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MICROSCOPIC CHARACTERIZATION OF SURFACE MORPHOLOGY OF NANOSTRUCTURED COPPER

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Abstract. Nanocrystalline (nc) copper was produced by severe plastic deformation through the equal-channel angular pressing (ECAP) method. Surface morphology of pure copper was studied by means of atomic force microscope (AFM) and scanning electron microscope (SEM) techniques on different scales and stages under tensile loading. The main objectives of this study are (i) examination of mechanisms of damage evolution and fracture and (ii) exploitation of the capabilities of AFM to accurately perform an analysis of the surface features of nc copper to provide the basis necessary for the development of a model for explaining the specific mechanical properties of nano-materials. It is shown that the AFM can be extremely useful for the investigations of a surface topography. It is confirmed that the dislocation-dominated plasticity and nano-voids formation are the dominant deformation mode although the deformation evolution and fracture occur through the interplay of various microstructural features.

1. INTRODUCTION

With the emergence of nanotechnology and nanoscience the investigation and application of nanostructured materials is growing rapidly. Nanocrystalline materials possess some appealing mechanical properties, such as high strength, increased resistance to tribological and environmentally – assisted damage, increasing ductility with increasing strain rate, and potential for enhanced superplastic deformation at room temperature [1]. One of the methods of grain refinement is SPD through ECAP [2]. Due to extensive and repeated deformation, the grain boundaries are highly dislocated. The microstructure is usually characterized by enough large grains up to 1000 nm separated from each other by high-angle boundaries. These grains have an internal sub-grain boundary structure [2]. Plastic instabilities in such non-equilibrium systems as heavily deformed metals are widely accepted by research community and have been proven to occur in ultra-fine grained (ufg) materials, also [1,3]. However, neither phenomelogical nor microstructural aspects of nano-materials behavior under loading have been fully understood to date.

Advances in the processing of nano-materials along with the prompt development of the sophisticated methods of investigations on micro- and nanoscales have provided unprecedented opportunities to probe the structure and mechanical response of materials. High resolution SEM and AFM are ideally suited for both visualization of nanostructured materials and for measuring the spatial dimensions of features at the surface of nanomaterials. In situ surface characterization helps to develop a better understanding of microstructure evolution of materials subjected to any kind of mechanical loading as well as to heat treatment.

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For this purpose, AFM and SEM have been used for the investigations of (i) surface morphology of deformed material; (ii) nucleation and formation of slip bands and protrusions in materials; (iii) crack propagation. The main objectives of this study are (i) examination of mechanisms of damage evolution and fracture and (2) exploitation of the capabilities of AFM to analyze the surface features of nano-structured copper subjected to plastic deformation.

2. MATERIAL AND EXPERIMENTAL PROCEDURES

In this study the attention is focused on the nc pure copper having a face-centered cubic structure. To reduce a grain size, an equal channel angular pressing (ECAP) through the route B_C using a die with an angle of 90^0 between two channels was applied [2,4]. The pressing was conducted at room temperature and a material was treated with eleven passes. Following ECAP, tensile deformed specimens were machined perpendicular to the longitudinal axes of the produced bars and those specially prepared sample segments were examined using the scanning electron microscope (SEM) Gemini, LEO, Supra 35 and AFM technique, Smena B (NTMDT) with a scanning module of 50 mm. The cantilever tip curvature was 10 nm with a force constant of 11 N/m, operating in a tapping mode. A mean grain size was calculated on the basis of an X-ray investigation. Mechanical testing was conducted at room temperature using the Instron 8516 machine operating at a constant rate of a ramp of 0.03 mm/s. First of all, samples were pulled to failure to obtain a stress – strain curve and to prepare the test segments for microstructural investigation.

Up to now there is no clear definition of grains in nanostructured materials obtained by the method of severe plastic deformation (SPD). In particular, most of the reports do not specify whether the indicated grain size is related to the size of fragments surrounded by high angle boundaries or the subgrains and cells divided by low misorientation angles. Materials produced by SPD technique usually have grains in the size range from 100 to 1000 nm. However, X-ray analysis distinguishes subgrains or X-ray coherent diffraction domains, which are much smaller than 100 nm. Therefore, taking into consideration the size of subgrains, the copper manufactured in the present work can be assumed as nano-crystalline metal.

3. RESULTS AND DISCUSSION

Examination after ECAP revealed subgrain size refinement to smaller than 100 nm, Fig. 1. It should be noticed that there was a quite broad size distribution range from 10 to 200 nm, although the average grain size was about 60 nm. Two types of grain morphologies exist in the nc copper: elongated and equiaxed. The diameters of most equiaxed grains and the widths of most elongated grains were 60 nm.

The tensile properties of ufg and nc copper have been described in detail elsewhere [2,3,5]. It just should be noticed that stress-strain curves of copper used in the present study are very similar to those reported in [2,3]. The nc pure copper shows almost no strain hardening during tension. An increase in yield strength of material up to 430 MPa is accompanied by ductility loss. Strengthening through SPD results in obtaining of the material with a tensile curve that peaks immediately after yielding. The change in material behavior after being treated by the SPD method suggests the change in the mechanisms of deformation. Plastic deformation of coarse–grained metals mostly correspondents to the motion of large numbers of dislocations. A grain boundary structure becomes particularly important in nano-materials for which the grain boundary to grain volume ratio is high. These boundaries act as sources and sink for dislocations and facilitate such stress-relief mechanisms as grain boundary sliding [6], diffusion creep of grain boundaries and grain rotation [7]. The extent of the mentioned mechanisms is still under debate. The low elongation at fracture is often attributed to plastic instability originating from the lack of an effective hardening mechanisms and/or internal flows. This instability appears as either shear bands or through 'early' necking [8]. Figs. 1b and 1c show the formation of shear bands in the severely deformed nc copper in a form of almost parallel slip lines oriented at about 45° to the loading axis.

The lower atomic packing of the grain boundaries (GB) allows more rapid diffusion of atoms in the grain boundary region. High energy of the GB makes them a more profitable area for the nucleation and growth of micro-flaws. During intensive plastic deformation micro voids are developed in the interior of the necking area of specimen. Necking results in significant increase in a surface roughness and formation of a surface 'waves' (Fig. 2a), each of them corresponds to grain sliding or other dislocation activity in the boundaries interiors. The presence of protrusions at this stage shows their

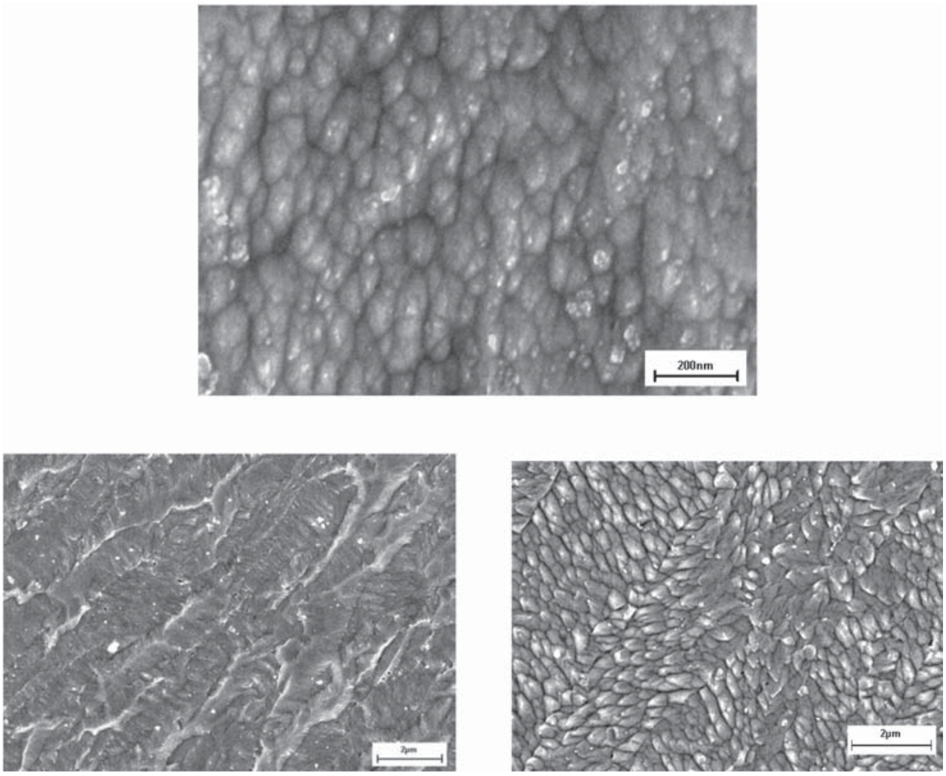

Fig. 1. SEM micrographs of nc copper: (a) – nano subgrains through cross-section at the direction perpendicular to the direction of the last ECA pressing; (b, c) – shear bands in specimen subjected to a tensile loading.

formation is uniquely related to the magnitude of the strain amplitude.

The adjacent grains are separated by a pronounced step of about 70 nm along the plane of maximum shear stress. Enhanced grain boundary diffusivity is well recognized mechanism of deformation for the materials subjected to a heat treatment, moreover, it is shown [9] that ECA pressed ultra – fine metals have the similar possibility of boundary diffusivity and, therefore, can significantly facilitate sliding at room temperature. Nanocrack generation (Fig. 2b) releases the internal stresses and diffusion processes strongly affect the competition between different deformation modes and failure mechanisms. However, a detailed analysis of the very complicated relationships between deformation, failure and diffusion processes is beyond the scope of this paper focused on AFM characterization of surface morphology.

To investigate the dislocation activity during deformation, in situ experiments were conducted using nc copper specimens produced by inert gas condensation followed by compaction [10] and electrodeposited nc nickel [11]. Very compelling and direct evidence for copious dislocation activity in nc face-centered-cubic metals was provided. In the present study, the AFM images have revealed fine steps visible at grains interior that can be attributed to a dislocation motion during deformation (Fig. 2). The average height of those fine steps inside the grain is less than 10 nm, which is a characteristic for the dislocation slip. These observations combined with results obtained by other authors [1,10,11] have confirmed that the dominant deformation mode is dislocation-mediated plasticity.

Moreover, AFM has shown the formation of the micro- and nano-scale voids evolving at grain boundaries and triple junctions, Fig. 2c. SEM micrographs of flaws are presented in Figs. 3a and 3b. The voids eventually grow and coalesce to form larger dimples. The dislocation motion at triple junctions and their role in deformation/fracture process is theoretically predicted and described in [7,12]. Deformation is instigated by the emission of dislocations at grain boundaries and then voids and/or wedge cracks (Figs. 2b and 3c) form along grain boundaries and triple junctions as a consequence of transgranular slip and non-accommodated grain boundary slid-

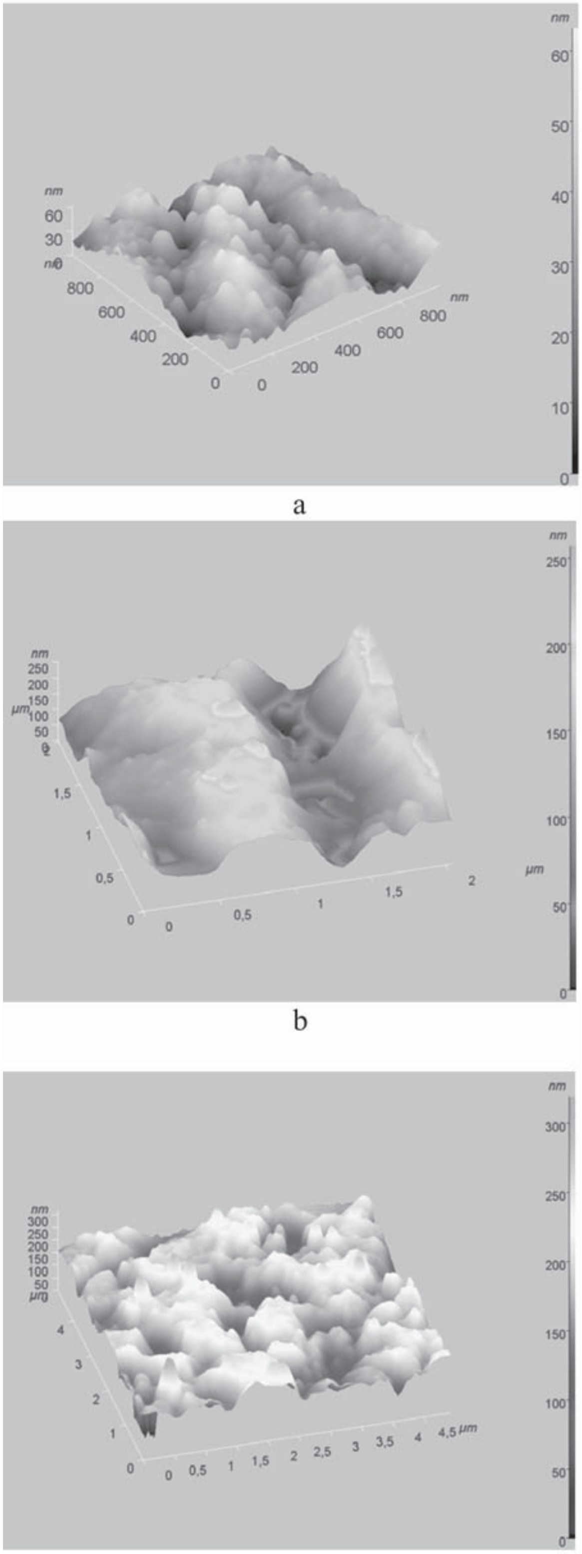

Fig. 2. AFM images of nc copper: (a) – surface topography of the pre-fractured specimen; (b) – nano-cracks nucleation; (c) micro- and nano- scale voids formation.

ing. It has been theoretically revealed that the nucleation of nano-cracks in deformed microcrystalline materials can effectively occur at triple junctions due to GB sliding. The driving force of the nucleation of triple junction nano-cracks is the release of the elastic energy associated with the GB-sliding-induced storage of GB dislocations. The similar mechanism may operate in nc materials, also.

The heavily deformed specimens in the necking area exhibit fracture surface features that primarily prove the occurrence of dimpled rupture. Fig. 3d reveals dimpled rupture with the dimple diameter and dimple depth being an order of magnitude larger than the average grain size. Those dimples exhibit a wide size distribution that may confirm unstable propagation of the cracks. Further, in [11] the presence of significant stretching of the ligaments between the dimples was proposed and it was taken to be indicative of appreciable local plasticity. Such voids do not necessarily form at every boundary. Triple junction voids and wedge cracks can also result from grain boundary sliding if resulting displacements at the boundary are not accommodated by diffusion or creep. Deformation and fracture processes are closely related to the coupling of dislocation-mediated plasticity and formation and growth of voids.

Whatever the fracture mechanism, it is evident that the fracture will be heavily influenced by microstructural features.

4. CONCLUDING REMARKS

Investigations of the material structure and mechanical processes on micro- and nano- levels create a critical foundation for the understanding of the materials behavior and for development of new generations of advanced materials. AFM is shown to be a powerful and perspective tool to study the structural features of nanocrystalline materials.

In considering the structure of nano-metals and its impact on deformation behavior, the role of grain boundaries is clearly paramount. The field of grain boundary studying would benefit immensely from dedicated experimental efforts on structures in nanometals and their response to applied stress. Evolution of damage and final fracture are all substantially affected by the starting microstructure and its consequence on microstructural evolution.

Deformation of nanocrystalline copper at low homologous temperatures (e.g. of the order of 0.1–0.2 of the melting temperature) is accompanied by the formation of localized deformation bands (shear bands), the dislocation-mediated deformation of the

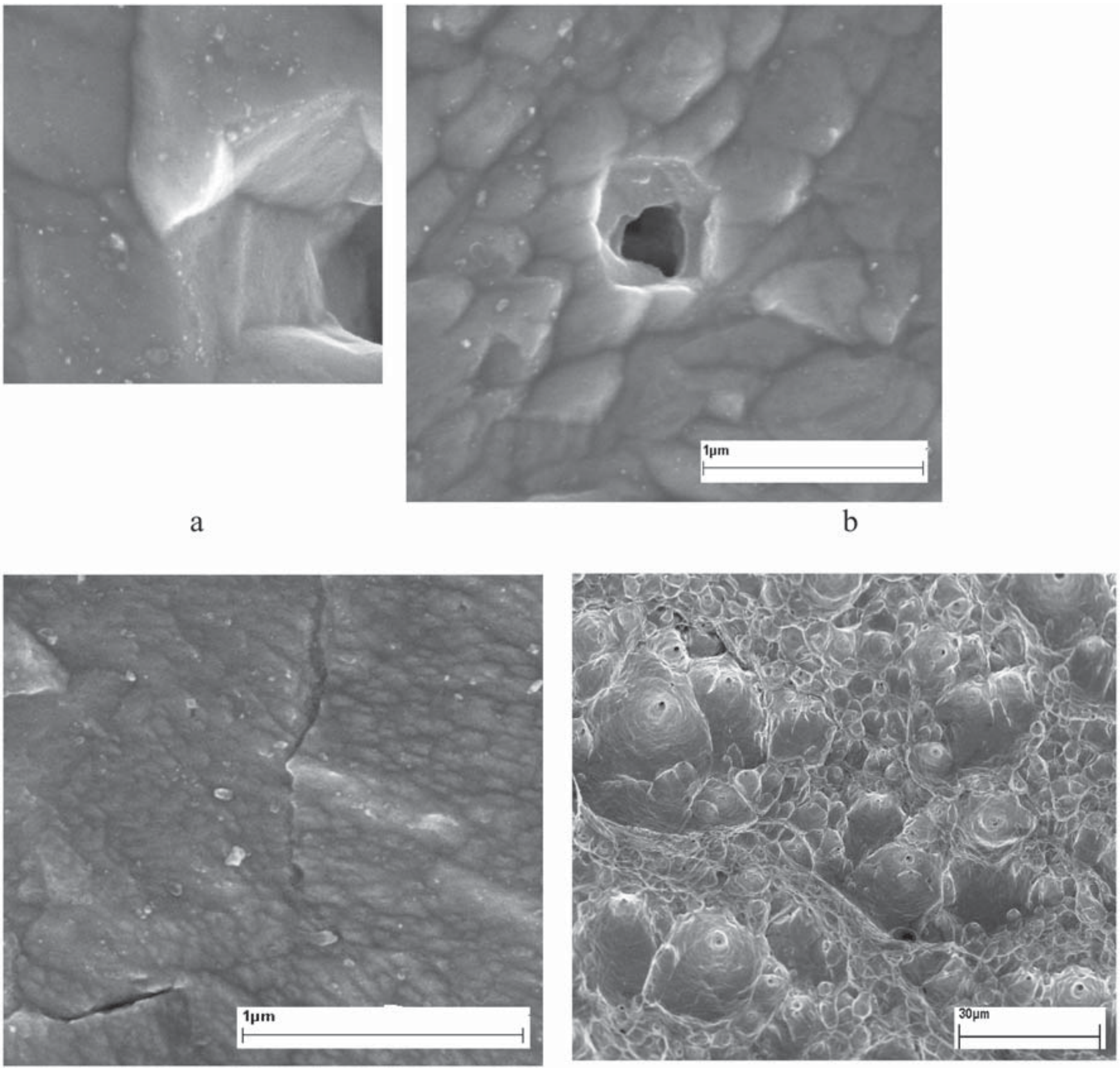

Fig. 3. SEM micrographs: (a, b) – voids and (c) – wedge cracks at the pre-fractured nano-copper; (d) – fracture surface of nc Cu tensile specimen.

grains, nano- voids formation and their growth, and nucleation of the microcracks at near boundaries regions.

The possibility of twinning deformation mode requires further discussion. Multifold deformation twins have been recently observed in nano-crystalline face-centered-cubic metals produced by SPD method [13]. The experimental observations demonstrate that nc copper can deforms by twinning at a low strain rate and room temperature. The main mechanism is proposed to be due partial dislocation emissions from grain boundary [14]. However, the analysis of SEM micrographs or AFM images does not allow confirming or disproving the twinning in nc copper. For such kind of investigations TEM technique is needed.

Fracture is characterized by the presence of dimples on the fracture surfaces, being much larger than the average grain size (dimple rupture).

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