



MRS Singapore - ICMAT Symposia Proceedings
7 th International Conference on Materials for Advanced Technologies

Enhancement in Strength and Ductility of Al-Mg-Si alloy by Cryorolling followed by Warm rolling

P.Nageswara rao^a, Amritha Kaurwar, Dharmendra Singh^a, R.Jayaganthan^{a,*}

Department of Metallurgical and Materials Engineering, Indian Institute of Technology Roorkee,

Roorkee 247667, India

Abstract

The effect of cryorolling followed by warm rolling (CR+WR) on strength and ductility of Al- Mg-Si alloys has been studied in the present work. Ultrafine grained Al 6063 alloy was developed through cryorolling up to 80% and warm rolling up to 50% at 200 °C followed by ageing at 100 °C for 22 hr. It is compared with the material processed through cryorolling followed by low temperature ageing. By implementing this new approach, simultaneous improvement in strength (290 MPa) and ductility (15 %) was achieved in Al 6063 alloys. The microstructure and precipitate evolution were characterized by Optical microscope, Electron back scattered diffraction (EBSD) and Transmission electron microscope (TEM) techniques. The ultrafine grain structure with 150-300 nm was observed in CR+WR sample. Mechanical properties were studied by performing hardness and tensile testing at room temperature. Strength and ductility of CR+WR samples are found to be superior to CR sample after peak ageing treatment.

© 2013 The Authors. Published by Elsevier Ltd.

Selection and/or peer-review under responsibility of the scientific committee of Symposium [Advanced Structural and Functional Materials for Protection] – ICMAT.

Keywords: Cryorolling; Warm rolling; Al-Mg-Si alloy; Mechanical properties; EBSD; TEM.

1. Introduction

Al 6063 is a medium strength age hardenable alloy used for automotive and aerospace applications due to its high strength to weight ratio, better weldability and higher corrosion resistance [1-4]. The primary strengthening mechanisms for Al 6063 alloys are precipitation hardening and work hardening [5]. Another effective technique to strengthen the Al alloys is developing ultrafine grain structure (UFG) in the material [6-7]. Severe plastic deformation (SPD) is an approach being used to realize the UFG/nanocrystalline structure in the material by imparting severe strains [8]. SPD techniques involve heavy tooling and it is expensive. . Alternative to the SPD process is deforming the material at very low temperatures (near or at the liquid nitrogen temperature), the so called cryorolling, suppresses the dynamic recovery and in turn enhances the accumulated dislocation density. Proper heat treatment of the cryorolled samples results in development of UFG structure in

* Corresponding author. Tel.: 91-1332-285869; fax: 91-1332-285243.

E-mail address: rjayafmt@iitr.ernet.in

the material. The combination of precipitation hardening, solid solution strengthening, work hardening, and grain boundary strengthening can give better strengthening effect to the material [9-10]. A significant improvement in mechanical properties was observed in precipitation hardenable Al alloys, by deforming at supersaturated solid solution (SSSS) state and post deformation annealing and ageing treatments [11-12]. In precipitation hardenable alloys, during post deformation ageing, the excess alloying elements will precipitate as a fine second phase particles [13]. These acts as an obstacle for the movement of dislocation, thus strength gets improved [14]. During post deformation ageing, work hardening effect will reduce due to annihilation of dislocations. This will improve the ductility but reduce the strength of the alloy. In recent published literature, it was observed that combination of cryorolling followed by warm rolling has substantial effect on increasing strength and ductility of the material [15-16]. But there is no reported literature on the effect of mechanical and microstructure characteristics of 6063 Al alloy subjected to cryorolling and warm rolling. Therefore, the present study is carried out to analyze the combined effect of work hardening, dynamic ageing and grain refinement on the mechanical behavior and microstructural morphologies of Al 6063 alloy processed by cryorolling followed by warm rolling.

2. Experimental details

Al 6063 alloy used in the present investigation with chemical composition of 0.5% Si, 0.45% Mg, 0.13% Mn, 0.058% Fe, 0.015 % Cu (wt %) and the balance Al, was procured from Hindalco industries, Pvt. Ltd, India. Samples with $10 \times 30 \times 40$ mm³ dimensions were cut from the bulk and annealed at 510 °C for 1 hour and then water quenched to room temperature. These samples were kept in freezer at -20 °C to prevent room temperature ageing, till to perform rolling. Annealed and water quenched samples were rolled by using conventional rolling mill at or near to the liquid nitrogen temperature. For this, samples were dipped in liquid nitrogen at least 10 min before and after each rolling pass. Thickness reduction given per pass was 4% and samples with thickness reductions of 80% and 90% were obtained by giving several passes. These samples were designated as (cryorolled) CR 80% and CR for further reference. CR 80% samples were subsequently warm rolled at 200 °C up to 50% reduction. In each pass, 20% reduction was given. Warm rolling was performed by heating the samples in muffle furnace for 4 min before and after each pass. Total time taken to perform warm rolling was 10 min. These samples were designated as CR+WR. In order to study the effect of low temperature ageing on samples processed through CR and CR+WR, samples were kept at 100 °C temperature for 60 hr. Hardness was measured at room temperature using a Vickers hardness testing machine at test load of 5 Kgf with 15 s dwell time. The reported values of Vickers hardness number is an average of at least 6 measurements. Microstructure of initial material ST, and CR was investigated through optical microscopy using poultons reagent as an etchant under polarized light. Electron back scattered diffraction (EBSD) analysis and Transmission electron microscopy (TEM) was used to study the microstructural changes in CR, CR+WR and their peak aged conditions. The sample preparation for EBSD and TEM is discussed elsewhere [17]. TSL OIM analysis 4.6 software developed by TEXSEM laboratories Inc. was used to analyze the EBSD maps. For tensile testing, samples were prepared according to the ASTM subsize specimen with 25 mm gauge length along the plane parallel to the rolling direction (RD-TD). Minimum four samples were tested to get tensile test results. The strain rate used for tensile testing was 5×10^{-4} /s.

3. Results and discussion

3.1 Microstructure: The optical micrographs of initial solution treated, and CR were taken on the plane parallel to the rolling direction (RD-TD) as shown in Fig.1. The initial microstructure shows equiaxed morphology with an average grain size of ~100 µm. Fig.1.b shows CR 80% sample with deformed grains along the rolling direction. With increasing deformation up to 90% the grains got perturbed due to heavy deformation. The EBSD orientation micrographs of CR 90% and CR80%+WR 50 % al 6063 alloy with inverse pole figure are shown in Fig. 2. For EBSD analysis, data which is having CI value more than 0.035 is considered. EBSD scans are performed with step size 0.1 µm for CR and CR+WR samples. In the EBSD maps, grey and black line shows low angle grain boundaries (LAGB) (1.5° - 15°) and high angle grain boundaries (HAGB) ($\geq 15^\circ$). Cryorolled with 90 % reduction sample reveals predominantly with LAGB, which corresponds to the dislocation cell structures elongated in the rolling direction as indicated with arrow mark in the Fig. 2.a. Whereas CR+WR in Fig.2.b shows duplex microstructure of equiaxed subgrains along with elongated grain structure. Fig.2 c, d shows the TEM micrograph of CR and CR+WR sample. In CR sample, the formation of the slightly elongated dislocation cell structure with an average size of 500 nm was observed. The boundaries of this cell structure are of LAGB ($\leq 15^\circ$). These cell walls are formed with complex dislocation tangles [18]. The microstructure of CR+WR sample in Fig. 2d has shown formation of sub grains structure with an average size of 150-300 nm. The dislocation density is slightly decreased as

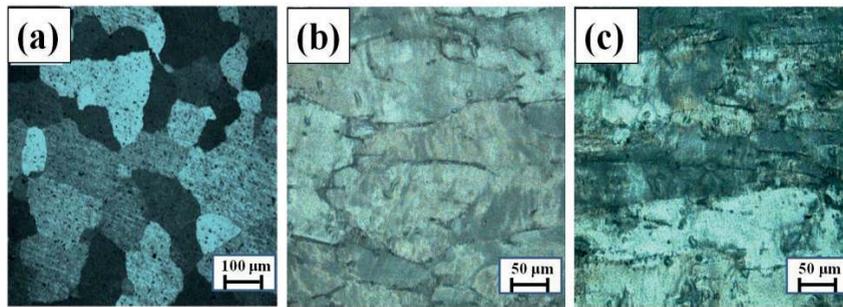


Figure 1: Optical micrographs of Al 6063 alloy; (a) Initial material (ST), (b) CR 90%

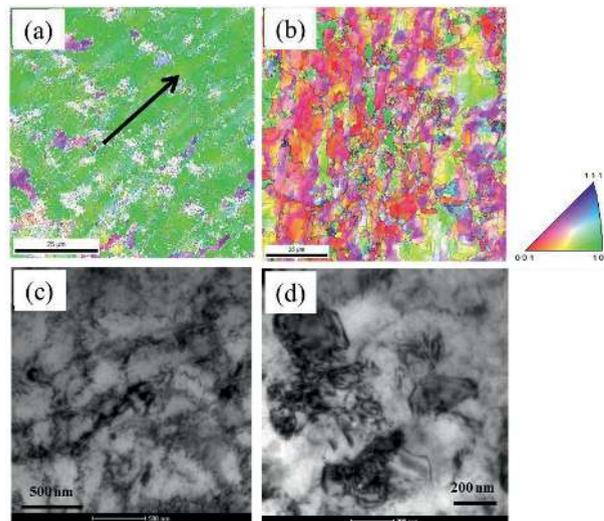


Figure 2: EBSD orientation maps and TEM micrographs of CR and CR+WR of Al 6063 alloy with the [001] inverse pole figure; (a), (c) CR, (b), (d) CR+WR.

compared to CR sample due to dynamic recovery effect during warm rolling at 200 °C. The TEM micrographs of CR+PA, WR+PA samples are shown in Fig.3. The dislocation density is reduced in CR and CR+WR sample after peak ageing treatment due to static recovery effect. During static recovery process, the tangled cell walls formed during cryorolling has transforming into more regular dislocation network to form sharp low angle grain boundaries. The dislocation content inside the subgrains got reduced, but still there are some regions remained with high density of dislocations. Fig. 3c shows very fine β'' needle shaped Mg_2Si precipitates with 10-15 nm diameters aligned in particular orientation. These β'' needle shaped precipitates were not clearly seen due to presence of dislocations.

3.2 Mechanical Properties: Fig.4 (a) shows the Vickers hardness values of initial solution treated, CR, CR+WR and its peak aged condition samples. The observed hardness of initial solution treated material is 40 Hv. After cryorolling up to 90% reduction, the hardness of the material has increased to 91 Hv (nearly 115 %). This rise in hardness is mainly attributed to solid solution strengthening and the presence of high dislocation density in CR samples by generation and accumulation of dislocations during rolling process at very low temperature. The yield strength (YS) and ultimate tensile strength (UTS) of the material after cryorolling up 90 % reduction has increased from 50 MPa to 234 MPa and 102 to 245 MPa, respectively. The improvement achieved in YS and UTS is 368% and 140%, respectively. Thus cryorolling had greater influence on YS than UTS [19]. But the tensile ductility has reduced from 30 % to 4.5 %. The hardness increased in

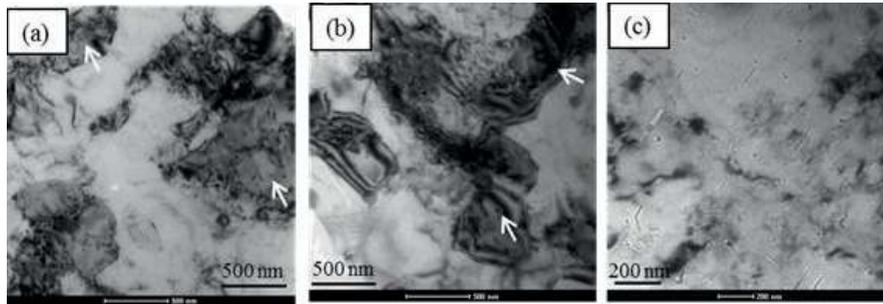


Figure 3: TEM micrographs of cryorolled (CR) and (CR+WR) of Al 6063 alloy at peak aged conditions (a) CR+PA, (b) CR+WR+PA, (c) Fine β' needle shaped precipitates observed in CR+WR+PA sample.

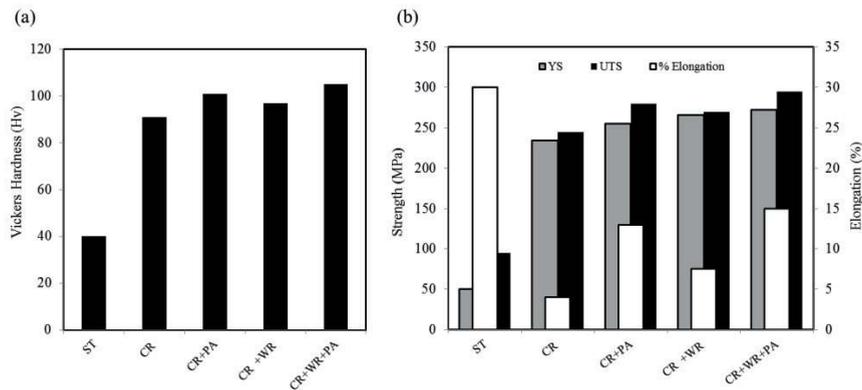


Figure 4: Variation in mechanical properties of Al 6063 alloy processed under various conditions; (a) Vickers hardness (b) UTS, YS and % Elongation

cryorolled followed by warm rolled (CR+WR) samples is from 40Hv to 97 Hv (142%). It is nearly 12% more than the CR samples. The improvement in yield strength and ultimate tensile strength in CR+WR samples compared to CR samples are 14% and 10%, respectively. The significant improvement in YS and UTS of CR+WR samples can be attributed to the fine precipitate particles evolved during warm rolling, which will act as barriers for dislocation movement and increases the dislocation accumulation. Formation of precipitates during warm rolling not only compensates the loss in strength by annihilation of dislocation due to dynamic recovery, but enhances the strength further. Simultaneously with the improvement in the strength and hardness, the percentage elongation of CR+WR materials is increased up to 7% which is double to the CR sample. The enhancement in the ductility is the result of dynamic recovery and restoration during warm rolling process. In our earlier studies [16] on Al 6061, we have observed that the percentage elongation after CR+WR at 145 °C was only 5 %. Gang et al., [19] have reported in Al 6061 after cryorolling followed by warm rolled at 175 °C was only 5 %. The same author has observed in Al 5052 alloy [15] processed at warm rolling temperature of 250 °C after cryorolling, the percentage elongation was nearly three times of the cryorolled sample with sacrificing strength. From the above observation, it can be concluded that warm rolling temperature has significant effect on strength and ductility of the material. In order to see the effect of low temperature ageing on strength and ductility CR and CR+WR samples were aged at 100 °C. The peak hardness of CR, and CR+WR samples were observed at 38 h and 22 h respectively. The YS and UTS of CR+PA sample were 255 MPa and 280 MPa respectively. The percentage elongation increased from 4.5 to 12 %. In CR+WR+PA sample, the improvement in UTS is slightly higher (~ 3.5) than the CR+PA sample. But the improvement in percentage elongation (15%) of CR+WR+PA sample is significantly more than the CR+PA sample. It is proved that cryorolling followed by warm rolling and low temperature ageing is superior to cryorolling and ageing to increase the strength and ductility of precipitation hardenable Al alloys. The important factors which are facilitating to give better

strength and ductility of CR+WR+PA materials are as follows: 1) Accumulation of high dislocation density during cryorolling increases strength, 2) Dynamic recovery and restoration during warm rolling leads to formation of ultrafine sub grain structure and annihilation of dislocations contribute for the enhancement in ductility, 3) Formation of very fine nano-sized β'' needle shaped precipitates during warm rolling and further low temperature ageing enhances the accumulation of dislocations surrounding to it and it will acts as barrier for dislocation motion leads to simultaneous improvement in strength and ductility.

4.0 Conclusions

- 1) CR +WR at 200 °C resulted substantial improvement in strength (UTS-270 MPa) and ductility (7%) over CR (UTS-245 MPa, ductility-4.5%).
- 2) Dynamic ageing during warm rolling resulted, formation of fine precipitates caused to increase in strength. Dynamic recovery and restoration during warm rolling caused to annihilation of dislocations and formation of subgrain structure through dislocation rearrangement.
- 3) The strength and ductility of CR+WR samples has further increased upto 290 MPa, 15 % respectively after low temperature ageing at 100 °C for 22 hr. It can be attributed to decrease in dislocation density further by annihilation of dislocations due to static recovery effect and precipitation of remnant excess solid solution from the matrix.

References

- [1] Chakrabarti, D.J., Laughlin D.E. Phase relations and precipitation in Al–Mg–Si alloys with Cu additions, *Progress in Material Science*, 2004, 49, pp. 389–410.
- [2] Gupta A.K., Llyod D.J., Court S.A. Precipitation hardening process in an Al-0.4%Mg-1.3%Si-0.25%Fe aluminium alloy, *Material Science Engineering A*, 2001, 301,140-146.
- [3] Tsao, C.S., Chen, C.Y., Jeng U.S., Kuo T.Y. Precipitation kinetics and transformation of metastable phases in Al-Mg-Si alloys, *Acta Materialia*, 2006, 54, 4621-4631.
- [4] Esmacili, S., Wang, S.X., Lloyd, D.J., Poole W.J. On the precipitation-hardening behavior of the Al–Mg–Si–Cu alloy AA6111, *Metallurgical and Materials Transactions A*, 2003, Vo. 34A, 751-763.
- [5] Smith, W.F. *Structure and Properties of Engineering Alloys* [M], New York, McGraw-Hill, 1993.
- [6] Valiev, R.Z., Alexandrov, I.V., Zhu, Y.T., Lowe, T.C. Paradox of strength and ductility in metals processed by severe plastic deformation, *Journal of Material Research*, 2002, 17, pp. 5–8.
- [7] Hong, S.J., Kim, T.S., Kim, H.S., Kim, W.T., Chun, B.S. Microstructural behaviour of rapidly solidified and extruded Al-14 wt. %Ni-14 wt. % Mm (Mm, Misch metal) alloy powders, *Material Science Eng A*, 1999, 271, pp. 469–476.
- [8] Valiev, R.Z., Langdon T.G., “Principles of equal-channel angular pressing as a processing tool for grain refinement”, *Progress in Material Science*, 2006, 51, pp. 881-981.
- [9] Cai, M., Field, D.P., Lorimer, G.W., A systematic comparison of static and dynamic ageing of two Al–Mg–Si alloys, *Material Science Engineering A*, 2004, 373, pp. 65–71.
- [10] Roven, H.J., Liu, M., Werenskiold, J.C. Dynamic precipitation during severe plastic deformation of an Al–Mg–Si aluminium alloy, *Material Science Engineering A*, 2008, 483, pp. 54-58.
- [11] Panigrahi, S.K., Jayaganthan, R. Effect of rolling temperature on microstructure and mechanical properties of 6063 Al alloy, *Material Science and Engineering A*, 2008, 492, pp. 300-305.
- [12] Panigrahi, S.K., Devanand, D., Jayaganthan, R. Effect of ageing on strength and ductility of ultrafine grained al 6061 alloy, *Material Science Forum*, 2010, 633-634, pp. 303-309.
- [13] Majid, V., Ali Karimi, T., Sun I.H., Hyoung S.K. Dynamic ageing and the mechanical response of Al–Mg–Si alloy through equal channel angular pressing, *Materials and Design*, 2010, 31, pp. 4076-4082.
- [14] Eshelby, J.D. The determination of the elastic field of an ellipsoidal inclusion, and related problems, *Proceedings of Royal Society London A*, 1957, 241, pp. 376-96.
- [15] Gang, U.G., Lee, S.H., Nam, W.J., The evolution of microstructure and mechanical properties of a 5052 aluminium alloy by the application of cryogenic rolling and warm rolling, *Material Transactions*, 2009, 50, pp. 82-86.
- [16] Rao, P., Jayaganthan, R. Effects of warm rolling and ageing after cryogenic rolling on mechanical properties and microstructure of Al 6061 alloy, *Materials and Design*, 2012, 39, pp. 226-233

- [17] Panigrahi, S.K., Jayaganthan, R. Effect of annealing on precipitation, microstructural stability, and mechanical properties of cryorolled Al 6063 alloy, *Journal of Material Science*, 2010, 45, pp. 5624-5636.
- [18] Humphreys, F.J., Hatherly, M. *Recrystallization and Related Annealing Phenomena*, Oxford. UK, Pergamon Press, 2004.
- [19] Rangaraju, N., Raghuram, T., Krishna B.V, Rao, K.P., Venugopal P. Effect of cryorolling and annealing on microstructure and properties of commercially pure aluminium, *Material Science Engineering A*, 2005, 398, 246-251.
- [19] Kang, U.G., Lee, H. J., Nam, W.J. The achievement of high strength in an Al 6061 alloy by the application of cryogenic and warm rolling, *Journal of Material Science*, 2012, 47, pp. 7883-7887.