Microstructural Peculiarities of Copper and Mechanisms of its Hardening after Mechanical Activation and Torsion in Bridgman Anvils

I. A. Ditenberg*1,2, K. I. Denisov1,3, A. N. Tyumentsev1,2,3, M. A. Korchagin1, and A. V. Korznikov5

1 Institute of Strength Physics and Materials Science, Siberian Branch, Russian Academy of Sciences, Tomsk, 634021 Russia
2 Siberian Physical-Technical Institute, Tomsk, 634050 Russia
3 National Research Tomsk State University, Tomsk, 634050 Russia
4 Institute of Solid State Chemistry and Mechanochemistry, Siberian Branch, Russian Academy of Sciences, Novosibirsk, 630128 Russia
5 Institute for Metals Superplasticity Problems, Russian Academy of Sciences, Ufa, 450001 Russia
* e-mail: ditenberg_i@mail.ru

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Abstract—The paper presents the results of complex research on microstructural peculiarities of copper and mechanisms of its hardening after mechanical activation in planetary ball mills, high-pressure torsion, and combined treatment which includes mechanical activation and subsequent consolidation under high-pressure torsion in Bridgman anvils. The main structural factors responsible for the hardening mechanisms are discussed depending on the pattern and degree of deformation.

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1. INTRODUCTION

A wide variety of deformation methods are presently available for the formation of micro-, submicro- and nanocrystalline states [1]. However, questions on mechanisms and fundamental laws of their formation are little understood, including their dependence on conditions of plastic deformation. Despite the abundant experimental data on microstructural peculiarities and strength properties of submicro- and nanocrystalline materials [1–5], there are few works on the relation of these parameters in metals and alloys of different classes.

This paper presents the results of complex research of the copper microstructure and mechanisms of its hardening after mechanical activation in planetary ball mills, high-pressure torsion, and combined treatment which includes mechanical activation and subsequent consolidation under high-pressure torsion in Bridgman anvils.

2. MATERIAL AND INVESTIGATION PROCEDURE

Specimens of several types are investigated:

1. Specimens of 99.98% pure copper shaped into disks of the thickness \( h = 0.2 \) mm and diameter 10 mm deformed by torsion under the pressure \( \approx 5 \) GPa at room temperature and with the number of anvil revolutions \( N = 1, 2, \) and 5.

2. Powder of 99.7% pure copper after mechanical activation in the AGO-2 high-energy (40 g) planetary ball mill in argon with steel grinding media. The time of activation comprises 0.5, 1.0, 1.5, 2.0, 3.0, 4.0 and 5.0 min.

3. Specimens (disks of the diameter 8 mm and thickness \( h = 0.2 \) mm) after combined treatment including mechanical activation (3 and 4 min) and subsequent consolidation by torsion in Bridgman anvils \( (N=2, \) pressure \( 7 \) GPa) at room temperature.
Microhardness $H_\mu$ is defined by indenting the specimen with a diamond Vickers pyramid using the Neophot 21 device under load 0.051 kg during 15 s. After mechanical activation the measurements are taken for separate particles and their conglomerates, for which purpose compacts are made of the studied powder and epoxy adhesive. The microhardness of disk specimens after consolidation and high-pressure torsion is measured in sections parallel and perpendicular to the anvil plane and at different distance from the torsion axis.

The X-ray diffraction analysis employs a Shimadzu XRD 6000 diffractometer and CuKα radiation with spacing 0.01°. The average size of coherent scattering regions and lattice distortion value $\Delta d/d$ are defined by the full-profile analysis using the POWDER CELL 2.4 software and Wilkinson’s method.

The scanning and transmission electron microscopy is carried out with Philips SEM 515 and Philips CM-30 microscopes. For transmission electron microscopy of mechanically activated powders their suspension in petroleum ether is deposited on a preliminary prepared carbon film. To form thin foils from specimens after high-pressure torsion as well as compacts and bulk specimens in cross-sections a required copper layer is electrolytically deposited on the specimens. Plane specimens in the above sections are cut in the electrical discharge machine and polished mechanically to a thickness of ~100 μm. Further thinning is performed by double-sided sputtering of argon ions at an accelerating voltage of 5 kV. The transmission electron microscopy employs dark-field techniques to analyze high continuous and discrete misorientations [6–8].

3. INVESTIGATION AND RESULTS

The X-ray diffraction analysis shows that with growing time of mechanical activation of copper powder the diffraction peaks are broadened in diffraction patterns (Fig. 1a, curve 1), which is related to decreasing size of coherent scattering regions and increasing lattice distortion value (Table 1). The above structural changes are accompanied by an increase in microhardness. It grows mostly in the first 2–3 minutes of the treatment slowing down afterwards and achieves the maximum value in 5 min of mechanical activation. In this case the microhardness ($H_\mu \approx 1.86$ GPa) is comparable with the earlier found one ($H_\mu \approx 2$ GPa [10]) in copper specimens after torsion in Bridgman anvils at the number of disk revolutions $N = 5$.

After consolidation of the specimens preliminary activated by torsion in Bridgman anvils the diffraction patterns (Fig. 1, curve 2) reveal more pronounced effects of peak broadening as compared to mechanical activation (Fig. 1, curve 1). As seen from Table 2, at the number of disk revolutions $N = 2$ the microstresses and size of coherent scattering regions have almost the same values in the peripheral and central parts of the specimens and are independent of time of preliminary mechanical activation. As evident from the comparison of the tables, with a significant (from 90 to 30 nm) reduction of coherent scattering regions during torsion in Bridgman anvils the microdistortion value $\Delta d/d$ does not exceed its maximum values ($\Delta d/d = 0.37\%$) achieved under mechanical activation while the microhardness increases significantly (from 1.9 to 3.2 GPa). In this case the microhardness $H_\mu$ is $\approx 70\%$ higher than its maximum values after torsion in Bridgman anvils without preliminary mechanochemical activation [10].

The electron microscopy shows that after mechanical activation powder particles become compressed into layered conglomerates of the plate-like shape and size up to several hundred microns (Fig. 2a). Smaller powder particles range in size from several microns to several

![Fig. 1. X-ray patterns for copper after 5 min mechanical activation (1) and after 3 min mechanical activation with subsequent consolidation by high-pressure torsion at the number of disk revolutions $N = 2$ (2).](image-url)