A two-step approach for producing an ultrafine-grain structure in Cu–30Zn brass

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A two-step approach involving cryogenic rolling and subsequent recrystallization annealing was developed to produce an ultrafine-grain structure in Cu–30Zn brass. The material so processed was characterized by a mean grain size of 0.5 μm, fraction of high-angle boundaries of 90 pct., a weak crystallographic texture, and strength twice that of initial material.

Keywords:

Copper alloys
Cryogenic deformation
Grain refinement
Electron backscatter diffraction
Microstructure
Texture

1. Introduction

Large deformation at cryogenic temperatures is sometimes considered as a simple and cost-effective technique for producing bulk ultrafine-grain materials [1–8]. For a number of alloys, the grain-refinement efficiency of this method is related to the enhancement of mechanical twinning and shear banding. Due to the sensitivity of these mechanisms to crystallographic orientation, however, the microstructures so produced may be extremely inhomogeneous [7,8]. One way to overcome this problem and thereby develop a more homogeneous ultrafine-grain structure may be recrystallization annealing following cryogenic deformation. In this regard, the energy stored in cryo-deformed materials tends to be large, and hence the size of recrystallization nuclei may be within the ultrafine-grain (UFG) range (≤ 1 μm). A recrystallization treatment may also be effective in promoting grain refinement for materials prone to annealing twinning. To examine the feasibility of such a two-step approach, microstructure response in Cu–30Zn brass subjected to cryogenic rolling and subsequent recrystallization annealing was determined in the present work.

2. Experiment

The program material was manufactured by ingot casting. Preforms extracted from the ingot were rolled cryogenically to 90 pct. overall thickness reduction (true strain¼2.3) using multiple passes of 10 pct. each. 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. Following cryo-rolling, small sections of the sheet were given a recrystallization anneal at 400 °C ($0.55T_m$, where T_m is the melting point) for times ranging from 1 min to 10 hours, followed by water quenching; an additional specimen was quenched immediately upon reaching 400 °C. The final microstructures were characterized primarily by an electron back-scatter diffraction (EBSD) technique. In all cases, the mid-thickness rolling plane (containing the RD and TD) was examined. 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).

3. Results and discussion

The effect of the two-step treatment on microstructure is summarized in Fig. 1. In the starting material, the microstructure was dominated by millimeter-scale dendrites containing poorly developed substructure (Fig. 1a). Cryogenic rolling yielded significant grain refinement, but the microstructure so-produced was very heterogeneous (Fig. 1b). To a first approximation, it could be described in terms of coarse remnants of original dendrites intermixed with ultrafine-grain domains. The latter regions consisted of shear bands, mechanical twins, and a dense LAB substructure. As indicated Fig. 1c, subsequent recrystallization greatly improved microstructure uniformity. The mean grain size (including twin boundaries) was rather fine, i.e., 0.5 μm (Fig. 2a), thus lying within the ultrafine-grain regime. Hence, cryogenic rolling coupled with low-temperature recrystallization annealing may indeed be used to produce an ultrafine structure in Cu–30Zn brass. Equally important, the recrystallized microstructure did not coarsen substantially during relatively long-term soaking at 400 °C (Fig. 2a), thus suggesting a reasonably-wide processing window for the annealing treatment.

Remarkably, the cryo-rolled material was completely recrystallized during the heat-up stage of the annealing treatment; i.e., prior to the isothermal hold at the peak annealing temperature. The rapid transformation of the heavily-deformed material was related presumably to the high density of defects (vacancies, in particular) which enhanced the diffusion-controlled processes. A somewhat similar effect has been recently observed in severely deformed copper-base alloys by Straumal et al. [9,10] who reported phase transformations at ambient temperature.

Not surprisingly, the final recrystallized material contained a significant proportion of annealing twins. Accordingly, the grainsize distribution was bimodal, consisting of a twin-related peak at 0.1 μm and a grain-related peak at 1 μm (Fig. 2b). These measurements indicated that the formation of annealing twins was the key mechanism providing grain refinement to the ultrafine-grain range.

The fraction of high-angle boundaries in the final recrystallized material was measured to be 90 pct. (Fig. 2c). It is noteworthy that 50 pct. of the grain-boundary area comprised twin-induced Σ3 and Σ9 misorientations with their characteristic peaks near 60° and 39°, respectively, in the misorientation-angle

distribution and corresponding rotation axes near ⟨111⟩ and ⟨110⟩ poles in the misorientation-angle plot (Fig. 2c).

The final recrystallized material was characterized by a relatively weak (≈ 3 x random) crystallographic texture (Fig. 3). The texture included several dominant components; i.e., the S {011}⟨634⟩, Q {013}⟨231⟩, Brass {011}⟨112⟩, and Dillamore {4, 4, 11}⟨11, 11, 8⟩ orientations (Table 1). The Brass and S components have been attributed to cryogenic rolling [7,8], whereas the source of the other orientations is not clear and requires further study.

The effect of the two-step treatment on the room-temperature hardness is summarized in Fig. 4. Cryogenic rolling almost tripled the microhardness relative to the as-received, as-cast condition. The pronounced material strengthening was likely related to the large degree of grain refinement associated with HABs and LABs (Fig. 1b) as well as the retention of dislocation substructure that led to work hardening. The subsequent recrystallization anneal resulted in 30 pct. reduction in hardness (Fig. 4). In view of concurrent grain refinement during recrystallization (Fig. 1c), the observed softening was likely associated with the elimination of dislocation substructure. Nevertheless, the strength of the final recrystallized material was still twice that of the as-received, as-cast material.

4. Conclusion

In conclusion, a relatively-simple two-step approach for producing an ultrafine-grain structure in Cu–30Zn brass was established. The proposed method comprised cryogenic rolling to 90pct. thickness reduction followed by recrystallization annealing at 400 °C ($0.55T_m$). The final microstructure produced by this means was characterized by a mean line-intercept length of 0.5 μm, HAB proportion of 90 pct. (with a fraction of annealing-twin boundaries of 50 pct.), and a weak crystallographic texture. The pronounced grain refinement induced by the two-step process doubled the material strength. The proposed technology may also be suitable for grain refinement in other materials with low stacking-fault energy.

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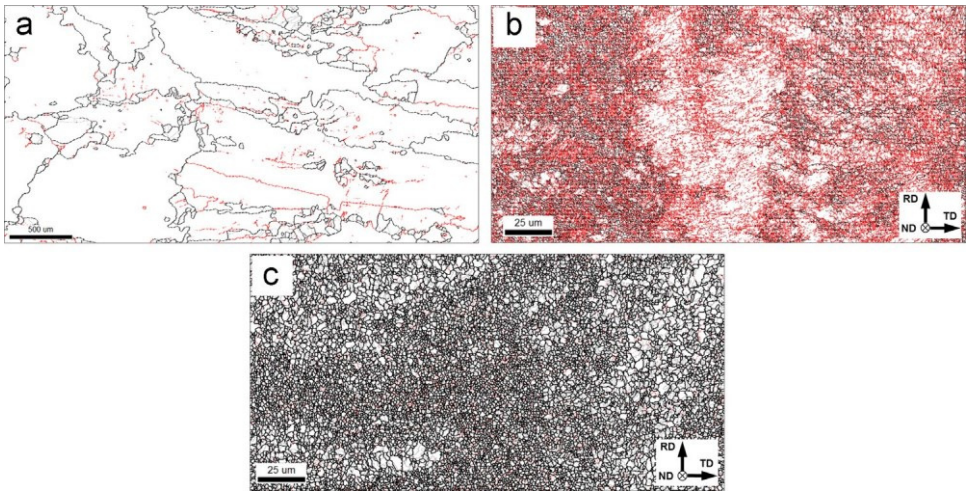

Fig. 1. EBSD maps showing the microstructure of Cu–30Zn (a) in the as-received condition, (b) after cryogenic rolling to 90 pct. thickness reduction, and (c) after rolling and subsequent recrystallization heat treatment. In the maps, LABs, HABs, and Σ3 twin boundaries are depicted as red, black, and gray lines, respectively. RD, TD and ND are rolling direction, transverse direction, and normal direction, respectively. Note the difference in magnification in the EBSD maps.

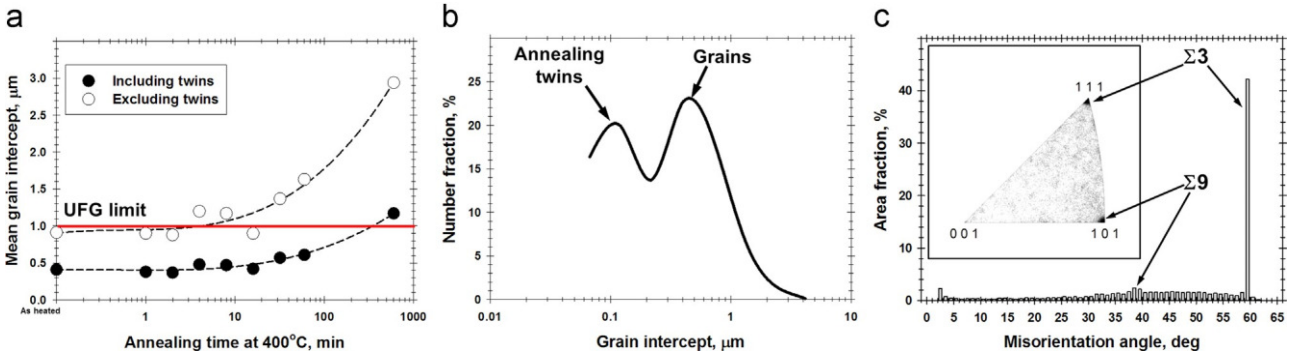

Fig. 2. (a) Effect of recrystallization time on mean grain size and (b, c) typical grain-size and misorientation distributions in the final recrystallized condition. The misorientation-axis distribution is shown as an inset in (c).

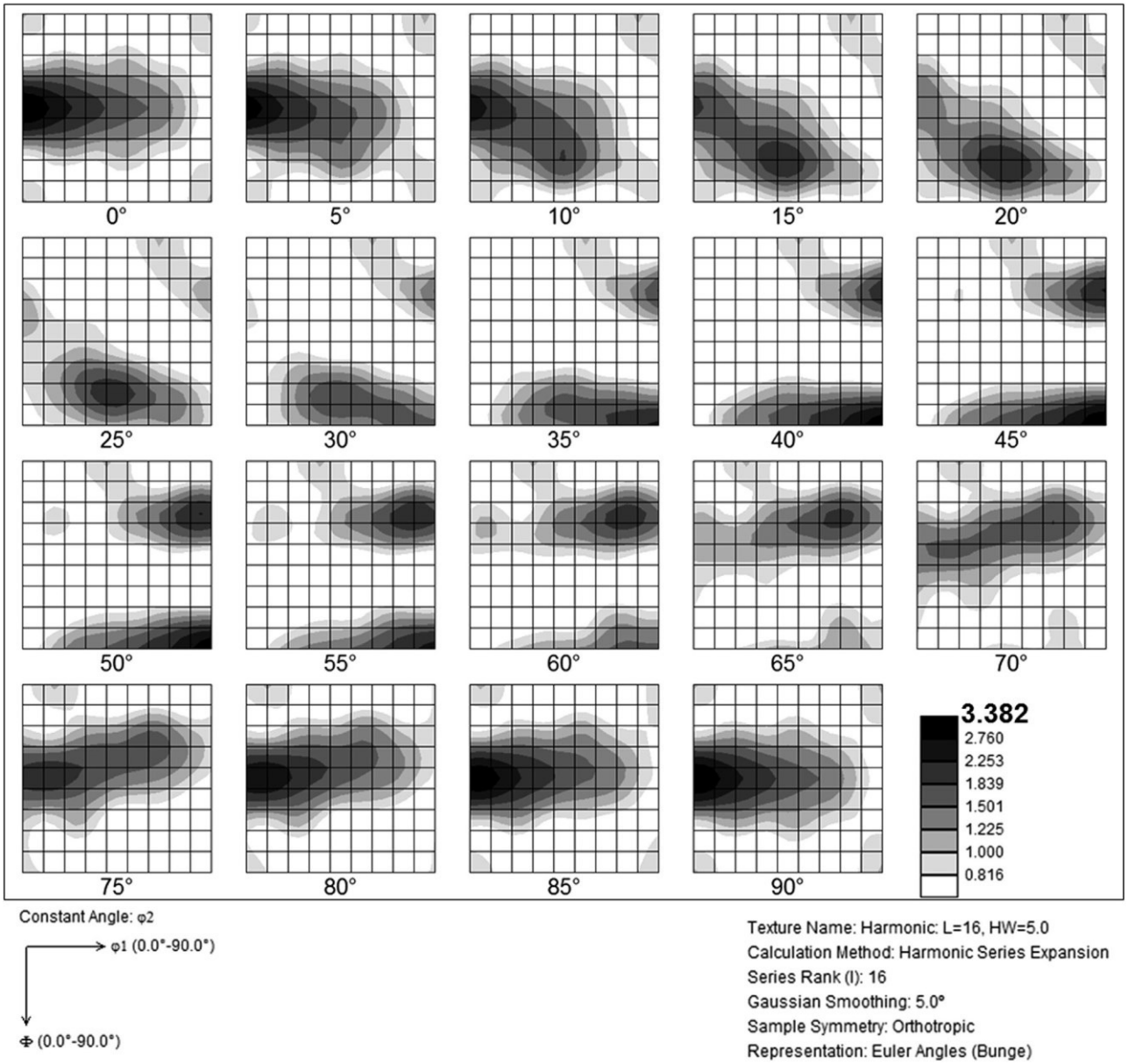

Fig. 3. Typical orientation-distribution function of the final recrystallized material.

Table 1

Volume fraction of texture components (within 15° tolerance) in recrystallized material.

Notation	Miller indices	Euler angles (φ1, Φ, φ2)	Fraction, %
Deformation texture			
Goss	{011}⟨100⟩	(0, 45, 90)	4.3
Brass	{011}⟨112⟩	(35, 45, 90)	7.8
Dillamore	{4,4,11}⟨11,11,8⟩	(90, 27, 45)	6.8
S	{123}⟨634⟩	(59, 37, 63)	9.9
Recrystallization texture			
Cube	{001}⟨100⟩	(0, 0, 0)	2.0
P	{011}⟨122⟩	(70, 45, 0)	4.5
Q	{013}⟨231⟩	(58, 18, 0)	8.0

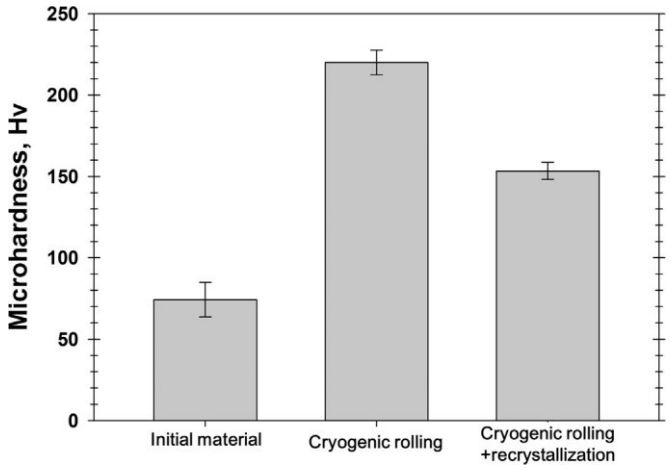

Fig. 4. Effect of two-step treatment on microhardness.