# Influence of Rolling Temperature on Microstructure and Mechanical Properties of Cryorolled Al-Mg-Si alloy

B. Gopi, N. Naga Krishna, K. Venkateswarlu, K. Sivaprasad

**Abstract**—An effect of rolling temperature on the mechanical properties and microstructural evolution of an Al-Mg-Si alloy was studied. The material was rolled up to a true strain of ~0.7 at three different temperatures *viz;* room temperature, liquid propanol and liquid nitrogen. The liquid nitrogen rolled sample exhibited superior properties with a yield and tensile strength of 332 MPa and 364 MPa, respectively, with a reasonably good ductility of ~9%. The liquid nitrogen rolled sample showed around 54 MPa increase in tensile strength without much reduction in the ductility as compared to the as received T6 condition alloy. The microstructural details revealed equiaxed grains in the annealed and solutionized sample and elongated grains in the rolled samples. In addition, the cryorolled samples exhibited fine grain structure compared to the room temperature rolled samples.

*Keywords*—Al-Mg-Si alloy, Cryorolling, Tensile properties, Ultra fine grainstructure

## I. INTRODUCTION

T HE Al-Mg-Si alloys, owing to their good combination of corrosion resistance, formability and high strength are widely used in automotive and aerospace applications for fabricating high strength and lightweight structures [1-3]. Ultrafine grain (UFG) structured materials can be achieved by severe plastic deformation (SPD) techniques [4]. However, majority of the SPD techniques require large plastic deformation and complicated procedures along with restricted geometries. The process of rolling overcomes the difficulty in producing sheet and lengthy products. Cryorolling (CR) is a unique mechanical deformation process at cryogenic temperatures by which high strength and ductility combinations can be achieved [5].

These combinations of properties were reported to be attributed to a bimodal grain size distribution with micrometer-sized grains embedded inside the matrix of nano/UFG microstructure. The UFG imparts high strength as expected from the Hall–Petch relationship, whereas the micrometer-sized grains contribute to the enhancement of ductility [6]. Many reports are available on cryorolling of pure metals, their alloys and even composites [7]-[13]. The suppression of dynamic recovery during deformation at cryogenic temperatures preserves a high density of defects generated by deformation, which can act as the potential recrystallization sites. Accordingly, the cryogenic deformation requires less plastic deformation for achieving UFG structure, compared to the SPD processes [5].

### II. EXPERIMENTAL

Al-Mg-Si alloy (AA6061) was procured in T6 condition in the form of one-inch diamter rod. The chemical composition of the as received alloy is having 0.48% Si, 0.86% Mg, 0.46% Fe, 0.9% Mn, 0.04% Cr, 0.005% Zn, 0.01% Ti and balance Al. The alloy was initially melted in an induction furnace at 950°C and after specified time, the molten alloy was cast into rectangular sheet of 130 mm  $\times$  130 mm  $\times$  5 mm. The cast alloy sheets were initially annealed at 415°C for 2.5 h and then solutionized at 530°C for 1 h followed by quenching in chilled water. Solutionized samples were rolled up to a total thickness reduction of 50% with a true strain of 0.69. Rolling was performed at three temperatures viz., room temperature (RT at 305 K), liquid propanal (LP at 193 K) temperature and at liquid nitrogen (LN at 77K) temperature. During rolling, the samples were initially maintained in respective media for 30 min duration and the total thickness reduction of 50% was achieved in multiple passes with about 1% reduction per pass. After each pass, the sheets were again maintained in the respective medium for 2 min. before further reduction. X-ray diffraction (XRD) analysis was carried out to identify the different phases of the samples rolled at different temperatures. The Bruker D8 advance instrument using Cu-Kα radiation was used for XRD analysis. Tensile tests were performed using an Instron tensile test machine, operated with a cross-head speed of 0.5 mm/min on the samples that were machined as per ASTM E8 sub size specifications with the gauge length of 25 mm. Fractograph analysis of the tensile samples rolled at different temperatures was performed using a scanning electron microscope (SEM) Hitachi (S3000H model), Japan.

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# III. RESULTS AND DISCUSSION

# A. Microstructure of Alloy during CR and RT Rolling

The light optical micrographs of the annealed and solutionized Al-Mg-Si alloy (Fig. 1(a)) indicated the presence of equiaxed grains and the sample rolled at RT (Fig. 1(b)) exhibited coarse elongated grains (arrow indicating the rolling direction), which is attributed to the parallel adiabatic heating during the process. Among the cryorolled samples, LN rolled sample exhibited finer grain structure than the LP rolled

samples. The extent of fineness in grain structure was improved with decreasing the rolling temperature. The presence of finer grains in the case of cryorolled samples is attributed to the suppression of dynamic recovery during deformation at cryogenic temperatures [5]. The XRD pattern of the alloy rolled at the three temperatures was shown in Fig. 2.

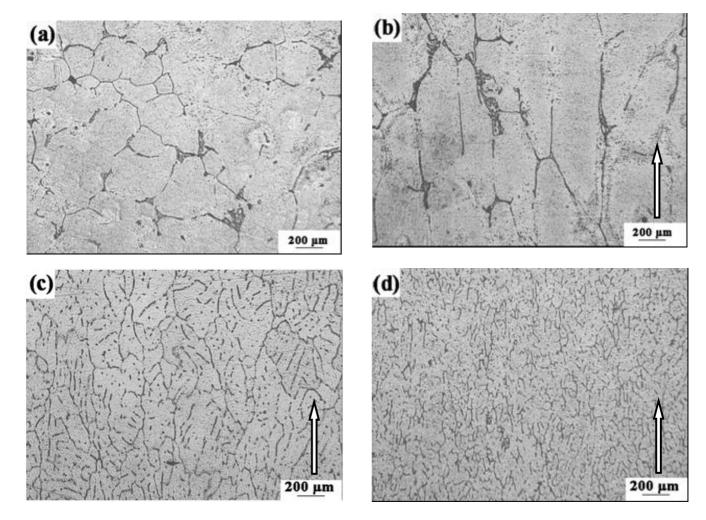


Fig. 1 Light optical micrographs of the alloy in different conditions; (a) full annealed and solutionized; (b) rolled at room temperature; (c) rolled at liquid propanal temperature and (d) rolled at liquid nitrogen temperature

# B. Tensile Properties

The engineering stress-strain curves for the rolled samples are shown in fig. 3. The RT rolled sample exhibited a yield and tensile strength of ~280 MPa and 338 MPa, respectively with a ductility of ~8%. Similarly, the LN rolled sample exhibited ~19% and 8% increase in yield and tensile strength and ~ 11% improvement in ductility as compared to the RT rolled sample.

However, the sample rolled at LP temperature exhibited inferior properties when compared to the RT and LP conditions. This may be attributed to the stored energy of the rolled material both at RT and LP.

Niranjani *et al.* [14] also made similar observations. They considered a modified version of AA 6061 alloy and rolled at room temperature and at liquid nitrogen temperature to  $\sim 80\%$  reduction and performed tensile tests, which revealed similar level of yield strengths and low ductility at both the temperatures. The reported amount of stored energy owing to deformation was found to be independent of rolling temperature [14].

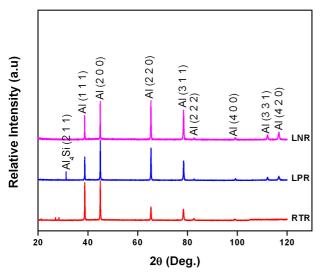


Fig. 2 XRD pattern of AA6061 alloy in different processing condition

However, in the present investigation, the tensile strength in the LN rolled sample is ~364 MPa and ductility ~9%. The lack of improvement in the work of Niranjani *et al*, can be due to the higher amounts of reductions in their work which led to partial dynamic recrystallization. When compared to the wrought alloy in T6 condition [15], an increase of 54 MPa (17%) in tensile strength is achieved, without much loss of ductility. This increase is due to the high dislocation density increase in LN rolled condition with more amount of stored energy than LP and RT rolled samples.

In contrast to the earlier explanation, an improvement of  $\sim$ 11% increase in tensile strength of LN rolled condition with a simultaneous reduction in ductility compared to the RT rolled alloy was also reported [16]. They have reported that, to improve ductility, the CR samples were subjected to short annealing treatment. In contrast, the LN rolled sample in the

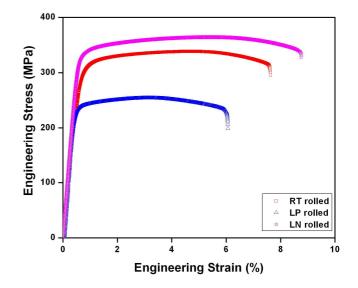


Fig. 3 Engineering stress-strain curves of alloy rolled at different temperatures

present work exhibited improvement with respect to both strength and ductility compared to the RT rolled sample. Hence, based on present work, further improvement in ductility without losing srength is expected up on short annealing and ageing treatment.

The intensity of Al (2 0 0) plane that has been normalized is compared at the three temperatures and is indicted in fig. 4. The peak intensity is almost similar for the case of RT, LP rolled sample, and for the LN rolled sample, a drastic reduction in the intensity was observed, which indicates higher strain in the material. In addition, as the rolling temperature decreases, a shift in the peak is observed, particularly in the case of LN rolled sample. The better properties exhibited by the LN rolled sample is attributed to the enhanced strain in the material compared to RT and LP conditions.

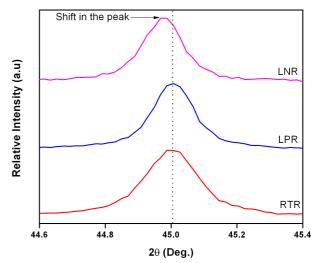
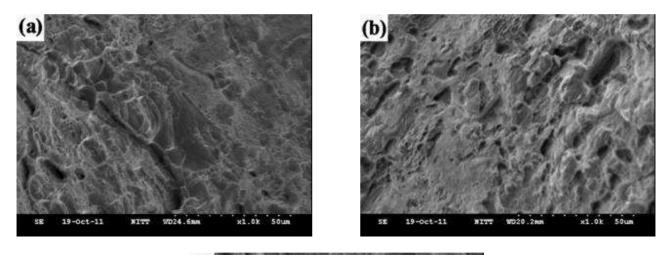


Fig. 4 Peak shift of Al (200) with reference to rolling temperature



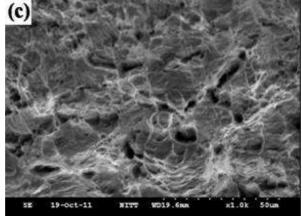


Fig. 5 Fracture surface morphologies of tensile samples of the alloy rolled at - (a) RT, (b) LP and (c) LN

# C. Fracture Morphologies

The presence of dimples in the case of alloy rolled at RT and at LN temperature indicates a ductile type of fracture and the behaviour of the alloy at LP temperature is in contrast with the other temperatures. It shows that, the sample fractured in more brittle manner, with cleavage planes on the entire surface with very less number of dimples in the fractograph.The inferior strength and poor ductility exhibited by the LP rolled samples is attributed to this brittle fracture. Panigrahi *et al.*, reported that [16], with increasing percentage of thickness reduction in the samples, the dimple size decreases which is due to the grain refinement and work hardening associated with the SPD of the samples. As LN rolled sample exhibited relatively finer and deep dimples than that of RT rolled samples, it exhibited better strength and ductility than the other two conditions.

## IV. CONCLUSION

Microstructural studies revealed a finer grain structures in LN rolled condition than the other conditions. The LN rolled sample showed ~19% and ~8% increase in yield and tensile strength and ~11% improvement in ductility compared to RT rolled sample. The improved mechanical properties of the LN rolled sample is attributed to the combined effect of grain

refinement, suppression of dynamic recovery and accumulation of higher dislocation density during cryorolling. It is anticipated that the strength can be retained and ductility can be significantly enhanced by further short annealing and aging the LN rolled sample.

#### ACKNOWLEDGMENT

K. Sivaprasad wishes to thank the Department of Science and Technology (DST), New Delhi, government of India for their financial support in carrying out this research work under Fast Track Scheme (DST Sanction No. SR/FT/ET-005/2008). 305, 2008.

#### REFERENCES

- A.K. Gupta, D.J. Lloyd and S.A. Court, "Precipitation hardening processes in an Al–0.4%Mg–1.3%Si–0.25%Fe aluminum alloy," Mater. Sci. Eng., A, vol. 301, pp. 140-146, 2001.
- [2] D.A. Chakrabarti and D.E. Laughlin, "Phase relations and precipitation in Al-Mg-Si alloys with Cu additions," Prog. Mater Sci., vol. 49, pp. 389-410, 2004.
- [3] S. Esmaeili, X. Wang, D.J. Lloyd and W.J. Poole, "On the precipitationhardening behavior of the Al-Mg-Si-Cu alloy AA6111," Metall. Mater. Trans. A, vol. 34, pp. 751-763, 2003.
- [4] R.Z. Valiev, R.K. Islamgaliev and I.V. Alexandrov, "Bulk nanostructured materials from severe plastic deformation" Prog. Mater Sci., vol. 45, pp.103–189, 2000.

- [5] Y. Wang, M. Chen, F. Zhou and E Ma, "High tensile ductility in a nanostructured metal," Nature, vol. 419, pp. 912-915, October 2002.
- [6] J.G. Sevillano and J. Aldazabal, "Ductilization of nanocrystalline materials for structural applications," Scr. Mater., vol. 51, pp. 795-800, 2004.
- [7] N. Rangaraju, T. Raghuram, B. V. Krishna, K. P. Rao and P. Venugopal, "Effect of cryo-rolling and annealing on microstructure and properties of commercially pure aluminium," Mater. Sci. Eng. A., vol. 398, pp. 246-251, 2005.
- [8] T. Shanmugasundaram, B.S. Murty and V. S. Sarma, "Development of ultrafine grained high strength Al–Cu alloy by cryorolling," Scr. Mater., vol. 54, pp.2013-2017, 2006.
- [9] S. K. Panigrahi, R. Jayaganthan and V. Chawla, "Effect of cryorolling on microstructure of Al-Mg-Si alloy," Mater. Lett., vol. 62, pp. 2626-2629, 2008.
- [10] S.K. Panigrahi and R. Jayaganthan, "Effect of rolling temperature on microstructure and mechanical properties of 6063 Al alloy," Mater. Sci. Eng. A., vol. 492, pp. 300-305, 2008.
- [11] K.G. Krishna, N. Singh, K. Venkateswarlu and K.C.H. Kumar, "Tensile behavior of ultrafine-grained Al-4Zn-2Mg alloy produced by Cryorolling," J. Mater. Engg. Perform., DOI: 10.1007/s11665-011-9843-1.
- [12] N. Naga Krishna, A.K. Akash, K. Sivaprasad and R. Narayanasamy, "Studies on void coalescence analysis of nanocrystalline cryorolled commercially pure aluminium," Mater. Des, vol. 31, pp. 3578-3584, 2010.
- [13] N. Naga Krishna and K. Sivaprasad, "High temperature tensile properties of cryorolled Al-4wt%Cu-3wt%TiB2 in-situ composites," T Indian I Metals, vol. 64, pp. 63-66, February – April 2011.
- [14] V.L. Niranjani, K.C.H. Kumar and V.S. Sarma, "Development of high strength Al-Mg-Si AA6061 alloy through cold rolling and ageing," Mater. Sci. Eng. A., vol. 515, pp.169-174, 2009.
- [15] ASM Metals Reference Book, 3rd edition, ASM International, Materials Park, p. 403, 2004.
- [16] S.K. Panigrahi and R. Jayaganthan, "A study on the mechanical properties of cryorolled Al-Mg-Si alloy," Mater. Sci. Eng. A., vol. 480, pp. 299-