# Grain refinement in copper via cryogenic deformation


Konkova Tatyana[a,1], Mironov Sergey[b,2] and Korznikov Alexander [a,3]

[a]Institute for Metals Superplasticity Problems Russian Academy of Science, 39 Khalturin Str., Ufa, 450001, Russia
[b]Department of Materials Processing, Graduate School of Engineering, Tohoku University, 6-6-02 Aramaki-aza-Aoba, Sendai 980-8579, Japan

[1] konkova_05@mail.ru, [2] smironov@material.tohoku.ac.jp, [3] korznikov@imsp.da.ru


There is currently significant interest in the development of materials with nanocrystalline grain structure for structural applications. Severe-plastic-deformation (SPD) processes have been found to be among the most effective methods for fabricating such materials [1-4]. It is believed that the microstructural refinement down to nanocrystalline (NC) range imparts a considerable increase in their strength. An important limitation of such techniques, however, is the development of an equilibrium (minimum) grain size ~ 100 nm at high levels of strain [5,6]. One of the possible means for further grain refinement is known as “cryogenic deformation”.

In present work we compare the structure formed after SPD by high-pressure torsion at cryogenic temperature in commercial-purity copper with the structure of the same material after long-term storage at room temperature. The imposed deformation comprised 20 consecutive, fully reversed 45$^{o}$ rotations in the clockwise and counter-clockwise directions under an applied pressure of 4.5 GPa. 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. Microstructural changes during static storage at room temperature were quantified for periods ranging from 2 weeks to 11 months. Microstructures were determined primarily via electron backscatter diffraction (EBSD) but were complemented by transmission electron microscopy (TEM).

The comparison of orientation maps and TEM micrographs revealed the following features:

- Severe cryogenic deformation of copper has been found to lead to very poor microstructural stability. After 11 months at room temperature, the principal feature of the microstructure was the appearance of a number of abnormal, coarse (~10 μm) grains within a matrix of fine grains (~0.4 μm), i.e. the microstructure had become essentially bimodal. The very large difference between the grain sizes suggests that the material had undergone abnormal grain growth.

- The misorientation-angle distribution measurements revealed the reduction of HAB area and formation of annealing twins during storage the sample at room temperature.

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