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Grain growth during annealing of cryogenically-rolled Cu–30Zn brass

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The grain-growth behavior of cryogenically-rolled Cu–30Zn brass during isothermal annealing at 900 °C was examined. The observed microstructure coarsening was interpreted in terms of normal grain growth with a grain-growth exponent of ~4. The relatively slow grain-growth kinetics was attributed to the formation of precipitates at the grain boundaries and the interaction of texture and grain growth. The development of a moderate-strength {110}<uvw> α fiber texture (~4 times random) as well as the presence of a limited number of twin variants within the grains suggested the occurrence of variant selection during annealing.

1. Introduction

The processing of metals involving large deformation at cryogenic temperatures has recently attracted considerable attention [1–18]. Low deformation temperatures are believed to suppress dynamic recovery and stimulate mechanical twinning, thereby enhancing grain refinement. This may reduce the level of strain necessary to achieve an ultrafine microstructure and thus enable the use of industrial working processes to produce ultrafine-grain materials. The cryogenic deformation is believed to be most effective for the materials prone to mechanical twinning and shear banding, e.g. for Cu–30Zn brass [13,14]. In some cases, cryogenic deformation has indeed been reported to be effective in producing substantial grain refinement [2,4,8,10]. For significant commercial application, however, ultrafine-grain materials so produced must typically be thermally stable over a range of temperatures. Hence, the annealing behavior of cryo-deformed materials is currently of interest.

For pure aluminum and copper, ultrafine-grain structures have been found to be unstable and prone to rapid, discontinuous grain growth [19–22]. Considering the large driving force for grain coarsening in such materials as well as the high density of defects inherent to cryogenic processing, this behavior is not surprising. In cryo-rolled Cu–30Zn brass, on the other hand, only a modicum of microstructure coarsening has been observed at typical recrystallization temperatures, and the ultrafine-grain microstructure has been preserved to ~0.55 T_m (where T_m is the melting point) [23]. The reason for this effect is not completely clear.

The work presented in this article is part of a wide-ranging research program whose goal is to establish the feasibility of applying cryogenic rolling to produce an ultrafine-grain structure in Cu–30Zn brass. The objective of the specific effort reported herein was to quantify the grain-growth behavior of the cryo-rolled material.

2. Material and experimental procedures

The program material comprised Cu–30Zn with a measured

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composition (in wt. %) of 29.5 Zn, 0.5 Pb, balance Cu, with traces of other elements. The material was produced by ingot casting followed by 10% cold rolling and a subsequent 30 min anneal at 800 °C. Sections of this material were then cryogenically rolled to 90-pct. overall thickness reduction (true strain = −2.3) using multiple passes of ~10 pct. each. The final sheet thickness was ~1 mm. 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 investigate the subsequent grain-growth behavior of the cryo-rolled material, samples were furnace annealed in air at 900 °C (0.95 T_m) for times ranging from 1 min to 1 h, followed by water quenching. An additional specimen was quenched immediately upon reaching 900 °C.

To provide in-depth insight into the evolution of microstructure and crystallographic texture, characterization was performed using an electron back-scatter diffraction (EBSD) technique. In all cases, the mid-thickness rolling plane (containing the RD and TD) was examined. For this purpose, samples were mechanically ground with water abrasive papers, diamond polished and finally vibratory polished with a colloidal-silica suspension for 24 h. EBSD analysis was conducted using a Hitachi S-4300SE field-emission-gun scanning-electron microscope (FEG-SEM) equipped with a TSL EDAX OIM™ EBSD system. To determine the microstructure at different length scales, several EBSD maps were acquired from each sample using different scan-step sizes ranging from 0.05 to 5 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). Grain size was quantified by the determination of the area of each grain and the calculation of its circle-equivalent diameter.

The chemical composition of different phases in the material was determined using an energy-dispersive X-ray spectroscopy (EDS) system installed in a FEG-SEM Philips XL-30.[1]

3. Results and discussion

3.1. Dezincification

During annealing of Cu–30Zn brass at relatively-high temperatures, evaporation of Zn from the free surface (i.e., dezincification) occurred. This effect is well known [24,25], and was quantified in the present work by determining the concentration of zinc *at mid-thickness rolling plane* as a function of annealing time (Fig. 1). It was determined that the zinc content at this location was reduced to ~26 wt. pct. after a 1 h exposure at the annealing temperature.

3.2. Microstructure morphology and grain size

Selected portions of EBSD grain-boundary maps illustrating microstructure evolution during annealing are shown in Fig. 2. In these maps, LABs, HABs, and Σ3 twin boundaries (within a 5° tolerance) are depicted by red, black, and gray lines,[2] respectively. The corresponding grain-size measurements are summarized in Fig. 3.

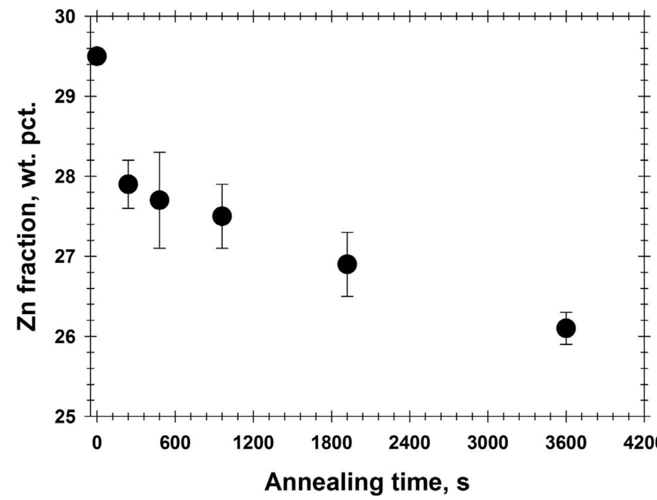

Fig. 1. Effect of annealing time on the evaporation of zinc. Error bars indicate the standard deviation of the measurements.

The microstructure of the as-cryo-rolled sample (Fig. 2a) was markedly inhomogeneous and could be described in terms of remnants of coarse, original grains with poorly-developed substructure and ultrafine-grain domains. The latter regions consisted of shear bands, mechanical twins, and a dense LAB substructure. The mean grain size of the ultrafine-grain areas was ~0.2 μm. As shown in previous works [13,14], the formation of this microstructure was related to the very heterogeneous character of deformation twinning during cryogenic rolling.

The cryo-rolled material heated to 900 °C, followed immediately by water quenching, revealed a fully-recrystallized grain structure (Fig. 2b). This microstructure was dominated by low-aspect ratio grains containing a significant proportion of annealing twins but almost no LABs. To evaluate the average number of twins per grain, the approach proposed by Field et al. [26] was used. According to this method, the number of grains both including and excluding Σ3 twin boundaries was measured and quotient of these values was calculated and summarized in Table 1. As follows from the obtained results, the number of twin variants within each recrystallized grain was limited being typically less than three; this presumably indicated the occurrence of variant selection during recrystallization [27]. The grain-size distribution was relatively wide (Fig. 3a) and likely resulted from the specific character of recrystallization of the heterogeneous cryo-rolled microstructure seen in Fig. 2a.

During subsequent soaking at 900 °C, the grain structure coarsened substantially (Fig. 3b). In terms of the largest grains, the normalized grain-size distributions became somewhat narrower (Fig. 3a), thus presumably reflecting gradual elimination of the microstructure heterogeneity inherent in the original cryo-rolled state. Nevertheless, the distributions did not change fundamentally with annealing time (Fig. 3a). Together with the similarity of the morphology of the microstructure (Fig. 2b–d), these results underlined the relatively continuous character of grain coarsening and an absence of abnormal-like features.

The kinetics of normal grain growth were quantified using the

[1] The Cu–30Zn brass is well accepted to be a *single-phase* alloy. However, microstructural observations in the current work revealed precipitation of grain-boundary segregations and even second-phase particles after annealing at 900 °C. See Section 3.4 for details.

[2] The reader is referred to the on-line version of the paper to view the figures in color.