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Nanostructural States in Nb-Al Mechanocomposite after Combined Deformation Treatment

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Abstract. Nanostructural states were investigated, that were formed in Nb-Al system-based mechanocomposite after combined deformation treatment that includes mechanical activation in a planetary ball mill and subsequent consolidation by torsion under pressure on Bridgman anvils. The formation of the layered structure, consisting of Nb and Al nanobands with width from several to several tens of nanometers was revealed. The structural states with high elastic curvature of crystal lattice and high level of local internal stresses found in Nb and Al subgrains were investigated by transmission electron microscopy.

INTRODUCTION

As it is known [1], the combined use of mechanical activation of metal powders followed by consolidation by torsion under pressure provides extensive opportunities for the creation of composite materials from multicomponent mixtures. The development of this approach requires examination of features of formation and evolution of microstructure of mechanocomposites of various systems consisting of metal powders with different types of crystal lattice, initial strength, relaxation ability, homological deformation temperature, etc.

This paper presents the results of study of microstructure of Nb-Al system-based mechanocomposite after combined deformation treatment that includes mechanical activation of pure powders mixture in a planetary ball mill and subsequent consolidation by torsion under pressure on Bridgman anvils.

EXPERIMENTAL MATERIALS AND PROCEDURES

Powders of pure Nb (99.98%) and Al (grade PA-4, purity >98%) were used. Mechanical activation was conducted on powders mixture Nb + 50 at % Al in a planetary ball mill AGO-2 in Ar atmosphere at centrifugal acceleration of milling bodies 400 m/s^2. The treatment duration was 3 minutes. Consolidation of the obtained precursor was carried out by torsion under pressure (7 GPa) at room temperature with revolutions number (N) of 1. As a result, samples discs of 8 mm in diameter and with thickness of $h \approx 0.2$ mm were obtained.

X-ray diffraction analysis was carried out using a Shimadzu XRD 6000 diffractometer. Electron microscopic investigation was conducted using transmission electron microscope Philips CM-30-TWIN (300 kV). Cross-sections, normal to anvil planes, were cut out from the consolidated samples by electric spark machine. Thin foils were prepared by sputtering the material with argon ions at an accelerating voltage of 5 kV. Research of high-defect structural states was conducted using the method of dark-field analysis of high continuous and discrete misorientations [2, 3].

Advanced Materials with Hierarchical Structure for New Technologies and Reliable Structures
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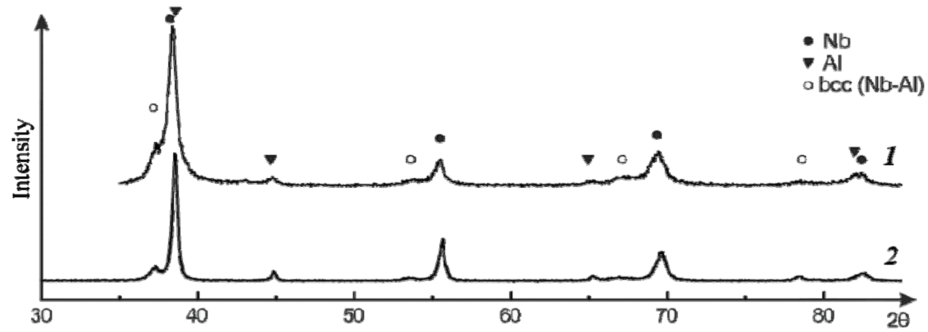

FIGURE 1. X-ray patterns of Nb + Al composite after 3 min of mechanical activation (*1*) and subsequent consolidation by torsion under pressure (*2*). Radiation CuK_α

RESULTS AND DISCUSSION

The results of investigation of microstructure features of Nb + Al mixture as well as of pure Nb after mechanical activation were presented in [2]. It was shown that two-level nanostructural states—nanograins from 50 to 100 nm in size, comprising subgrains less than 20 nm in size with low-angle misorientation boundaries and high elastic curvature (χ_{ij}) of crystal lattice—are formed in precursors after treatment. In Nb, in conditions of effective suppression of plastic relaxation of high-defect substructures in nanofragmens of about 10 nm or less in size, the elastic curvature of crystal lattice reaches (100°–200°) μm^{-1}. In Al at a relatively high homological temperature of deformation and intensive development of diffusion type relaxation processes, specifically dynamic recrystallization, (χ_{ij}) values are tens of degrees/μm.

Figure 1 shows the X-ray patterns of Nb + Al composite before and after consolidation by torsion under pressure. As it can be seen, at the mechanical activation (for Nb, a = 3.3 Å), which does not match any of the equilibrium compounds of Nb and Al. This feature was previously found in [4, 5]. Authors of [4] showed that this phase is metastable version of intermetallic Nb_2Al, which turn into the stable modification upon heating.

From the submitted X-ray patterns (Fig. 1), it follows that both at stages of mechanical activation and consolidation by torsion under pressure there is a substantial broadening of diffraction peaks, indicating a decrease in size of coherent scattering regions (CSR) and increase in values of microdistortions ($\Delta d/d$) of crystal lattice. As a result of analysis of X-ray peaks profiles it was found that after complex processing characteristic CSR size for Nb is about 19 nm and $\Delta d/d$—0.13%, in the aluminum component these parameters are respectively 12 nm and 0.1%.

As a result of electron microscopic study of the cross-sections of Nb+Al mechanocomposite (at true logarithmic deformation $e \approx 2.8$, 0.5 mm from the center) alternation of Nb and Al layers (Fig. 2a) was detected. These layers are generally partitioned into bands with large-angle boundaries parallel to anvils' planes (AP). By increasing the deformation degree (up to $e \approx 4.7$, 3.5 mm from the center) a significant decrease in the thickness of the layers (Fig. 2b) is observed. Meanwhile, layers partition on bands was detected only in Nb, while Al is presented in the form of thin (10–30 nm) interlayers, fragmented by transverse boundaries (Fig. 2b). Figure 3 represents histograms of Nb and Al bands width distribution in regions with deformation degree $e \approx 2.8$ (a) and 4.7 (b), constructed from the data of dark-field analysis of misorientations. Analysis showed that with increase in deformation degree from $e \approx 2.8$ to $e \approx 4.7$ average band width of Nb decreases from 47 to 19 nm, and of Al—from 45 to 14 nm. Thus, during the torsion under pressure not only material consolidation occurs but also a significant transformation of the microstructure is observed with increase in deformation degree. It should be noted that the characteristic width of nanobands, formed after the combined treatment, is substantially less than the minimum grain size after torsion under pressure of bulk materials (about 200 nm for Nb and Al [6, 7]). On the corresponding selected area diffraction patterns this intensification of grain structure fragmentation is observed in the form of increase in density of the diffraction maxima (Figs. 2a and 2b). In addition there is the azimuthal (up to a few degrees) reflexes blur (Fig. 2b), indicating the presence of a continuous misorientations.

Against the background of the above nanoband structure after reaching $e \approx 4.7$ the formation of "twists" and "vortexes" (Fig. 2c) was revealed. Their spatial scales are in the range from tens of nanometers to a few microns. As can be seen in these areas (Fig. 2c) there are local variations in direction of bands propagation from the direction of the external shear stresses (that are parallel to the anvils planes).

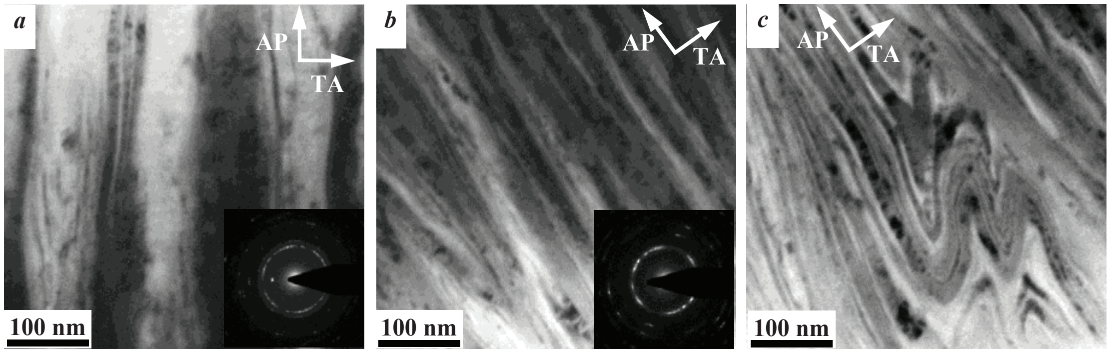

FIGURE 2. Brightfield electron microscopic images of Nb + Al mechanocomposite microstructure after consolidation by torsion under pressure when $e \approx 2.8$ (a) and $e \approx 4.7$ (b, c). TA—torsion axis, AP—anvils plane

Analysis of discrete and continuous misorientations, conducted by the methods [2, 3], showed that after combined treatment the characteristic values of crystal lattice curvature are identical to those observed immediately after mechanical activation [2]. In Al nanobands the lattice curvature reaches several tens of degrees/μm. For Nb, crystal lattice curvature values of 100°–200° μm^{-1} are characteristic, and dimensions of regions of their detection usually ranges from several nanometers to several tens of nanometers. The fact that in these areas lacks both individual dislocations and their clusters indicates the elastic nature of high curvature of crystal lattice [2, 3]. Estimates of local internal stresses (σ_{loc}) and their gradients ($\partial\sigma_{loc}/\partial r$), carried out in accordance with [2, 3, 8], showed that in Nb the maximum values of these parameters reaches $\sigma_{loc} \approx E/20$ and $\partial\sigma_{loc}/\partial r \sim E/2\ \mu m^{-1}$ (E—Young's modulus), while in Al they do not exceed $\sigma_{loc} \approx E/120$ and $\partial\sigma_{loc}/\partial r \sim E/12\ \mu m^{-1}$.

Thus, the parameters of grain structure and elastic-stressed state characteristics of components of composite are determined not only by method and plastic deformation degree (e) reached, but also greatly depend on the relaxation ability of the material, its homological deformation temperature, initial and acquired strength. It should be noted that in consolidated samples, local inhomogeneity of microstructure, and consequently of strength properties, are often legacy of heterogeneity of powder particles after mechanical activation. These inhomogeneities may become stress concentrators that promote the formation of the above "vortex"-type structures (Fig. 2c) during consolidation by torsion under pressure.

In accordance with [3], the presence of structural states with high curvature of crystal lattice and high level of local internal stresses indicates the realization of two-stage dislocation-disclination mechanism of fragmentation and reorientation that provides transformation of microstructure on initial stages of deformation (consolidation) by torsion under pressure. On the nanoscale of deformation (<100 nm), relaxation of high-defect structural states by separating of dislocation ensembles into two subsystems of dislocations of opposite signs is impossible.

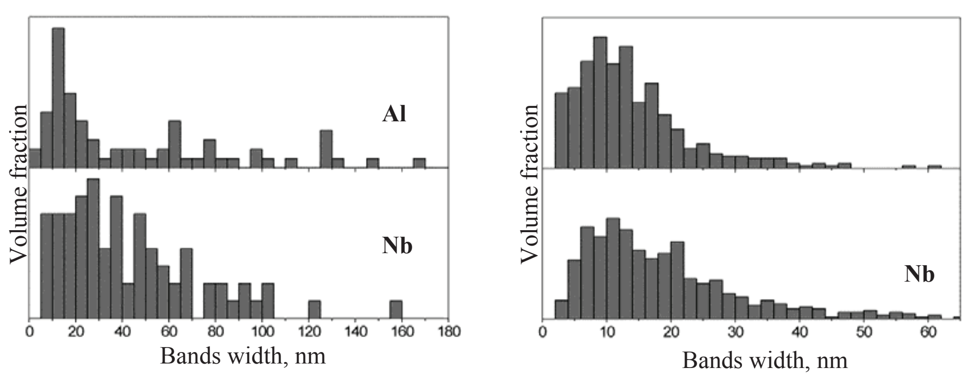

FIGURE 3. Histograms of Nb and Al bands width distribution in Nb + Al composite after consolidation by torsion under pressure when $e \approx 2.8$ (a) and $e \approx 4.7$ (b)

In [8] in the discussion of such nanostructural states, it was suggested that their formation can be described in terms of a two-stage mechanism of plastic deformation and reorientation of the crystal: formation of nanobands of localized elastic distortion by means of generation and propagation of nanodipoles of partial disclinations and subsequent plastic relaxation by flows of nonequilibrium point defects in the fields of high local pressure gradients.

Nanoband structural state, discovered in the composite under study, is similar to laminated structures formed by vacuum rolling [9] or magnetron sputtering [10]. What is essential that in comparison with these methods, combined treatment used in this study is technologically simpler and does not require additional thermal exposure. Composites received are characterized by a specific area of interphase boundaries at 10 m^2/g, so they can be regarded as precursors for the controlled production of nanostructured intermetallic compounds based on Nb-Al system. Moreover, an important advantage of combined treatment is intensive fragmentation of Nb and Al layers on grains and subgrains with high density of crystal defects, which, firstly, promotes hardening of the resulting material; secondly, related to these defects high stored energy of deformation and a significant increase in the diffusion coefficients stimulate an increase in reactivity of mechanocomposite components.

SUMMARY

After the combined treatment of Nb + Al composite the formation of the layered structure, consisting of Nb and Al nanobands with width from several to several tens of nanometers, preferably extending parallel to anvils, was revealed. These nanobands are usually fragmented by low-angle boundaries onto subgrains with high defectiveness.

It was found that the curvature of the crystalline lattice in Al grains reaches several tens of degrees/μm, while in Nb high (100°–200° μm^{-1}) elastic curvature of crystal lattice is localized in areas no larger than several tens of nanometers. It is assumed that the difference in microstructure parameters of Nb and Al is due to different homological deformation temperatures, relaxation ability and strength of materials.

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