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# A study on the mechanical properties of cryorolled Al–Mg–Si alloy

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**Abstract**

The mechanical properties of a precipitation hardenable Al–Mg–Si alloy subjected to cryorolling and ageing treatments are reported in this present work. The severe strain induced during cryorolling of Al–Mg–Si alloys in the solid solutionised state produces ultrafine microstructures with improved mechanical properties such as strength and hardness. The improved strength and hardness of cryorolled alloys are due to the grain size effect and higher dislocation density. The ageing treatment of cryorolled Al–Mg–Si alloys has improved its strength and ductility significantly due to the precipitation hardening and grain coarsening mechanisms, respectively. The reduction in dimple size of cryorolled Al–Mg–Si alloy upon failure confirms the grain refinement and strain hardening mechanism operating in the severely deformed samples.


*Keywords:* Al–Mg–Si alloy; Cryorolling; Ageing; Precipitation hardening; Tensile properties

## 1. Introduction

In recent years, the production of ultrafine grained and nanostructured bulk materials from their bulk materials through severe plastic deformation (SPD) processes such as equal channel angular pressing (ECAP), multiple compression, and severe torsional straining has been given lot of focus to realize its potential for structural and functional applications [1–4]. The formation of ultrafine grain structures by SPD methods provides very large deformations at relatively low temperatures under high pressures. However, majority of these methods require large plastic deformations with strains much larger than unity and the amount of materials produced is very limited [5]. Recently, cryorolling has been identified as one of the potential routes to produce nanostructured/ultrafine grained pure metals Cu, Al, Ni [6–8] and Al alloys [9,10] from its bulk counterpart by deforming them at cryogenic temperature. It is well known in the literature that the rolling of pure metals and alloys in cryogenic temperature suppresses dynamic recovery and the density of accumulated dislocations reaches a higher steady state level as compared to room temperature rolling. These higher density of dislocations act as driving force for the initiation of large number of nucleation sites during annealing, resulting in sub-microcrystalline or ultrafine grain structures (ufg) [6–8].

Wang et al. [6] have produced the ufg pure copper from its bulk material by cryorolling treatment and investigated the effect of heat treatment on its microstructures and mechanical properties. They observed a formation of bimodal structures consisting of ultrafine grains and coarse grains during low temperature annealing, contributing to the excellent combination of high strength and ductility. The formation of ultrafine grain microstructures in Al, Ni, and 5083 Al alloys with higher strength and low ductility has been reported in the literature [7–9]. Shanmugasundaram et al. [10] have investigated the mechanical behavior of 2219 Al alloy subjected to cryorolling and subsequently annealing and ageing treatment. An improved yield strength and ultimate tensile strength of 485 MPa and 540 MPa, respectively were observed due to the formation of ultrafine grain microstructures in the alloy.

An aluminum alloy (6063) is extensively used in structural applications due to its good mechanical properties, higher corrosion resistance, better weldability and lower cost compared to 2XXX and 7XXX Al alloys [11]. The formation of $Mg_2Si$ intermetallic compound in Al 6063 alloy has beneficial effect on improving its mechanical and corrosion resistance properties. Since it is a heat treatable alloy, it can be strengthened appreciably during ageing treatment. A combination of equal channel angular pressing and low temperature ageing of precipitation hardenable Al alloys has resulted in significant improvement in their mechanical properties as reported in the literature [12,13].

Kim et al. [12] studied the effect of ECAP combined with post-ECAP ageing treatment on 6061 Al alloy and observed

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an enhancement of its room-temperature strength. However, an application of cryorolling to the precipitation hardenable Al alloys of 6000 series has received only limited attention. An optimization of strength and ductility of Al alloys (6000 series) processed by cryorolling is very essential for its potential use in structural applications. Therefore, the present investigation has been focused to investigate the mechanical properties of precipitation hardenable commercial Al–Mg–Si alloy subjected to cryorolling followed by ageing treatment. The formation of precipitates and microstructures of the alloys were characterized by X-ray diffraction and field emission gun scanning electron microscopy (FE-SEM), respectively.

## 2. Experimental

The thick plate (9.6 mm) of T4 temper treated Al–Mg–Si alloy, with 0.45% Si, 0.3% Mg, 0.015% Cu, 0.013% Mn, 0.058% Fe, 0.022% Zn, 0.02% Cr (wt%) was procured from the Hindalco Industries Ltd., Aditya Birla Group, Renukoot, India for the present work. These plates were solutionised at 520 °C for 45 min and then quenched in water. The three different thickness reductions of 55%, 70% and 90% on annealed Al–Mg–Si alloy plates were achieved by cryorolling. The samples were dipped in liquid nitrogen for 30 min prior to each roll pass during the rolling process. The diameter of the rolls was 110 mm and the rolling speed was 8 rpm. The rolling reduction per pass was 5% but many passes were given to achieve the required reduction of the samples. In order to improve the mechanical properties, the cryorolled (CR) and the solution treated samples (ST) before cryorolling were subjected to artificially ageing at 100 °C, 150 °C and 175 °C, respectively.

Microhardness and tensile tests were carried out to evaluate the strength and ductility of the Al–Mg–Si alloys cryorolled to various strains, annealing and ageing treatment. Vickers microhardness ($H_V$) was measured on the plane parallel to longitudinal axis (rolling direction) by applying a load of 100 g for 15 s. Prior to each $H_V$ measurement, the surface of the specimen was polished mechanically using emery paper and diamond paste of 0.25 μm to remove the surface reactions that may occur during the heat treatment. An average of at least 10 readings on the surface of the specimen were taken to obtain microhardness value. The tensile specimens were machined as per ASTM E-8 sub-size specifications parallel to the rolling direction with gauge lengths of 25 mm. The tensile test was conducted after polishing the samples in air at room temperature using a S-series, H25K-S materials testing machine operated at a constant crosshead speed with an initial strain rate of $5 \times 10^{-4}\,s^{-1}$. For tensile test, the samples with different percentage of thickness reduction after cryorolling were machined to the same length and without changing the thickness.

The microstructure of the ST (starting material solution treated and quenched) and CR (cryorolled) sheets were examined in a field emission gun scanning electron microscope (FEI, Quanta 200F) using EBSD analysis. The samples for electron back scattered diffraction (EBSD) characterization were prepared by mechanical polishing using 1000 grit emery paper, fine polishing to mirror finish with diamond paste, and then finally electro polished at −15 °C using an electrolyte of methanol:perchloric acid (80:20) at 11 V dc. The EBSD measurements were performed at the center of the samples with a scan area of 600 μm × 525 μm and 300 μm × 200 μm for ST and CR samples, respectively. The step size of 1.5 μm for ST samples and 0.5 μm for CR samples was used during the measurements. TSL OIM analysis 4.6 software developed by TEXSEM Laboratories Inc. is used to analyze the EBSD maps. The average confidence index was found to be 0.73 and 0.51 for ST and CR samples, respectively. X-ray diffraction (XRD) analysis was carried by Bruker AXS D8 Advance instrument using Cu Kα radiation for identifying the precipitates present in the original and cryorolled samples.

## 3. Results and discussion

### 3.1. Microstructures of Al–Mg–Si alloy before and after cryorolling

An electron backscattered diffraction (EBSD) maps of the starting material (ST) after annealing and the cryorolled (CR) material after 90% reduction are shown in Fig. 1. The microstructure of the ST material, annealed at 520 °C for 45 min exhibits equiaxed grains with an average grain size of 60 ± 10 μm as shown in Fig. 1a. The cryorolled samples with 90% reduction show severely deformed grains, which are elongated along the rolling direction as observed in Fig. 1b. The parallel bands of elongated substructures containing high dislocation density are evident from this figure.

### 3.2. Tensile and hardness properties

The hardness values of the solution treated alloys rolled at cryogenic temperature and room temperature (RT), respectively as a function of true strain are shown in Fig. 2a. The true strain corresponds to different percentage of thickness reduction in the samples. The hardness of the cryorolled samples has increased from 58 to 85.5 $H_V$ (nearly 47% increase) after 55% thickness reduction ($e$ = 0.79). Subsequent thickness reductions of the samples increased its hardness further and after 90% thickness reduction ($e$ = 2.262), it has increased about 75%. An enhancement of hardness in the cryorolled Al–Mg–Si alloys could be directly attributed to the higher dislocation density and the formation of submicrocrystalline structures, which occurs during severe plastic deformation. It is observed that the hardness of cryorolled alloys is higher than that of RT rolled materials at different strains. It can be explained based on the mechanism that dynamic recovery was effectively suppressed during cryorolling leading to a higher dislocation density as known in the earlier literature [6,14,15]. The ultrafine-grained microstructures formed during cryorolling of Al–Mg–Si alloy obeys the Hall-Petch effect [16,17] to substantiate the improved hardness observed in the present work. With the formation of ultrafine grains in the cryorolled alloys, hardness would increase due to the restricted mobility of dislocation imposed by higher dislocation density, low angle grain boundaries, and misorientation of the grains.