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Evolution of annealing texture in cryo-rolled copper

G. Anand [a,*], K. Barai [b], R. Madhavan [c], P.P. Chattopadhyay [d]

[a] Department of Materials Science and Engineering, University of Sheffield, Sheffield S1 3JD, UK
[b] SABIC Technology Centre, Al Jubail 35723, Saudi Arabia
[c] Department of Materials Engineering, Indian Institute of Science, Bangalore 560012, India
[d] Department of Metallurgy and Materials Engineering, Indian Institute of Engineering Science and Technology, Shibpur, Howrah 711103, India

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ABSTRACT

The present study addresses the evolution of texture and microstructure during annealing in a cryo-rolled copper. Transition from copper to brass texture during the cryo-rolling has been illustrated. Twinning and interaction between twins and shear bands have been found to play the important role in grain refinement and strengthening. The low temperature vacancy clustering and its effect on the recrystallization have been experimentally demonstrated. Fine scale twinning, and grain refinement have been attributed to the higher yield strength found in the case of samples subjected to cryo-rolling.

1. Introduction

Cryo-rolling has been identified as the potential material processing technique for developing ultrafine grain structure [1]. In plastic deformation processes, the refinement of grain size is limited by the occurrence of dynamic recovery even at room temperature. In the view of this, the cryogenic plastic deformation process has been adopted to attain finer grain size and to stabilize the deformed microstructure. The heterogeneous grain-size distribution in rolled materials is likely to possess a good strength–ductility combination. Apart from grain size distribution, crystallographic texture is also likely to affect the mechanical properties of a material. Therefore, it may be of interest to understand the evolution of texture under cryogenic processing condition. It is well known that the evolution of texture during deformation is strongly dependent on the stacking fault energy (SFE) of the material. Smallman *et al.* reported that high SFE materials tend to form copper-type texture, whereas, brass type texture is common in low SFE systems after rolling at room temperature [2]. In FCC materials, copper-type texture comprises S {123} < 634 > Cu {112} < 111 > and Bs {110} < 112 > components, while, brass-type texture is characterized by the presence of Bs and G {110} < 001 > components, but no Cu component. The materials with SFE ranging from medium to low usually deform by twinning and shear banding mechanisms [3]. Cryogenic deformation promotes the propensity of twinning in the material and facilitates grain refinement [4]. It is also reported that SFE could affect the grain refinement during plastic deformation since it determines the probability of cross slip and hence the possibility of dynamic recovery [5–7]. It has been already reported that the recrystallization temperature for cryo-rolled (CTR) Cu is relatively low (∼100 °C) [8]. Earlier, the transition of the copper-type texture to Bs-type texture has been observed with the addition of alloying elements [9]. The medium SFE materials, like pure Cu, having coarse grain size have been found to form a strong copper component {112}< 111 > during room temperature rolling, whereas cryo-rolling and ultrafine grains lead to brass-type texture [10,11]. It is proposed that the suppression of cross slip under cryogenic condition enhances the evolution of brass component during deformation [12,13]. According to Wassermann's hypothesis, mechanical twinning is considered to be responsible for deviation of texture from copper-type to brass-type [14]. At relatively small strains, very narrow twins (∼50 nm) extensively arrange themselves as twin colonies and efficiently refine the grains [15,16]. When twinning saturates, shear band and twinning-shear band interaction plays an important role in grain refinement [15,17]. Development of adiabatic shear bands at higher strains has been also found to be responsible for grain refinement [18]. Recently, low homologous temperature [19] and even room temperature [20] annealing of cryogenic rolled Cu has been studied. With an aim to understand the recrystallization behavior at low homologous temperature, the present study aims to study the evolution of texture and microstructure in pure copper during cryo-rolling and subsequent annealing.

* Corresponding author. Tel.: +44 1142225998; fax: +44 1142225943.
E-mail address: anandgautam6@gmail.com (G. Anand).

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2. Experimental technique

The hot rolled sheet of commercially pure copper (99.9%), having a thickness of 10 mm, was used in the present study. The plate was homogenized at 750 °C for 1 h. The rolling was carried out at near-cryogenic temperature (−150 °C to −100 °C). Samples were soaked in the liquid nitrogen until the boiling of N_2 stopped. Samples were subjected to a maximum of 90% thickness reduction (equivalent to a true strain of 2.3). Annealing in the present case was carried out at 200 °C for durations of 3, 10, 30 and 60 min.

Microstructural characterization of the deformed and annealed sample was carried out using a field emitting scanning electron microscope (FE-SEM) attached with an electron back-scattered diffraction (EBSD) setup. The samples for SEM–EBSD was prepared using mechanical polishing up to 3000 grit silicon carbide emery paper and followed by electro-polishing using a Struers Electropol-5. The electrolyte consisting of 23% phosphoric acid, 23% ethanol, 47% distilled water, 4% propanol and 5 gm urea and the polishing was done at 35 V for 15 s. All the EBSD microstructure was taken on the transverse (ND–RD) plane of the rolled sheet. Transmission electron microscopy was carried out on the selected samples to observe the defect structure evolution under different stages of processing using FEI-Tecnai F-30 and T-20 transmission electron microscopes. The specimens were mechanically thinned down to 0.06 mm by manual polishing using emery papers followed by a finer polishing using Gatan model 6951 precision ion polishing system (PIPS). Transmission electron microscopy (TEM) studies were carried out on the rolling plane sections at 200 kV acceleration voltage.

Bulk texture measurement was carried out for the deformed and annealed samples using a Bruker D8 Discover texture goniometer based on Schultz reflection geometry, with Cu-K_α radiation. Four incomplete pole figures corresponding to (111), (200), (220) and (311) peaks were measured for each sample. Orientation Distribution Function (ODF) was calculated from the measured pole figures using the Labotex Software, which is based on Arbitary Defined Cell (ADC) algorithm. All measurements were performed on mid-thickness section of the specimens.

A computer controlled Instron Universal Testing machine was used to carry out tensile testing for the as received, cryo-rolled and cryo-rolled subsequently annealed samples. Dog bone shaped sample was prepared by electric-discharge machining (EDM) having the gauge length 6 mm, width 2 mm and thickness 0.5 mm. The tensile samples were cut along the rolling direction (RD). All tests were conducted at room temperature and at a strain rate of $6 \times 10^{-3}\ s^{-1}$. The specimens were elongated till failure and specimen elongation was calculated from the gauge length extension.

3. Results and discussions

3.1. Microstructural and texture characterization

Fig. 1(a)–(e) shows the EBSD image quality (IQ) map superimposed with grain boundaries and the grain boundary character distribution (GBCD) for as CTR and subsequently annealed samples. Microstructure of the as-rolled material clearly shows elongated grains along the rolling direction (Fig. 1(a)). Annealing for 3 min at 200 °C leads to a transformation of morphology from elongated type to the partially equiaxed type. However a large fraction of boundaries appear to be the low-angle boundaries in the microstructure (Fig. 1(b)). It is also clear from the microstructure that at initial stage recrystallization takes place in the elongated bands present in the deformed state. Fig. 1(c)–(e) shows the progress in recrystallization with subsequent grain growth as manifested in terms of reduction in the fraction of low angle grain boundaries, as the time of annealing increases from 10 min to 60 min. It is important to note that, there is gradual increase in the coincidence site lattice (CSL) boundaries in the annealed samples.

Cu being a medium stacking fault energy material is amenable to the formation of deformation twinning. However, in the present EBSD investigation, it has been shown that although there is less twin boundaries i.e. Σ3, Σ9, Σ27ba and Σ27b at the deformed stage, the number fraction of boundaries Σ3 increases with the progress of annealing (Fig. 2). Hence, it can be postulated that annealing twinning plays a major role in the determination of recrystallization texture and recrystallization process as a whole.

The {111} pole figures for the as-deformed and annealed samples, as calculated from EBSD, are shown in Fig. 3(a), (c), (e), (g) and (i). Fig. 3(a) and (c) reveals the presence of the under developed brass type texture. Annealing for 10 min indicates the retention of brass type texture. Further annealing for 30 min leads to the weakening of the texture. After annealing for 60 min, the sample exhibits almost random texture. The {111} pole figures determined from XRD (Fig. 3(b), (d), (f), (h) and (j)) show the similar nature, as observed from EBSD pole figures depicting close correlation in micro-texture and bulk texture.

The recrystallized grains were separated from the deformed grains in the EBSD scan based on the aspect ratio. Grains with aspect ratio greater than one were considered as deformed grains. The texture of the recrystallized grains have been determined from the partitioned scans. Volume fraction of the deformed grain has been plotted with respect to the annealing time (min), in Fig. 4(a). It can be seen that there is a sharp decrease in the fraction of deformed grains with annealing. Beyond 3 min of annealing, the changes in the elongated grain fraction are minimal, which implies the saturation in the recrystallization kinetics of cryo-rolled Cu. However, a rapid increase in the sigma boundaries is observed with increasing annealing times. The present study also explored the evolution of CSL boundaries, particularly Σ3 boundaries having misorientation of $60^\circ < 111 >$. The fast annealing response in the case of cryo-rolled sample may be attributed to the static recrystallization induced by high concentration of defect structures in the deformed material. It may be noted that high strain deformation might lead to the formation of defects [21,22]. Fig. 4(b) shows the TEM micrograph of cryogenically rolled sample. It shows fine scale deformation twin having dimension in the range of 5–10 nm. Moreover, vacancies generated at low temperature cannot get easily annihilated during annealing (marked with arrow) and are instrumental in enhancing the grain boundary mobility by providing the driving force for boundary migration [23].

The deformation microstructure in CTR sample has exhibited the distinct discontinuity in terms of size, morphology, distribution and texture of the deformed grains. Twinning has been found to be predominant deformation mechanism. The deformation structure may be sub-divided into the deformed matrix-crystal and twin-crystal areas, with the distinct discontinuity of orientation at the twin boundary. This constrains the selective growth at the twin planes. Moreover, in the case where shear band cuts through the cube oriented deformation bands, the nucleation site for the cube oriented nuclei are destroyed [24,25] and the shear band provides the nuclei of alternative orientation in the CTR sample.

In the case of CTR sample, in the cube oriented bands i.e. bands stretched in rolling direction recrystallization takes place by the nucleation of dislocation free grains and their growth. Such cube orientated grains are known to have preferable growth relationship with the as deformed structure. It is well known that the cube texture develops from cube oriented deformation bands in the face centered cubic metals like Al and Cu [26]. The origin of the cube texture has been explained in terms of oriented nucleation theory [27], where recrystallization texture originates from the preferred