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Grain structure evolution during cryogenic rolling of alpha brass

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High-resolution electron backscatter diffraction (EBSD) was used to study grain structure development during cryogenic rolling of Cu–29.5Zn brass. Microstructure evolution was found to be broadly similar to that occurring during rolling at room temperature. Specifically, favorably-oriented grains (Copper {112}⟨111⟩ and S {123}⟨634⟩) experienced profuse deformation twinning followed by extensive shear banding. This eventually produced an ultrafine structure with a mean grain size of ~0.2 μm. On the other hand, grains with crystallographic orientations close to Brass {110}⟨112⟩ and Goss {110}⟨100⟩ were found to be stable against twinning/shear banding and thus showed no significant grain refinement. As a result, the final structure developed in heavily-rolled material was distinctly inhomogeneous consisting of mm-scale remnants of original grains with poorly developed substructure and ultra-fine grain domains.

1. Introduction

Large deformation at cryogenic temperatures is sometimes considered as a promising cost-effective method for producing bulk ultra-fine grain materials [e.g. 1–12]. To date, the majority of research in the field of cryogenic working has focused on aluminum and copper alloys, most likely because of the superior ductility of these materials [1,2,4–10]. It has been established that the key mechanism governing grain-structure evolution in both materials at cryogenic temperatures is the geometrical effect of strain per se [1,2]. In other words, grains change their shape in proportion to the imposed strain, and noticeable grain subdivision and mechanical twinning are not observed [1,2]. By this means, a reasonably homogeneous grain structure, dominated by heavily elongated grains aligned with the direction of macroscopic material flow, is developed [1,2]. Such grain structures typically contain a significant proportion of low-angle boundaries [1,2] and, in the case of copper, a high density of free dislocations [2]. The limited formation of deformation-induced boundaries during cryogenic deformation is believed to be partially associated with suppression of cross-slip at low temperatures [2]. This effect is also responsible for the strengthening of the {110}⟨112⟩ Brass texture in cryo-rolled materials [1,2].

On the other hand, pronounced microstructural refinement has been observed during cryogenic deformation of commercial-purity titanium [3] and alpha brass [11,12]. In these materials, the formation of nano-scale structure has been reported and this effect has been essentially attributed to extensive mechanical twinning and shear banding [3,11,12]. Thus, it appears that cryogenic deformation is most effective in materials prone to activation of these two deformation mechanisms.

Due to its very low stacking fault energy, extensive twinning and shear banding usually occur during cold deformation of Cu–30Zn brass, and thus significant grain refinement may be expected. This effect is well documented for rolling of this material at *ambient* temperature [13]. Because dislocations in this alloy are dissociated into partials, a distinct cell structure is not developed [13]. This gives rise to significant strain hardening and thus activates profuse deformation twinning after ~50 pct. of reduction [13]. Furthermore, twinning develops very heterogeneously. The twins concentrate preferentially in grains with crystallographic orientations close to Copper {112}⟨111⟩ and S {123}⟨634⟩, whereas Goss {110}⟨100⟩ and Brass {110}⟨112⟩ orientations are typically twin-free [13]. The extensive twinning produces a nano-scale lamellar-like twin-matrix structure [13]. Due to the very small slip distance, subsequent slip in the twinned areas occurs primarily along a common twin/matrix {111} plane [13]. This provides a rotation of the slip plane toward the rolling plane, thus leading to the formation of a {111}⟨uvw⟩ γ-fiber texture and a reduction

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in the associated Schmid factor for slip to zero [13]. Simultaneously, alternate octahedral slip planes undergo latent hardening. Thus, after 50–60% thickness reduction, the twins are strongly aligned, and intense shear banding occurs as a result of the suppression of grain-scale slip [13]. The crystallographic orientations within the shear bands are widely scattered but show a certain preference for Goss {1 1 0}⟨1 0 0⟩ and Brass {1 1 0}⟨1 1 2⟩ texture components [13]. The shear bands consist of very fine (~0.1 μm) crystallites [13].

It may be hypothesized that the pronounced grain-refinement effect observed during rolling of Cu–30Zn brass at room temperature may be enhanced at cryogenic temperatures. The first studies in this area confirmed the development of extensive twinning eventually leading to the formation of a nano-scale structure [14–16]. It should be noted however that the microstructure observations in these works were performed primarily by transmission electron microscopy (TEM). Despite the excellent resolution of TEM, the statistical reliability of such results is not clear. Hence, the objective of the present work was to provide deeper insight into the mechanisms of grain refinement and texture evolution during cryogenic rolling of alpha brass using electron back-scatter diffraction (EBSD) imaging.

2. Material and experimental procedures

The program material comprised alpha brass, with a measured composition (in wt.%) of 29.5 Zn, 0.5 Pb and balance Cu. The material was manufactured by ingot casting. In an attempt to recrystallize it prior to cryogenic rolling, the material was cold rolled to a 10% thickness reduction and subsequently annealed at 800 °C for 30 min. However, recrystallization was not complete, and the resulting material contained a significant fraction of retained millimeter-size dendritic grains (Supplementary data, Fig. S1).

The material was cryogenically rolled to 90 pct. overall thickness reduction (true strain = 2.3) using a reduction per pass of 10 pct. In order to provide cryogenic deformation conditions, the rolling perform and work rolls were soaked in liquid nitrogen prior to each pass and held for 20 min; immediately after each pass, the workpiece was re-inserted into liquid nitrogen. The typical flat-rolling convention was adopted in this work; i.e. the rolling, long-transverse, and thickness/normal directions were denoted as RD, TD, and ND, respectively.

To preserve the deformation-induced microstructure, the cryo-rolled material was stored in a freezer at ~−20 °C prior to examination.

Microstructure characterization was performed primarily via EBSD examination of the mid-thickness rolling plane (containing the RD and TD). For this purpose, samples were prepared using conventional metallographic techniques followed by long-term (24 h) vibratory polishing with a colloidal-silica suspension. EBSD analysis was conducted with a JSM-7800F field-emission-gun, scanning-electron microscope (FEG-SEM) equipped with a TSL OIM™ EBSD system. To examine microstructure at different scales, several EBSD maps were acquired in each sample with a scan step size ranging from 2 to 0.05 μm. To improve the reliability of the EBSD data, small grains comprising three or fewer pixels were automatically removed from the maps using the grain-dilation option in the TSL software. Furthermore, to eliminate spurious boundaries caused by orientation noise, a lower limit boundary-misorientation cutoff of 2° was used. A 15° criterion was employed to differentiate low-angle boundaries (LABs) and high-angle boundaries (HABs).

To obtain a broader view of deformation and underlying microstructure changes, the Vickers microhardness was also measured on each sample at *ambient* temperature using a load of 100 g for 10 s. At least 25 measurements were made in each case to obtain an average value.

Because of the very large initial grain size, there was substantial variation in the deformed microstructures throughout the samples. This made *quantitative* evaluation of the typical microstructure and texture parameters difficult, and, consequently, qualitative microstructure and texture trends are shown in the present paper.

3. Results and discussion

3.1. Broad aspects of deformation and grain structure evolution

The program material showed a high level of strain hardening as evidenced by the dependence of microhardness on true thickness strain (Fig. 1a). The microhardness was approximately tripled after a true strain of ~1, thus indirectly suggesting major microstructural changes. However, at strains larger ~1, the microhardness tended to saturate (Fig. 1a) and the hardening rate approached to zero (Fig. 1b). This latter seemed to indicate a stabilization of the strain-induced microstructure.

Several different types of EBSD data (i.e., orientation maps,[1] Kikuchi-band-contrast maps, and grain-boundary maps) provided insight into the microstructures developed after different levels of reduction (Figs. 2–5). For example, after a 10-pct. reduction (true strain ~0.1), the original coarse-grained microstructure at low magnifications appeared to be almost unchanged (Fig. 2a). The sole exception to this observation was splitting of a few sporadic grains (circled in Fig. 2a). On the other hand, closer inspection of the microstructure at higher magnifications revealed evidence of substructure development. In the Kikuchi-band-contrast map (Fig. 2b), for example, the substructure appeared as alternating dark and bright bands aligned with traces of a {1 1 1} plane. In addition, the misorientations across the subboundaries were well below the EBSD detection limit of 2° (Fig. 2c).

After a reduction to 30 pct. (true strain ~0.4), there was extensive formation of deformation-induced boundaries, and their misorientations increased (Fig. 3a). Simultaneously, profuse twinning was observed in some grains[2] (Fig. 3b and c). This agrees well with prior TEM observations of cryo-rolled brass [14–16]. Therefore, an onset of the profuse twinning during cryogenic rolling seems to be shifted to lower strains as compared to rolling at room temperature (~50 pct. reduction [13]). Remarkably, the deformation-induced LABs tended to cluster in the twinned areas, and thus the microstructure became noticeably inhomogeneous (Fig. 3a). As expected, the twins had a lenticular morphology and formed packages of narrow twin/matrix lamellae aligned with {1 1 1} planes (Fig. 3b and c).

After 50-pct. reduction, the LABs and twin substructure became much denser (Fig. 4a). The microstructure heterogeneity became more pronounced as well. Closer inspection of the twinned grains revealed extensive shear banding[3] (dark bands in Kikuchi-band-contrast map in Fig. 4b). The shear bands typically comprised fine, highly-misoriented grains (an example is circled in Fig. 4c). Thus, the shear banding during cryogenic rolling also seems to be activated earlier that that during rolling at ambient temperature (~60 pct. reduction [13]).

Further increase of thickness reduction to 90 pct. (true strain ~2.3) resulted in no major changes in microstructure (Fig. 5). Specifically, the final microstructure was markedly inhomogeneous and still could be described in terms of remnants of coarse original grains with poorly developed substructure ("stagnant grains") and ultra-fine grain domains located in previously twinned areas (Fig. 5a). The latter regions consisted of twins, shear bands, and LAB substructure (Fig. 5b and c). The observed microstructure stabilization agrees well with the revealed saturation of the microhardness at high levels of reduction (Fig. 1).

Per the microstructural observations, the grain refinement process was primarily attributable to deformation twinning and shear banding. Considering the importance of these two mechanisms, they are analyzed in more detail in the following two sections.

3.2. Twinning

To provide fundamental insight into twinning, the crystallographic orientations of the twinned areas were extracted from EBSD maps and plotted as orientation distribution functions (ODFs) (Fig. 6). At relatively low strains, the ODF was dominated by the Copper {1 1 2}⟨1 1 1⟩, S {1 2 3}⟨6 3 4⟩, and Twinned Copper

[1] To see figures in color, a reader is referred to online version of this paper.

[2] The first (sporadic) twins were found after 20-pct. reduction (true strain of ~0.2).

[3] The first (sporadic) shear bands were found after 30-pct. reduction (true strain of ~0.4).