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Effect of cryorolling and warm rolling on precipitation evolution in Al 6061 alloy

P. Nageswara Rao [a], B. Viswanadh [b], R. Jayaganthan [a,*]

[a] Department of Metallurgical and Materials Engineering, Indian Institute of Technology Roorkee, Roorkee 247667, Uttarakhand, India
[b] Materials Science Division, Bhabha Atomic Research Center, Mumbai 40085, Maharashtra, India

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ABSTRACT

In the present investigation, the effect of deformation on precipitation sequence and microstructural evolution in Al 6061 alloy was studied using low temperature differential scanning calorimetry (DSC) and transmission electron microscopy (TEM). The precipitation sequence was greatly influenced by deformation in cryorolled and warm rolled samples. At low temperatures (<150 °C), two distinct cluster peaks have been observed upon cryorolling (CR). At high temperatures (>150 °C), the peak corresponding to β' formation has disappeared in the deformed alloy as compared to undeformed coarse grained material. TEM investigation of the deformed alloy revealed bimodal distribution of precipitate sizes with both very fine and coarse structures. Pre-deformation of the alloy led to the simultaneous formation of β'' and β' precipitates. Activation energies of clusters and major strengthening phase formation were calculated for the cryorolled and warm rolled materials and compared with the undeformed coarse grained material. The reduction of free energy for the formation of strengthening phase has occurred in the deformed material as compared to undeformed bulk material.

1. Introduction

Al 6*xxx* is the potential and versatile material for automobile body panel applications due to its light weight, good strength, corrosion resistance, and good precipitation hardening response [1]. Traditionally, these alloys are strengthened by solid solution and precipitation hardening. Mg and Si are the primary elements in the alloy and forms metastable nanometer scale precipitates of Mg_2Si to strengthen the matrix through proper heat treatment. Other traditional strengthening mechanisms such as grain boundary and dislocation strengthening through cold working supplements to precipitation hardening of the Al alloys. The precipitates and grain boundaries serve as barriers to dislocation motion, and consequently, increase the macroscopic strength of the material [2]. In the last two decades, many techniques were developed to reduce grain size of the material from several micrometers to submicron or nanometer region. Such techniques are equal channel angular pressing (ECAP) [3], accumulative roll bonding (ARB) [4], high pressure torsion (HPT) [5], multi-axial forging (MAF) [6] and cryorolling (CR) [7]. CR has been widely used to reduce the grain size of several metals and alloys. Combined enhancement of strength and ductility has been achieved in precipitation hardenable alloys by cryorolling followed by warm rolling. As cryorolled alloys provide increased yield strength through grain refinement and high dislocation densities but often show inadequate ductility [8]. The post-cryorolling heat treatment through low temperature ageing of the Al alloy led to simultaneous enhancement in strength and ductility through nanoprecipitation as reported in the literature [8]. The sequence, size, type and distribution of precipitates play a crucial role in determining the final properties of the material. Precipitation kinetics and its evolution of bulk Al alloys have been studied by several investigators using DSC and TEM [9,10]. The introduction of dislocations influences the precipitation kinetics and its sequence by providing heterogeneous nucleation sites and thereby accelerated precipitation kinetics [1,11,12]. The effects of cold rolling on the precipitation processes in Al–Mg–Si alloys have been studied extensively [1,13–18]. Lee et al. have reported the enhanced precipitation reaction and altered precipitation sequence by cold rolling in Al alloy [13]. Christman et al., Panigrahi et. al, and Matsuda et al. have reported that cold rolling accelerates the ageing process with increasing dislocation density [14,15,19]. Hirata and Matsuo have reported that plastic deformation reduces the activation energy for the intermediate β' phase formation and the needles nucleate preferentially on dislocations [14]. The kinetics of hardening has been enhanced by two ways: (i) adding alloying element, Cu to Al–Mg–Si resulted in enhanced hardening kinetics [20,21]. (ii) Presence of structural defects such as dislocations and low angle grain boundaries in cryorolled Al alloys has

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[*] Corresponding author. Tel.: +91 1332 285869; fax: +91 1332 285243.
E-mail address: rjayafmt@iitr.ernet.in (R. Jayaganthan).

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promoted faster precipitation kinetics through enhancement of the nucleation events [16].

The stimulation of the precipitation process could be attributed to introduction of high dislocation densities in the Al alloy due to cryorolling immediately after solution treatment. Hence, it is essential to render an insight into the precipitation hardening behavior of cryorolled material for optimizing the properties and realize the simultaneous improvement of strength and ductility in Al–Mg–Si alloys. The microstructural evolution and mechanical properties of Al 6061 alloy processed through CR and CR followed by WR conditions were reported in our earlier study [22]. The WR material has shown 27% improvement in UTS (390 MPa) as compared to CR material. Further, low temperature ageing of WR material resulted in 7% improvement in UTS. The literature on precipitation kinetics of cryorolled and warm rolled alloys and their influence on mechanical properties are very limited. Therefore, the present work has been envisaged to investigate the precipitation kinetics in deformed Al alloy through DSC and TEM, which would enable to elucidate their role in deformation behavior of ultrafine grained materials.

2. Experimental details

The material used in the present investigation was Al 6061 alloy with chemical composition (in wt%) of 1.01 Mg, 0.67 Si, 0.28 Fe, 0.2 Cu, 0.04 Mn, 0.05 Cr, 0.06 Zn, remaining Al. Samples with $10\times30\times40\ \mathrm{mm}^3$ were machined from the as received material and subjected to solution treatment (ST) at 530 °C for 3 h followed by cold water quenching to room temperature. To study the effect of deformation on the precipitation behavior and its kinetics, ST Al 6061 alloy was subjected to two different deformation processes as shown in Fig. 1. In the first process, the ST material was deformed up to 90% thickness reduction through cryorolling (CR). Cryorolling was performed by dipping the samples in liquid nitrogen for 15 min prior to rolling [22]. The final thickness is achieved through several passes with thickness reduction of ~4% given in each pass. In the second process, ST material was deformed up to 70% through CR followed by deformation up to 66% through warm rolling (WR) at 145 °C. The detailed procedure for WR has been discussed elsewhere [22]. The WR in the present study refers to the CR alloy subjected to warm rolling.

DSC measurements were carried out by using a Perkin Elmer Paris Diamond DSC under a nitrogen atmosphere at the rate of 20 ml min^{-1} to reduce the oxidation. All the samples were tested from −5 °C to 450 °C and tests were run at least two times to ensure reproducibility. DSC was precooled up to −10 °C before loading the samples and allowed for 3 min to get equilibrated before starting the test. ST, CR and WR materials are used as specimens and pure Al annealed at 450 °C for 3 h followed by furnace cooling was used as a reference material for all the specimens. The weights (30 mg) of both specimens and reference material were maintained constant for DSC testing. All conditions were tested with heating rates of 10, 15, 20 and 25 °C/min. For ST condition, samples with 5 mm diameter and 0.6 mm thick were punched and subjected to ST at 530 °C for 3 h followed by water quenching to room temperature were used. Whereas in CR and WR conditions, 5 mm discs were punched from processed sheets and then used for DSC testing. Baseline correction was performed to isolate the heat effects of transformation reactions corresponding to specimen from the data of reference sample.

Vickers hardness measurements were taken on the samples of ST, CR and WR material aged up to various temperatures with 20 °C/min heating rate. Load with 5 kg and 15 s dwell time was used for Vickers hardness measurements. In order to substantiate the DSC and Vickers hardness results, the microstructural investigation was performed along normal plane. Transmission electron microscopy (TEM) investigation was carried out in a FEI Technai 20 electron microscope operated at 200 kV. For TEM sample preparation, ST, CR and WR materials were mechanically ground up to 0.1 mm thickness using 600, 800, 1000 and 12000 grit size emery papers and subsequently thinning by electro-polishing using TenuPol-5 digitally controlled automatic electro-polisher. The electrolyte with 20:80 Perchloric acid to Methanol was used at −40 °C, 30 V for the electropolishing of samples.

3. Results and discussion

3.1. DSC studies

Fig. 2a shows DSC thermograms of Al 6061 alloy of ST, CR and WR conditions obtained with the heating rate of 20 °C/min over the temperature range of 0 to 450 °C. The close up of peaks formed at low temperatures (from 0 to 150 °C) is shown in Fig. 2b. The standard precipitation sequence in the material after solution treatment as reported in the literature is as follows [23–25]: super saturated solid solution (SSS)→clusters→G.P. zones→$\beta''\rightarrow\beta''$ and $\beta'\rightarrow\beta$ (Mg_2Si) phase. In the DSC thermogram of ST material (Fig. 2a), the number

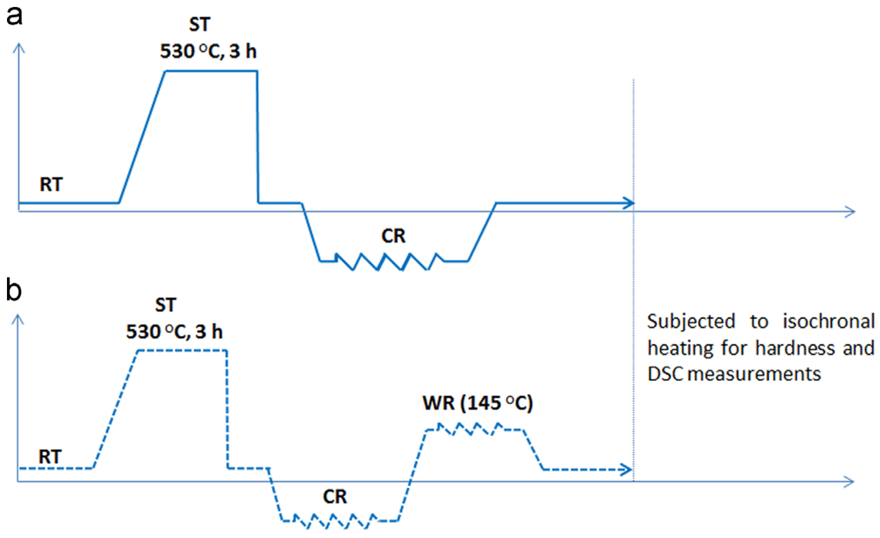

Fig. 1. Heat treatment flow charts of CR and WR.