Available online at www.sciencedirect.com

ScienceDirect

Procedia Engineering 75 (2014) 129 – 133

Procedia Engineering

www.elsevier.com/locate/procedia

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

Comparative study of Microstructure and Mechanical properties of Al 6063 alloy Processed by Multi axial forging at 77K and Cryorolling

Maruff Hussain[a],P.Nageswara rao[a], Dharmendra Singh[a], R.Jayaganthan[a,*], Surendra Singh[a]

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

Roorkee 247667, India

Abstract

In the present work, commercially available Al 6063 alloy was subjected to multiaxial forging (MAF) at 77K. The evolution of ultrafine grain structure during MAF at 77k at various strain levels (∑Δε=1.2 and ∑Δε =2.4) was investigated using Optical microscopy and Transmission electron microscopy. Cryorolling was performed up to true strain 2.4 and the mechanical properties and micro structural changes were compared with MAF at 77 K sample of same strain. At lower strain level (∑Δε=1.2), the microstructure reveals formation of dislocation cells along with sub grain structure, predominantly low angle grain boundaries. Formation of elongated sub grain structure with high angle grain boundaries through dynamic recovery was observed with increasing strain up to (∑Δε=2.4) at shear bands. Microstructure observed in cryorolled sample is more homogeneous with low angle grain boundaries. The improvement in mechanical properties observed in both the samples processed through CR and MAF are nearly same.

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

* Corresponding author. Tel.: 91-1332-285869; fax: 91-1332-285243.
E-mail address: rjayafmt@iitr.ernet.in

1877-7058 © 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.
doi:10.1016/j.proeng.2013.11.028

1. Introduction

Ultrafine grained materials (UFG) have gained significant importance from scientific and industrial community due to their superior mechanical and physical properties over its conventional bulk material [1]. Several severe plastic deformation (SPD) techniques are developed to produce bulk UFG material with homogenized equiaxed structure [2]. The commonly used SPD techniques are Equal channel angular pressing (ECAP), Accumulative roll bonding (ARB), High pressure torsion (HPT) and Multi axial forging (MAF). The size and shape of the samples obtained from the each method are quite different. Processing temperature and strain rate employed during the process will influence the grain refinement [3]. UFG structure has been successfully developed in several metals and alloys by deforming at very low temperatures (near or at liquid nitrogen temperature) even with medium strains [3-4]. Very low temperature deformation leads to suppression of dynamic recovery or restoration of dislocations, results accumulation of high dislocation densities in the material [7-8].

Attempts have been made to study the effect of cryorolling on microstructure and mechanical properties of the material, which has been already processed through SPD [8]. In the present investigation, MAF was chosen to produce UFG structure in Al 6063 alloy. MAF is a simple technique where the material is subjected to repeated forging in three orthogonal directions. It is used to produce UFG structure in relatively brittle materials by operating at high temperatures [9]. The advantage of MAF is that, the initial shape remains same with minimum distortion even after several pressings. Till now, studies on effect of MAF at 77K on precipitation hardenable alloys are scarce in literature. The main aim of the present investigation is to study the effect of MAF at liquid nitrogen temperature on microstructure and mechanical properties of Al 6063 alloy.

2. Experimental details

The material used in present investigation is commercially available Al 6063 alloy with chemical composition (wt. %) 0.51% Si, 0.46% Mg, 0.132% Mn, 0.15% Fe, 0.015 % Cu (wt%) and balance Al. Samples with dimensions 27×30.5×33 mm^3 and 12×30×40 mm^3 were cut from the as received material to deform through MAF and rolling, respectively. Rolling and MAF was performed at 77K to investigate effect of deformation temperature on mechanical behaviour and microstructure development in the material processed through different routes. Before subjecting to deformation, samples were homogenized at 510 °C for 1 hour in muffle furnace, then water quenched to room temperature. These samples were noted as ST. The higher dimensions of the samples were cut along initial extruded direction of the billet. The samples were subjected to rolling and MAF at liquid nitrogen temperature for an equivalent true strain 2.4. The rolled samples are noted as CR. For rolling and MAF at liquid nitrogen temperature, samples were dipped in liquid nitrogen for 15 min for initial pass and afterwards 10 min for the successive passes. Rolling was performed through multipass and thickness reduction per pass was 4%. Whereas, in MAF true strain per pass was $\Delta\varepsilon=0.2$. After each pass, the sample was turned to an angle of 90° then the next pass was given. Fig. 1a, b shows the schematic illustrations of the processes followed in the present work. The MAF process diagram shown in Fig. 1a corresponds to one cycle. For microstructural studies, MAF samples were processed up to 2 cycles (equivalent true strain ($\sum\Delta\varepsilon=1.2$)) and 4 cycles (equivalent true strain ($\sum\Delta\varepsilon=2.4$)). These samples were denoted as MAF 2 and MAF 4 for the further reference.

Microstructural characterization was carried out using optical microscopy (Leica DMI 200) and transmission electron microscopy (TEM) (FEI Technai 20). Samples were etched using Poultons reagent. TEM samples were prepared by twin jet polishing of 3 mm disc with mixture of 20% perchloric acid and 80% methanol at - 40 °C temperature, 40 V. Samples for microstructural characterization and tensile testing were prepared along the plane perpendicular to the last forging axis for MAF samples and in plane parallel to rolling direction for CR samples. Details of sample preparation for TEM testing were discussed elsewhere [10]. Vickers hardness testing was performed with load of 5 kg having dwell time of 15 s.Tensile testing was conducted on non-standard sub size specimen of 10 mm gauge length at room temperature on H25K-S universal testing machine.

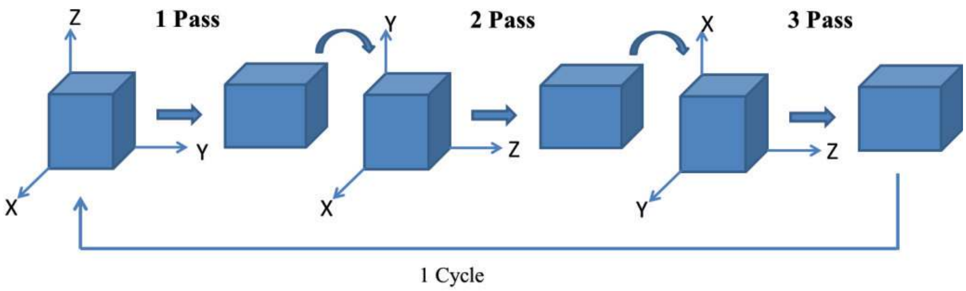

Figure 1.a: Schematic illustration of Multi axial forging for 1 cycle

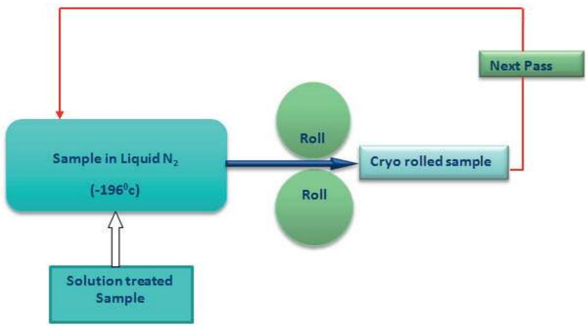

Figure 1.b: Schematic illustration of principle of rolling at liquid nitrogen temperature

3. Results and discussion

3.1 Optical microscopy

Figure 2 shows the changes in optical microstructure of Al 6063 alloy after rolling and multi axial forging at 77K. The initial solution treated (ST) material (Fig.2 (a)) contains elongated band structure formed during extrusion. The black particles aligned along the extrusion direction on the grain boundaries in ST sample are believed to be undissolved second phase of $FeAl_3$, Fe_3SiAl_{12} and Mg_2Si [11]. During solution treatment, Mg and Si dissolved in the matrix and further it gets precipitated by forming Mg_2Si phase during ageing treatment. Figure 2(b) shows a clear distribution of undissloved second phase along the rolling direction in CR sample. Whereas in the sample, MAF at 77 K in Figure 2.c shows irregular distribution of particles, represents the formation of deformation bands in various directions and mutual crossings [12].

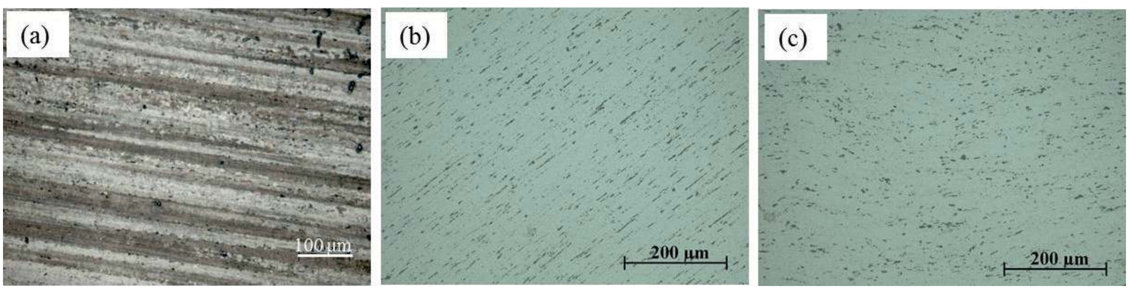

Figure 2: Optical microstructures of Al 6063 alloy; (a) ST, (2) CR (3) MAF-4

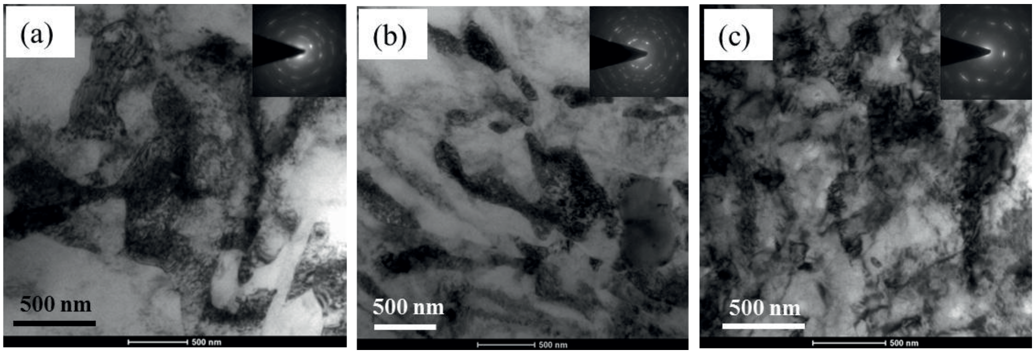

Figure 3: TEM Microstructure of Al 6063 alloy ; (a) MAF-2 (b) MAF- 4 , (c) CR

3.2 TEM observations

It can be seen from Figure 3a that MAF at 77 k after 2 cycles leads to the formation of sub grain structure along with dislocation networks. Selected area electron diffraction (SAED) shown in inset in Fig.2 (a) reveals the microstructure developed after 2 cycles of MAF at 77 K is composed primarily with low angle grain boundaries (grain boundary misorientation $\leq$ 15 degrees). With increasing number of MAF cycles to 4 (equivalent strain 2.4), stretched type substructure was observed. The average thickness of stretched substructure is around 300 nm. Along with the stretched substructure, nearly equiaxed subgrains with average size of 300 nm were also observed. Nakao et.al have observed band substructure in copper deformed through MDF at 77k up to an equivalent strain 2.4 [12]. It was reported that these elongated substructure could be deformation twins. Materials like Al with high stacking fault energy, deforms by dislocation slip rather deformation twin at CR temperature and room temperature deformation [13]. Liu et al. have observed that the formation of large scale of shear bands due to strain localization in AA 3104 aluminium alloy deformed through multiple constrained compression at room temperature. It can be concluded that the stretched substructure observed in the present study is due to strain localization at shear bands. SAED map corresponding to MAF-4 sample shows ringed pattern, which indicates that localized strain at shear bands caused transformation of low angle grain boundaries to high angle grain boundaries. The CR sample shown in Figure 3(c) reveals homogeneous formation of fine sub grain structure along with high densities of dislocations. The dislocation density observed in CR sample is more than MAF-4 sample.

3.3 Mechanical Properties

Mechanical properties were studied through Vickers hardness testing and tensile testing performed at room temperature. Hardness measurements and tensile sample preparation were done on the plane perpendicular to the last forging axis in MDF sample and plane parallel to the rolling direction (RD-TD) in CR sample. The obtained results are listed in Table 1. CR 92% sample has shown significant improvement in hardness (88±2 Hv) compared to ST sample (42±2 Hv). The UTS of CR sample has increased from 106 MPa to 248 MPa, which is nearly 133% increment, whereas YS has increased from 56 MPa to 239 MPa (326% increment).It can be understood that YS has shown more response to the cryorolling. The increment in hardness and strength of CR 92% reduction sample is combined effect of solid solution strengthening, work hardening and Hall-Petch effect [14]. The observed improvement in mechanical properties of CR material is in agreement with the reported literature [14]. The MAF-4 material has shown slightly more hardness (92±4) than CR material.

The YS and UTS and % elongation of MAF-4 are slightly more than CR sample. Higher % elongation observed in MDF-4 cycles sample over CR-92% sample could be due to sample size effect or the formation of subgrains with high angle grain boundaries at the shear bands due to strain localization which can absorb more dislocation during tensile deformation. The variation in hardness observed in MAF -4 cycle sample is more than CR sample, this could be due to different strain path associated with the process. Figure 4 shows the variation in Vickers hardness value on the sectioned surface along the plane perpendicular to last forging axis. The trend observed in improvement of mechanical properties through both the process is nearly same.

Table 1: Mechanical properties of Al 6063 alloy after MDF and cryorolling

State	σ_{YS}	σ_{UTS}	% Elongation	Vickers Hardness
ST	56	106	30	42 ± 2
CR	239	248	4.5	88 ± 2
MDF-4	241	252	6	92 ± 4

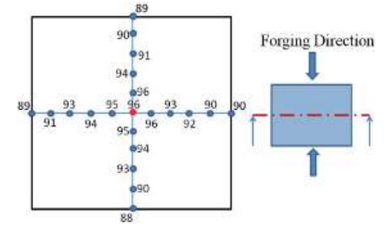

Figure 4: Variation in Vickers hardness value along the centre line of MDF-4 sample

4. Conclusions

1. Al 6063 alloy was processed by two different routes, rolling and multi directional forging at 77K in the present work. Microstructure and mechanical properties were compared.
2. The improvement in mechanical properties observed in Al 6063 alloy processed through different routes is nearly same.
3. Stretched sub grain structure with an average thickness of 300 nm was developed in material processed through multi directional forging at 77k up to an equivalent strain 2.4 due to strain localization at shear bands.

References

[1] Valiev, R.Z. Structure and mechanical properties of ultrafine-grained metals, *Material Science Engineering A,* 1997, 234-236, pp. 59-66.
[2] Zhilyaev, A.P., Langdon, TG. Using high pressure torsion for metal processing: fundamentals and applications, *Progress in Material Science*, 2008, pp. 893-979.
[3] Singh, J.B, Kapoor, R., Prasad, D., Chakravarty, J.K. Comparison of microstructures and strengths of an Al-2.5 Mg alloy subjected to severe plastic deformation at room and liquid nitrogen temperature, *Material science and Engineering A,* 2013, 581, pp. 25-30.
[4] Singh, D., Rao, P.N., Jayaganthan, R. Effect of deformation temperature on mechanical properties of ultrafine grained Al-Mg alloys processed by rolling, *Materials and Design,* 2013, 50, pp. 646-655.
[5] Rao, P.N., Singh, D., Jayaganthan, R. Effect of annealing on microstructure and mechanical properties of Al 6061 alloy processed by cryorolling*, Material Science and Technology,* 2013, 29, pp. 76-82.
[6] Shanmugasundaram T., Murty T.S., Sarma V.S. Development of ultrafine grained high strength Al–Cu alloy by cryorolling, *Scripta Materialia*, 2006, 54, 2013–2017.
[7] Wang, Y.M., Ma, E. Three strategies to achieve uniform tensile deformation in a nanostructured metal, *Acta Materialia*, 2004, 52, 1699-1709.
[8] Lee, Y.B., Shin, D.H., Park, K.T., Nam,W.J., Effect of annealing temperature on microstructure and mechanical properties of a 5083 Al alloy deformed at cryogenic temperature, *Scripta materialia,* 2004, 51, pp. 355-359.
[9] Padap, A.K, Chaudhari, G.P., Nath, S.K., Pancholi, V. Ultrafine- grained steel fabricated using warm multiaxial forging: Microstructure and mechanical properties, *Materials and Design*, 2009, 527, 109-117.
[10] Panigrahi SK, Jayaganthan R. Effect of rolling temperature on microstructure and mechanical properties of 6063 Al alloy. *Mater Sci Eng A,* 2008, 492,300–305.
[11] Das, G., Das, M., Ghosh, S., Dubey, P., Ray, A.K. Effect of aging on mechanical properties of 6063 Al-alloy using instrumented ball indentation technique, *Materials Science and Engineering A,* 2010, 527, pp. 1590–1594.
[12] Nakao, Y., Miura, H., Sakai, T. Microstructural evolution and recrystallization behaviour in copper multi directionally forged at 77 K, *Advanced Material Research*, 2007, 15-17, pp. 649-654.
[13] Sarma, V.S., Wang, J., Jian, W.W., Kauffmann A., Conrad, H., Freudenberger, J., Zhu Y.T. Role of stacking fault energy in strengthening due to cryo-deformation of FCC metals, *Materials Science and Engineering A*, 2010, 527, pp. 7624-7630.
[14] Panigrahi, S.K., Jayaganthan R. Influence of solutes and second phase particles on work hardening behaviour of Al 6063 alloy processed by cryorolling*, Materials Science and Engineering A,* 2011, 528, pp. 3147-3160.