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Microstructure instability in cryogenically deformed copper

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High-resolution electron backscatter diffraction was employed to establish the microstructural stability in severely cryodeformed copper during long-term static storage at room temperature. The material was shown to exhibit grain growth including some aspects of abnormal grain growth.

Keywords: Cryogenic deformation; Electron backscatter diffraction; Copper; Nanocrystalline microstructure

There is considerable interest in the potential use of cryogenic deformation for the production of nanocrystalline materials (e.g. [1–3]). It is believed that low homologous temperatures may suppress dynamic recovery and stimulate mechanical twinning [2,3], thus enhancing the refinement of grain size. The subsequent ambient-temperature stability of the microstructures thus produced is an important consideration with regard to the practical use of such processing approaches. For example, primary recrystallization during static storage at room temperature has been observed in cryogenically rolled copper [4,5]. This unusual phenomenon was hypothesized to be associated with a high density of vacancies in the cryodeformed material, giving rise to the exceptionally high grain-boundary mobility [4]. It may also be conjectured that such instabilities may be exacerbated with an increase in the imposed cryogenic strain such as is common during severe plastic deformation (SPD). The objective of the present work was to quantify in more detail such microstructural instability. For this purpose, the microstructural changes occurring in severely cryodeformed copper during long-term storage at room temperature were documented.

The program material consisted of commercial-purity copper whose nominal chemical composition is given in Table 1. The as-received hot-rolled bar was preconditioned by severe "abc" deformation in the temperature range of 500–300 °C [6], giving an average grain size of ~1.7 μm. Following forging, disk-shaped samples measuring 10 mm in diameter and 2 mm in thickness were cut from the central part of the billet and subjected to cryogenic deformation via high-pressure torsion (HPT) under an applied pressure of 4.5 GPa. The imposed deformation comprised 20 consecutive, fully reversed 45° rotations in the clockwise and counter-clockwise directions. To provide cryogenic deformation conditions, each test sample and the tooling anvils were soaked in liquid nitrogen and held for 20 min prior to testing. Heat-transfer calculations revealed that the warming of the sample and anvils prior to HPT due to free convection in air was relatively small, resulting in temperature increases of approximately 6 and 3 °C, respectively. Immediately after deformation, each sample was quenched in liquid nitrogen and kept at 77 K for approximately 1 year prior to examination.

Microstructural changes during static storage at room temperature were quantified for periods ranging from 2 weeks to 11 months. To provide a clear idea of the evolution of the microstructure, all observations were made at nearly the same (mid-radius) location in a given sample. Microstructures were determined primarily via electron backscatter diffraction (EBSD) but were complemented by transmission electron micros-

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Table 1. Nominal chemical composition (wt.%) of the material used.

O	Fe	Pb	S	Zn	Ag	Ni	As	Sb	Sn	Bi	Cu
0.05	0.005	0.005	0.004	0.004	0.003	0.002	0.002	0.002	0.002	0.001	Bal.

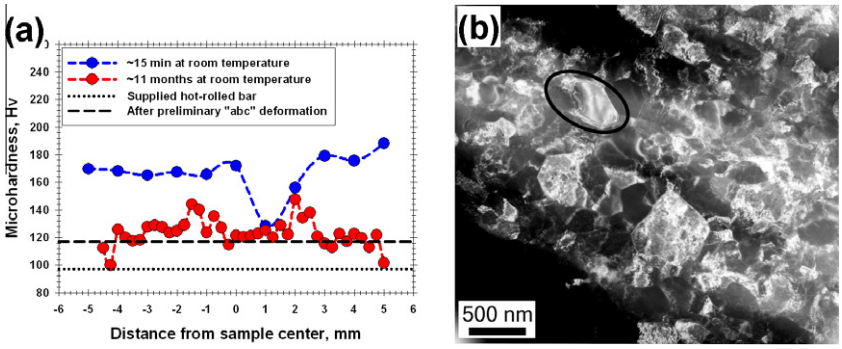

Figure 1. Effect of static storage at room temperature on Vickers microhardness (a) and typical TEM image of the microstructure taken 30 min after cryogenic deformation (b).

copy (TEM). For this purpose, samples were prepared using conventional metallographic techniques followed by electropolishing in a solution of 70% orthophosphoric acid in water at ambient temperature with an applied potential of 5 V. High-resolution EBSD analysis was conducted with a Hitachi S-4300SE field-emission-gun scanning electron microscope equipped with a TSL OIM™ EBSD system. All of the EBSD data were measured on the disk (shear) plane; the sample radial direction (RD) is vertical and the shear direction (SD) is horizontal in all results reported below. Specifically, EBSD scans consisted of ~400,000–500,000 pixels and were acquired with a step size of 25–100 nm. Although the spatial resolution of EBSD is a matter of ongoing debate, it is believed that its magnitude may be as low as ~10 nm in relatively high Z-number metals [7]; thus, a scan step size as small as 25 nm was reasonable. To minimize measurement errors, all grains comprising <3 pixels were automatically removed from the maps. Furthermore, to eliminate spurious boundaries caused by orientation noise, a lower limit boundary-misorientation cutoff of 2° was used. All misorientation angles quoted are relative to the rotation axis with the minimum misorientation. A 15° criterion was employed to define low-angle boundaries (LABs) vs. high-angle boundaries (HABs).

All measurements of grain size were made by the linear-intercept method. Because the microstructures developed by SPD are frequently characterized by a complex mixture of HABs and LABs, there is often some confusion in the definition of grains. To clarify this issue, the term "grain" in the present work refers to a crystallite bordered by a continuous boundary having a misorientation of >15°.

To obtain a broader view of underlying microstructural changes, the Vickers microhardness was also measured on each sample using a load of 50 g for 10 s. The effect of the static storage at room temperature on the microhardness profile across the sample diameter is shown in Figure 1a. After 15 min, the microhardness distribution was inhomogeneous; the well-known softening near the center of the sample was found. After 11 months at room temperature, the distribution was much more uniform and, more importantly, the magnitude of microhardness was substantially lower. These measurements thus provided indirect evidence that the cryodeformed material had experienced significant changes over time at ambient temperature.

A typical TEM image of the microstructure taken approximately 30 min after cryogenic deformation is given in Figure 1b. The microstructure consisted of nearly equiaxed grains/subgrains the size of which ranged from 50 nm to 1 μm. Coarse grains typically contained residual substructure. The dislocation density was generally high, but there were isolated grains containing almost no defects; an example of the latter is circled. In addition, some grains contained fine twins (not shown), but the twin fraction was very low.

EBSD microstructure maps of the cryodeformed copper obtained after 2 weeks and 11 months at room temperature are shown in Figure 2. The observations revealed major differences in grain structure. These differences were noticeable in maps taken with both the coarser step size of 100 nm (Fig. 2a and c) and the finer step size of 25 nm (Fig. 2b and d). In all maps, LABs are depicted as red lines[1] and HABs as black lines.

The microstructure after 2 weeks at room temperature was reasonably homogeneous. It was dominated by nearly equiaxed fine grains with an average size of ~0.3 μm (Fig. 2a). The grains contained a high density of LABs with various degrees of misorientation (Fig. 2b); the total LAB fraction constituted ~40% of total boundary area. A comparison of Figures 1b and 2b indicated that the microstructure was not fundamentally altered during the first 2 weeks of storage at room temperature.

A detailed inspection of the high-resolution EBSD map revealed that some grains were highly elongated (circled area in Fig. 2b). These high-aspect ratio grains typically contained transverse sub-boundaries tending to

[1] The reader is referred to the online version of the paper for the color figures.