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# Role of stacking fault energy in strengthening due to cryo-deformation of FCC metals

V. Subramanya Sarma<sup>a,b,\*</sup>, J. Wang<sup>b,c</sup>, W.W. Jian<sup>b</sup>, A. Kauffmann<sup>d</sup>, H. Conrad<sup>b</sup>, J. Freudenberger<sup>d</sup>, Y.T. Zhu<sup>b</sup>

- <sup>a</sup> Department of Metallurgical and Materials Engineering, Indian Institute of Technology Madras, Chennai 600 036, India
- <sup>b</sup> Department of Materials Science and Engineering, North Carolina State University, Raleigh, NC 27695, USA
- c School of Mechanical and Automotive Engineering, South China University of Technology, Guangzhou 510640, China
- <sup>d</sup> IFW Dresden, Institute for Metallic Materials, P.O. Box 270116, 01171 Dresden, Germany

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#### ABSTRACT

The effectiveness of the cryogenic (CT) rolling vis-à-vis room temperature (RT) rolling on strengthening is significantly affected by stacking fault energy (SFE) and there is an optimum SFE at which CT rolling is most effective. Studies on Al, Al alloy AA6061, Cu, Cu-4.6Al, Cu-9Al and Cu-15Al (in at.%) alloys revealed that in metals with very high and very low SFEs, the strength difference between CT and RT rolled samples is <10%. The Cu-4.6Al alloy with an intermediate SFE revealed maximum enhancement of strength (25–30%). These results are explained by changes in deformation mechanisms with SFE and temperature. High SFE metals deformed by dislocation slip and low SFE metals deformed by twinning during CT and RT rolling. Metals with intermediate SFEs deformed by twinning during CT rolling but by dislocation slip during RT rolling, and this makes the CT rolling most effective over RT rolling in enhancing the strength.

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#### 1. Introduction

In recent years there is a considerable scientific/technological interest in bulk ultrafine grained (UFG)/nanostructured (NS) materials due to their superior mechanical properties [1-3]. Bulk UFG/NS materials are usually produced by severe plastic deformation (SPD) processes such as equal channel angular pressing (ECAP), high pressure torsion (HPT), accumulative roll bonding (ARB) and severe cold rolling [1-18]. Severe cold rolling processes are particularly attractive for commercial applications because: (a) there is no necessity for special equipment/tooling and, (b) it is easy to scale up for industrial production. Recently, it was reported that deformation (rolling) at cryogenic (liquid N2) temperature resulted in enhanced grain refinement and associated strengthening in Cu when compared to rolling at room temperature [7]. Decreasing the deformation temperature results in suppression of dynamic recovery (as recovery processes are thermally activated) resulting in higher defect density and consequently higher strength [8]. Cryogenic rolling has since been applied by many researchers for

E-mail addresses: vsarma.iitm@gmail.com, vsarma@iitm.ac.in (V.S. Sarma).

producing UFG Al, Ni, Cu and their alloys with the aim of increasing strength [9–18].

It is well known that stacking fault energy (SFE) is also an important material parameter that could affect grain refinement during SPD processing, as SFE determines the probability of cross slip, which along with dislocation climb are possible mechanisms of dynamic recovery [8,19]. In alloys with high SFE (e.g.: Al and Al alloys), it appears that dynamic recovery is not significantly suppressed by cryogenic deformation. This is evident from the reported results on cryorolled Ni, Al and Al alloys, which show that the strength improvement due to cryogenic rolling (CT) vis-à-vis room temperature (RT) rolling is  $\leq 10\%$  [9-11,14,16,18]. On the other hand, in medium and low SFE alloys (Cu. Cu alloys) the strength enhancement appears to be significant [7,15,20]. This seems to indicate that reducing the SFE results in enhanced strengthening following deformation at cryogenic temperature. It is noted in all the above studies, the material is deformed at cryogenic (liquid N<sub>2</sub>) temperatures but the strengths were determined at room temperature. It is well established that lowering the SFE and deformation temperature promotes deformation twinning over dislocation slip [21–24]. The dislocation slip-twinning transition temperature also increases with decreasing SFE and increasing strain rate [25]. In addition, it was reported that while dislocation motion (slip) is highly sensitive to deformation temperature and strain rate, twinning has much lower sensitivity to the above parameters [25].

<sup>\*</sup> Corresponding author at: Department of Metallurgical and Materials Engineering, Indian Institute of Technology Madras, Chennai 600 036, India. Fax: +91 44 22574752.

The question therefore is, what is the role of SFE on the extent of enhancement of strength due to the lowering of deformation temperature? In other words, is there an optimum SFE as was observed in the case of ductility [26] or is the enhancement in strength continuously increases with lowering of SFE? This issue is important not only scientifically but also technologically and economically because there are additional processing costs for implementing the cryogenic deformation processing in industry. The extra cost is justifiable only if the property enhancement is significant as compared with the properties achieved by room temperature SPD processing. In the present work, we have investigated the above issue through a systematic study of microstructure and mechanical properties on high SFE (Al and Al alloy), medium SFE (pure Cu) and low SFE (Cu–Al alloys) alloys subjected to severe cold rolling at room and cryogenic (liquid  $N_2$ ) temperatures.

#### 2. Experimental details

Commercially pure Al (in annealed condition), Al-Mg-Si alloy (AA6061 in T4 condition) and high purity Cu (in annealed condition) were procured in the form of sheets of 2-3 mm thickness. Cu-Al alloys, i.e., Cu-4.6Al, Cu-9Al and Cu-15Al (in at.%) were melted in an induction furnace under argon atmosphere and cast into  $15 \,\mathrm{mm} \times 15 \,\mathrm{mm} \times 150 \,\mathrm{mm}$  bars. For this purpose elements of a purity of at least 99.9% were used. The bars were homogenised at 800 °C for 18 h in argon atmosphere. The 6061 alloy was solutionised at 530 °C for 1 h and water guenched. The homogenised/solutionised samples were rolled at room temperature and at liquid N<sub>2</sub> temperature in a laboratory rolling mill to a strain of  $\sim$ 2 in multiple passes ( $\sim$ 5–10% thickness reduction per pass). Samples were taken at different strains for study of microstructure and mechanical properties. For CT rolling, the samples were cooled with liquid N<sub>2</sub> between the individual passes. Resistivity was measured at ambient temperature using a Hocking AutoSigma 2000 conductivity meter [27]. Samples for optical microscopy were polished and etched with 12.5 g FeCl<sub>3</sub> + 12.5 ml HCl+50 ml H<sub>2</sub>O solution for Cu and Cu alloys and Kellers agent (5 ml HNO<sub>3</sub> + 3 ml HCl + 2 ml HF + 190 ml distilled water) was used for Al and AA6061. Microhardness measurements were performed on a Buehler microhardness tester at a load of 100 g for 10 s. Tensile tests were carried out on small tensile samples (gauge length of 2 mm, width of 1 mm and thickness of 0.3-0.5 mm) using a non-commercial tensile testing machine at an initial strain rate of  $\sim 2 \times 10^{-3} \, \text{s}^{-1}$ . Hardness and resistivity measurements were carried out at room temperature, i.e., measurements on the CT rolled samples were taken after they reached room temperature.

#### 3. Results

#### 3.1. Microstructure

The microstructural details (grain size/twin density) of the starting alloys prior to cold rolling are given in Table 1. The grain sizes of the Cu–Al alloys and AA6061 are in the range of  $\sim\!40{-}50\,\mu m$  (neglecting twin boundaries). For pure Cu and Al the grain size is about 100  $\mu m$  (Table 1). In the Cu–Al alloys the twin density increases and the grain size (accounting twin boundaries) decreases with increasing Al content (Table 1). It is well known that apart from SFE, the starting grain size has also considerable influence on the deformation response (especially twinning [23–25]) of materials during severe plastic deformation. Larger grain size has been shown to promote twinning in low SFE metals and alloys [23]. Apart from the pure elements, the grain sizes of Cu–Al alloys are similar and therefore the deformation response of these materials could be attributed to SFE only.

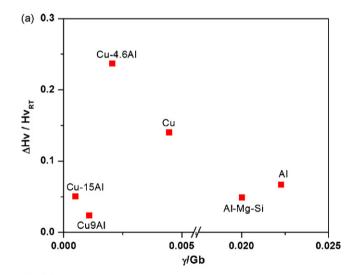
**Table 1**Microstructural details of Al, AA6061, Cu, Cu–4.6Al, Cu–9Al, and Cu–15Al in at.% alloys.

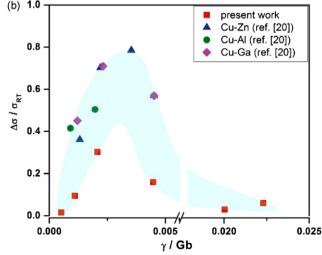
Material	Grain size (excluding twins) (µm)	Grain size (including twins) (µm)	Twin density $(\times 10^6  m^{-2})$
Cu Cu-4.6 at.% Al Cu-9 at.% Al Cu-15 at.% Al Al (commercial pure) AA 6061	$130 \pm 22$ $46 \pm 8$ $46 \pm 10$ $42 \pm 6$ >100 $34 \pm 4$	$106 \pm 18$ $32 \pm 8$ $24 \pm 6$ $21 \pm 5$ NA	11.3 ± 1.8 282 ± 14 429 ± 6 672 ± 14 NA NA

NA: not applicable.

#### 3.2. Hardness and strength changes due to RT and CT rolling

Fig. 1a and b shows the variation of  $\Delta Hv/Hv_{RT}$  and  $\Delta \sigma/\sigma_{RT}$  (where  $\Delta Hv = Hv_{CT} - Hv_{RT}$  and  $\Delta \sigma = \sigma_{CT} - \sigma_{RT}$ ) with normalised SFE ( $\gamma/Gb$ ) at a rolling strain of  $\sim$ 1.5. The  $\gamma$ , G and b values for the different alloys are given in Table 2. The values of  $\gamma$  reported for Cu range from 40 to 78 mJ/m² [8,20,28]. In Fig. 1b, data corresponding to Cu–Zn, Cu–Ga and Cu–Al alloys were also included (data from Ref. [20]). However, these Cu–Zn, Cu–Ga and Cu–Al alloys were processed differently, i.e., the deformation at room temperature was carried out by rotary swaging and cryogenic deformation by pro-





**Fig. 1.** Plot of variation of normalised (a) hardness difference, and (b) tensile strength difference between CT and RT rolling vs. normalised stacking fault energy at rolling strain ∼ 1.5. Data for Cu–Zn, Cu–Al and Cu–Ga alloys are from Ref. [20].

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