. The limitation of SPD is that it requires severe plastic strain, expensive tooling, design difficulties, production of relatively small quantities of material and cost associated makes it difficult to be used for industrial applications.

Deformation at cryogenic temperature has emerged as a potential route to develop UFG material with high density of dislocation for realizing the improved mechanical properties. Cryorolling suppresses the dynamic recovery during rolling at liquid nitrogen temperature, thereby enhancing the grain-refinement effect. It also obviates the drawback associated with SPD techniques. However, suppression of the dynamic recovery preserves the high density of dislocation defects, which could act as sites for recrystallization to produce fine grain structure [11]. Cryorolling has been used by many researchers to produce UFG materials from pure metals and their alloys. A good combination of high strength and ductility was achieved by developing a bimodal grain size distribution of nanocrystalline and ultrafine grains in pure copper and aluminum through simple approach of cryorolling followed with annealing [12,14]. However, this behavior was not found in pure nickel due to increase in strength is accompanied with loss of ductility [13]. The effect of cryorolling on microstructure and mechanical behavior of heat treatable Al–Cu alloy, Al 6061 alloy and subsequent annealing/aging treatment has been reported in literature [15–17]. Panigrahi et al. [18] have investigated the effect of rolling temperature, deformation strain and post cryorolling aging on the mechanical and microstructure evolution of Al–Mg–Si alloys. Recently effect of cryorolling on ECA pressed pure vanadium [19], and structural uniformity of nanocrystalline titanium has been reported [20].

Non heat treatable Al–Mg (Al 5083) alloy has excellent corrosion resistance, high strength to weight ratio and better formability makes is suitable for structural applications. Lee et al. [21] have

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reported that cryorolling followed by subsequent annealing is effective to develop ultrafine grained Al 5083 alloy with improved strength and good ductility. Recently, Kang et al. and Nageswarao Rao et al. [22,23] have reported the combined effect of CR followed by WR in Al 6061 alloy on mechanical behavior and evolution of microstructure, and observed significant improvement in their properties as well as for CR followed by WR with peak aging (CR + WR + PA) samples due to precipitation of second phase particles. Microstructural evolution and mechanical behavior has been investigated for Al 5052 alloy and observed the same findings of high strength after WR at 175 °C due to work hardening and formation of fine precipitates and high ductility on subsequent annealing attributed to formation of equiaxed fine grains and reduction in dislocation density due to recovery [24,25]. There is no reported literature till now on mechanical behavior and microstructural evolution during combined effect of CR followed with WR on Al–Mg (Al 5083) alloy. Therefore, an attempt has been made in present study to investigate the combined effect of work hardening, grain refinement, and dynamic recovery on the mechanical behavior, microstructural evolution and strengthening mechanism of Al–Mg alloy processed by cryorolling followed by warm rolling at different temperatures (100 –200 °C).

2. Experimental procedure

Commercially available Al–Mg alloy plate with chemical composition as shown in Table 1 has been taken as starting material in the present work. Samples with $40 \times 30 \times 10\ \text{mm}^3$ were cut from as received plate and were solution treated at 510 °C for 2 h and then quenched in water, resulted in homogenized structure with approximately 85 μm grain sizes. Fig. 1 shows the schematic illustration of cryorolling (CR) and warm rolling (WR) process. Samples were deformed at liquid nitrogen temperature up to 0.7 and 2.3 true strain values. Cryorolled samples with 2.3 true strain deformations are denoted as CR. During cryorolling, samples were dipped into liquid nitrogen for 15 min for initial pass and then 10 min for the successive passes. Cryorolled samples up to 0.7 true strains were further deformed at 100 °C, 145 °C, 175 °C and 200 °C by using oil bath furnace. Soaking time given for each pass was 4 min and these samples are denoted as cryorolled + warm rolled (WR) samples. The total reduction achieved was 90% (equivalent true strain of 2.3) and the reduction per pass was 4%. Samples were rolled on two high laboratory mill having rolling speed and roll diameter of 8 rpm and 110 mm respectively. Mechanical behavior was investigated by performing tensile testing and Vickers hardness testing at room temperature. Samples for tensile testing were machined along the plane parallel to the rolling direction according to the ASTM: E8 sub size specimen of 25 mm gauge length. The strain rate of $5 \times 10^{-4}\ \text{S}^{-1}$ was used for all the samples. Vickers hardness testing was also performed on plane parallel to rolling direction with 5 kg for 15 s. From the hardness data WR at 175 °C was selected which has shown maximum hardness. In order to investigate the microstructure and thermal stability of selected condition (WR at 175 °C), samples were further annealed between temperature range 150–300 °C for 1 h in a muffle furnace.

Microstructure of starting material, CR (2.3 true strain) and WR was studied through electron back scattered diffraction analysis (EBSD) and transmission electron microscopy (TEM). TEM examination was carried out on FEI Technai 20 transmission electron microscope operated at 200 KV; the samples were prepared by twin jet polishing of 3 mm disk with solution of 20% perchloric acid and 80% methanol at −40 °C temperature, 40 V. EBSD was performed on FEI, Quanta 200F using TSL OIM analysis 4.6 software developed by TEXSEM laboratories Inc. EBSD samples were prepared by mechanical polishing followed by electro-polishing at −15 °C using solution of 80% methanol and 20% perchloric acid at potential of 11 V. All microstructure observations were made at the mid-thickness of the rolled specimens. In EBSD scans, step size of 0.1 μm was used for all samples. In order to investigate the thermal behavior of CR, WR samples differential scanning calorimetry (DSC) was performed under nitrogen atmosphere using Perkin Elmer Paris Diamond DSC. Samples weight is 30 mg and annealed pure aluminum is used as reference sample. The scan rate used in the current study was 25 °C/min.

Table 1
Chemical composition of the alloy used in present study.

Components	Mg	Mn	Cr	Si	Fe	Cu	Al
Wt%	4.6	0.7	0.08	0.07	0.17	0.02	Balance

3. Results and discussion

3.1. Effect of warm rolling on mechanical properties and microstructure

The Vickers hardness of starting material after solutionizing and water quenching is 58 HV. After deformation at liquid nitrogen temperature (Cryorolling, CR) up to true strain of 2.3 (90% reduction), the hardness has increased from 58 HV to 127 HV. The increase in hardness of CR sample is due to the microstructure refinement occurred during cryorolling. To investigate the effect of warm rolling on cryorolled samples, warm rolling was performed at different temperatures of 100, 145, 175, 200 °C on 50% cryorolled samples up to 90% reduction. The effect of cryorolling followed by warm rolling (WR) performed at various temperatures on hardness of Al 5083 alloy are shown in Fig. 2a. WR performed at 100 °C has shown hardness value of 134 HV which is 7 HV more than CR condition. With further increase in WR temperature up to 175 °C, the hardness value increases to 146 HV and on WR at 200 °C, the hardness decreases to 138 HV.

The ultimate tensile strength (UTS) and yield strength(YS) of starting solution treated material has significantly improved after cryorolling up to 90% reduction (UTS-277 to 478 MPa, YS-168 to 460 MPa), but its elongation to failure is decreased from 22% to 3% (Fig. 2 b) which is distinctive behavior of cold worked samples. Whereas warm rolling of 50% cryorolled sample at 100 °C increased not only tensile strength but also ductility (Fig. 2b). With increasing warm rolling temperature from 100 °C to 175 °C simultaneous improvement in strength and ductility is observed. The further increase in warm rolling temperature to 200 °C led to increase in elongation to failure but decrease in tensile strength. The ultimate tensile strength achieved in WR sample at 175 °C is (539 MPa) is nearly two times more than that of ST sample (277 MPa) and 13% more than CR sample. Furthermore the elongation to failure in WR sample at 175 °C is 6.8% which is more than CR sample (3%). The improvement in mechanical properties achieved in Al 5083 alloy through cryorolling followed by warm rolling are similar to earlier reported results on Al 6061 alloy [23]. Kang et al. [25], has observed formation of fine precipitates, during warm rolling, attributed to significant improvement in mechanical properties observed in Al 5052.

From the hardness and tensile testing data, it is reasonable to state that to achieve better strength and ductility in Al 5083 alloy, cryorolling followed by warm rolling at 175 °C is an optimum condition. The mechanical behavior of CR and WR samples can be well realized by examining the microstructural changes. WR at 175 °C sample has been chosen for detailed microstructural study. The optical microstructure of as received material consisting of equiaxed grains after solutionizing treatment is shown in Fig. 3.