

# Elasto-Plastic Properties of Cu–Nb Nanolaminate

V. I. Betekhtin\*, Yu. R. Kolobov, B. K. Kardashev, E. V. Golosov,  
M. V. Narykova, A. G. Kadomtsev, D. N. Klimenko, and M. I. Karpov

*Ioffe Physical Technical Institute, Russian Academy of Sciences, St. Petersburg, 194021 Russia*

*Research and Education Center for Nanomaterials and Technology, Belgorod State University, Belgorod, 308007 Russia*

*Institute of Solid State Physics, Russian Academy of Sciences, Chernogolovka, Moscow oblast, 142432 Russia*

\*e-mail: Vladimir.Betekhtin@mail.ioffe.ru

Received August 1, 2011

**Abstract**—The Young’s modulus, internal friction, and microplastic flow stress in Cu–Nb nanolaminate has been determined by an acoustic technique. The influence of high hydrostatic compression (1 GPa) on these elasto-plastic properties of the nanolaminate has been studied.

Multilayer composites with a layer thickness on a nanometer scale are called nanolaminates [1–3] and have good prospects for application as both structural and functional materials possessing special mechanical, magnetic, and electrical properties due to unique combinations of characteristics of the component layers, in particular, metals. This stimulates investigations of various characteristics of nanolaminates, including the structural parameters, thermal stability, ultimate strength, flow stress, microhardness, etc. [4]. The elasto-plastic properties of these multilayer composites with nanodimensional layers have been studied to a much lesser extent.

In this work, acoustic measurements were used to determine the elasto-plastic characteristics of a nanolaminate of the Cu–Nb system, including the characteristics of elastic deformation (Young’s modulus  $E$ ) and reversible microplastic deformation (amplitude-independent decrement  $\delta$  and microplastic flow stress  $\sigma$ ) related to the oscillatory motion of dislocations. A special feature of the acoustic experiment is that, at moderate amplitudes, the dislocation structure of samples is retained and the density of dislocations in the metal remains unchanged [5].

The samples were studied by a resonant method of a composite vibrator, which is described in much detail elsewhere [5]. Using this technique, it is possible to determine the Young’s modulus  $E$  and study the absorption of ultrasound (i.e., internal friction) and inelastic (microplastic) properties of samples. The data on inelastic properties are obtained by measuring the  $E$  and  $\delta$  values in a wide range of amplitudes of vibrational strain  $\varepsilon$ . At sufficiently large  $\varepsilon$ , a sample material exhibits nonlinear amplitude-dependent absorption  $\delta_h = \delta - \delta_i$  and amplitude-dependent shift of the Young’s modulus, which is defined as  $(\Delta E/E)_h = (E - E_i)/E_i$ , where  $E_i$  and  $\delta_i$  are the Young’s modulus

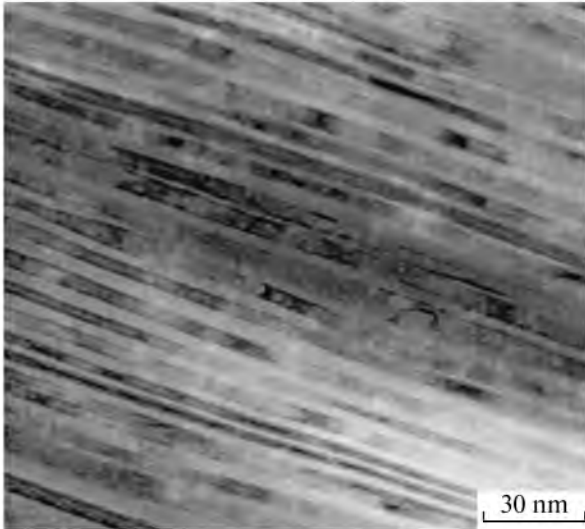
and decrement determined at small amplitudes (for which both  $E$  and  $\Delta$  are independent of  $\varepsilon$ ).

Using the results of acoustic measurements performed in a wide range of vibration amplitudes, it is possible to evaluate the mechanical (microplastic) characteristics of materials in the conventional mechanical testing coordinates of stress versus inelastic strain, where the ordinate represents the amplitude of vibrational stress  $\sigma = E\varepsilon$  and the abscissa represents the nonlinear inelastic strain  $\varepsilon_d = \varepsilon(\Delta E/E)_h$ .

The samples for acoustic measurements were rod-shaped with a  $2 \times 0.5$  mm rectangular cross section and a length of  $l = 20$  mm, which corresponded to resonance frequency longitudinal oscillations close to  $f \sim 100$  kHz. The Young’s modulus was defined as  $E = 4\rho l^2 f^2$ , where  $\rho$  is the material density.

The Cu–Nb nanolaminate was obtained by complex rolling of copper and niobium sheets and represented a sandwich of these metals, in which the thickness of each layer (see Fig. 1) was about 10 nm. The boundaries of these layers were oriented in the direction of propagation of a longitudinal acoustic wave. The effective density of the nanolaminate was determined by high-precision weighing in air and distilled water.

Figure 2a shows the amplitude dependences of the Young’s modulus  $E$  and logarithmic decrement  $\delta$  of the nanolaminate in the initial state and after hydrostatic compression to 1 GPa. Both the  $E(\varepsilon)$  and  $\delta(\varepsilon)$  curves were measured by sequentially increasing and decreasing the vibration amplitude. As can be seen, both quantities reveal a hysteresis, whereby the curves measured while increasing and decreasing the amplitude do not coincide with each other. However, both shape and numerical values significantly change upon high-pressure treatment (1 GPa, 16 min, 18°C). It



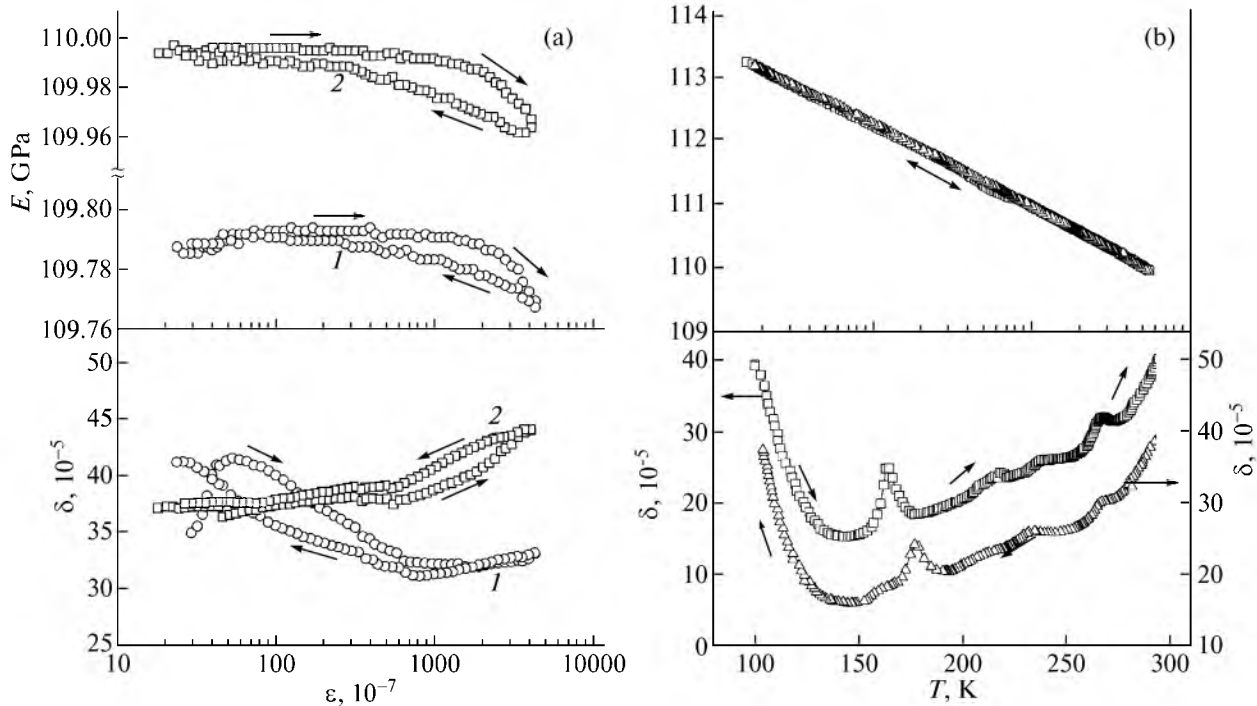
**Fig. 1.** Transmission electron microscopy image showing microstructure of nanolaminate of Cu–Nb system (transverse section).

should be noted that the samples were processed in protective shells.

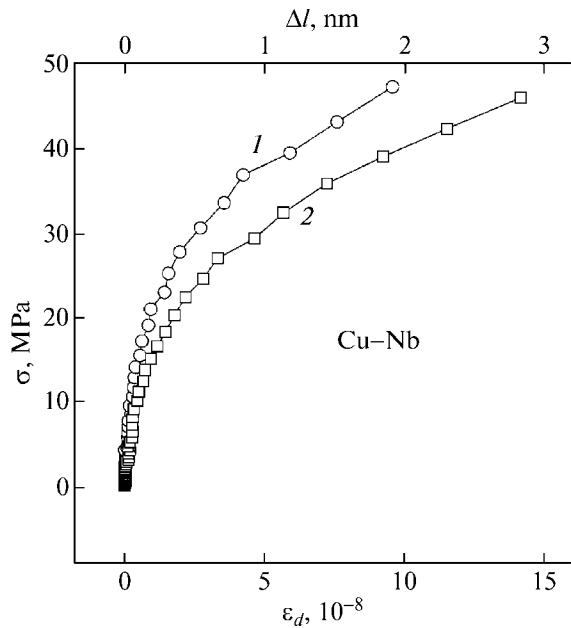
Figure 3 presents the diagrams of acoustic straining  $\sigma(\varepsilon_d)$ , where  $\varepsilon_d$  is the relative inelastic deformation. These plots were constructed using the curves of Fig. 2

measured in the first cycle of increasing amplitude. This figure also shows a plot of the absolute deformation  $\delta l$  determined for a 2-cm-long sample. The acoustic straining diagrams are presented for the same sample measured before (curve 1) and after (curve 2) hydrostatic compression.

As can be seen from the obtained experimental results, the Young's modulus somewhat increases after the hydrostatic compression. A more significant change upon pressure treatment is observed for the logarithmic decrement, which is probably related to the fact that  $\delta$  is more sensitive (than  $E$ ) to the regions of ultrasound absorption, which are related not only to dislocations. Indeed, the maximum observed on the amplitude dependence of the decrement (for the initial sample not subjected to hydrostatic compression) is most probably related to the presence of discontinuities (e.g., microscopic cracks, pores, regions of reduced density, etc.) in the sample. These discontinuities can appear at the Cu/Nb interfaces during manufacture of the laminate. It was demonstrated [6] that high-pressure treatment can lead to partial or complete healing of discontinuities. Evidence in favor of this hypothesis is provided by the results of determining the density of samples. Indeed, the initial density of the laminate ( $8.547 \text{ g/cm}^3$ ) was lower than the value ( $8.78 \text{ g/cm}^3$ ) calculated taking into account the densities of Cu and Nb and assuming their equal fractions in the laminate. After the hydrostatic compression, the



**Fig. 2.** (a) Amplitude and (b) temperature dependences of the Young's modulus  $E$  and logarithmic decrement  $\delta$  of Cu–Nb nanolaminate. The amplitude dependences were measured (1) before and (2) after hydrostatic compression at 1 GPa. The temperature dependences were measured on cooling, followed by heating (thin arrows indicate the direction of temperature variation); the  $\delta$  curves are shifted over the ordinate.



**Fig. 3.** Diagrams of microplastic deformation of the Cu–Nb nanolaminate, constructed using the results of acoustic measurements (1) before and (2) after hydrostatic compression at 1 GPa ( $\epsilon_d$  is the relative inelastic deformation, and  $\Delta l$  is the absolute deformation of a 2-cm-long sample).

sample density increased to  $8.625 \text{ g/cm}^3$ , which could be explained by a decrease in the porosity of the nanolaminate. It should be noted that the necessity of allowing for the porosity as a factor influencing the elastic properties of nanomaterials was already pointed out in [7, 8]. This probably also accounts for the increase in  $E$  observed in samples upon hydrostatic compression. The high-pressure treatment also somewhat changed the diagram of microplastic deformation (Fig. 3). After compression, the nanolaminate became “softer” and its microplasticity increased.

Figure 2b shows the temperature dependences of the Young’s modulus  $E$  and logarithmic decrement  $\delta$  measured in the cooling cycle followed by heating of a sample upon hydrostatic compression. As can be seen from these data, the Young’s modulus exhibits linear growth with decreasing temperature. The temperature dependence of the decrement reveals several features

that are manifested as maxima and bending points. These features are most probably related to small deformations arising at the Cu/Nb interfaces under the action of thermoelastic stresses. These deformations exhibit reversible variation in the cooling–heating cycle with a temperature shift of 10–20 K.

Thus, we have performed a complex investigation of the elasto-plastic properties (Young’s modulus, amplitude-independent decrement, microplastic deformation) of a Cu–Nb nanolaminate in a temperature interval of 100–293 K. Analysis of the obtained experimental data on the behavior of  $E$ ,  $\delta$ , and  $\sigma_s$  before and after high-pressure (1 GPa) treatment led to the conclusion that the samples of the nanolaminate contain discontinuities that could form at Cu/Nb interfaces during preparation of the material. Evidence in favor of this hypothesis comes from the results of measuring the density of samples before and after hydrostatic compression. It is important that a decrease in the volume of discontinuities improves the elasto-plastic properties of the nanolaminate.

## REFERENCES

1. M. I. Karpov, V. I. Vnukov, K. G. Volkov, N. V. Medved', and I. I. Khodos, *Materialovedenie*, No. 1, 48 (2004).
2. A. Misra, M. J. Demkowicz, X. Zhang, and R. G. Hoagland, *JOM*, 62 (2007).
3. B. S. Bokshstein, V. I. Vnukov, E. V. Golosov, M. I. Karpov, Yu. R. Kolobov, D. A. Kolesnikov, V. P. Korzhov, and A. O. Rodin, *Izv. Vyssh. Ucheb. Zaved., Fiz.*, No. 8, 40 (2009) [*Russ. Phys. J.* **52**, 811 (2009)].
4. Yu. R. Kolobov, A. G. Lipnitskii, M. B. Ivanov, and E. V. Golosov, *Kompoz. Nanostrukt.*, No. 2, 5 (2009).
5. S. P. Nikanorov and B. K. Kardashev, *Dislocational Elasticity and Inelasticity of Crystals* (Nauka, Moscow, 1985) [in Russian].
6. V. I. Betekhtin, S. Yu. Veselkov, Yu. M. Dal', A. G. Kadomtsev, and O. V. Amosova, *Fiz. Tverd. Tela* **45** (4), 618 (2003) [*Phys. Solid State* **45**, 649 (2003)].
7. R. Chaim and M. Hefetz, *J. Mater. Sci.* **39**, 3057 (2004).
8. V. I. Betekhtin, V. Scenicka, I. Saxl, B. K. Kardashev, A. G. Kadomtsev, and M. V. Narykova, *Fiz. Tverd. Tela* **52** (8), 1517 (2010) [*Phys. Solid State* **45**, 1629 (2010)].

*Translated by P. Pozdeev*