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THE STRENGTHENING AND THE ELECTRICAL RESISTIVITY OF THERMO-MECHANICAL DEFORMATION PROCESSED CU-NB COMPOSITE WIRES

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Abstract. The effects of deformation and heat treatment on the electrical resistivity and the tensile strength of nanostructured Cu/Cu-18%Nb composite wires are studied. The correlated behavior of electrical resistivity and strength depending on the mode of thermo-mechanical processing is ascertained. The main mechanism of strengthening and increasing the electrical conductivity, associated with the reduction in the size of the structure, is considered. A good agreement is shown between the average distances between the Nb filament measured by SEM and extracted using the inversion of the resistivity size effect model. On the other hand, direct calculations of the strength value in accordance with the most common modern models are not consistent with experiments. In conclusion, the reasons for this discrepancy are discussed.

Keywords: Cu-Nb, nanocomposute, size effect, strengthening, intrinsic material scale

1. Introduction

Nanostructured wires produced by heavy deformation of the in situ formed Cu-Nb composites are promising for innovative cable industry [1-3]. Owing to extremely high mechanical strength, these wires are used as winding wires of pulse magnet coils with a record peak magnetic induction of up to 100 T [4,5]. The impressive values of tensile strengths in the range of 1200-1300 MPa simultaneously with the electrical conductivity of 60-70% of the value adopted as the international standard for annealed copper have been attained in commercial Cu-Nb wires [6,7]. The simultaneous achievement of these properties is necessary because, on the one hand, the strength value determines the maximum permissible Lorentz force acting on the winding during current pulses, and hence the maximum permissible magnetic field induction. On the other hand, the level of electrical conductivity determines the permissible heating of the wire under the current flow and hence the duration of the pulse [8].

A typical Cu-Nb wire architecture includes a Cu-Nb core, longitudinal copper elements and an outer shell of high purity copper. The volume fraction of these elements in the ready-to-use wires can vary depending on the required cross-section and the strength/conductivity ratio. The main contribution to strength of the wire gives the Cu-Nb core. High strength of the Cu-Nb is achieved after a sufficiently high degree of deformation by drawing. The favorable combination of the elasticity moduli of FCC copper and BCC niobium due to strong

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Kurdyumov-Zacks type texturing provides the ribbon-like shape of the initial Nb particles with high aspect ratio and high density of Cu/Nb interfaces [9].

It is well known that simultaneously with the transition from micro- to nano-size of structural components during the deformation of Cu-Nb core, there are an anomalous increase in strength significantly exceeded the estimates obtained from the rule of the mixture [6,9,10] as well as an increase in the electrical resistivity caused by the scattering of the electrons on the internal interphase surfaces [11-13]. Both these phenomena are usually called size effects.

The increase in the resistivity is well described in the framework of the Dingle's model [14,15], which was originally developed for a thin copper wire. This model has been successfully applied to Cu-Nb nanocomposites. The size effect is manifested when the transverse size of the copper channels became of the same order with a certain characteristic length - the electron mean free path in the bulk copper [13].

On the other hand, the strength is determined by the mechanisms of motion/pinning of the dislocations. For a correct description of the *size effect*, by analogy with the electrical resistivity, it is necessary to introduce some characteristic scale of nanocomposite materials, under which the neglect of the size effect will lead to an underestimation of strength calculated by the rule of the mixture. However, the development of such a theory is not a trivial task and many attempts have been made [19-21].

In this paper, we test two modern models that have been proposed to explain the size effect influence on the Cu-Nb wire strength. The interrelation between the anomalous increase in strength and resistivity of Cu/Cu-18%Nb wires produced by the "melting-deformation" method is investigated experimentally in relation with the modes of the thermo-mechanical processing. We study the transverse sizes of the structural components, the mechanical strength and electrical conductivity in a wide range of wire deformation. Then the applicability of existing models for describing the strength increase size effect of Cu-Nb wires is analyzed.

2. Samples and methods

In this paper we investigate 3 model wires produced from two similar ingots of Cu-18wt.%Nb. These ingots have been obtained by double arc melting with consumable electrodes using high-purity electron-beam melted copper (99.99%) and electron-beam melted niobium. Wire A has been fabricated from first ingot by hot extrusion and consequent drawing without any heat treatments (Fig. 1). Wires B and C have been fabricated from the second ingot in accordance with the flow diagram in Fig. 1. The more detailed description of the fabrication procedure of in-situ nanocomposite Cu-Nb wires is given in ref. [7].

The degree of deformation has been defined as $\eta=\ln(S_0/S)$, where S_0 is the cross sectional area of the wire before cold drawing (28 mm in diameter) and S is the cross sectional area of the sample after the deformation.

Resistivity of Cu-Nb wires has been determined by the DC 4-probe technique on samples of about 80 mm in length with the accuracy not less than 2% at 293 K and 77 K.

Tensile tests have been performed on the samples of 200 mm in length by Instron TT-DM testing machine at 293 K with the accuracy not less than 3 % for fine wires.

The evaluation of average size of Cu-channels between Nb tape-like filaments (also known as the spacing or d_{Cu}) has been performed on the pieces of polished cross sections of wire A samples with diameters of 28; 10; 5 and 2 mm by method of random secant lines on Scanning Electron Microscope (SEM) images. The value of d_{Cu} for each diameter has been determined based on approximately 3 000 measurements.

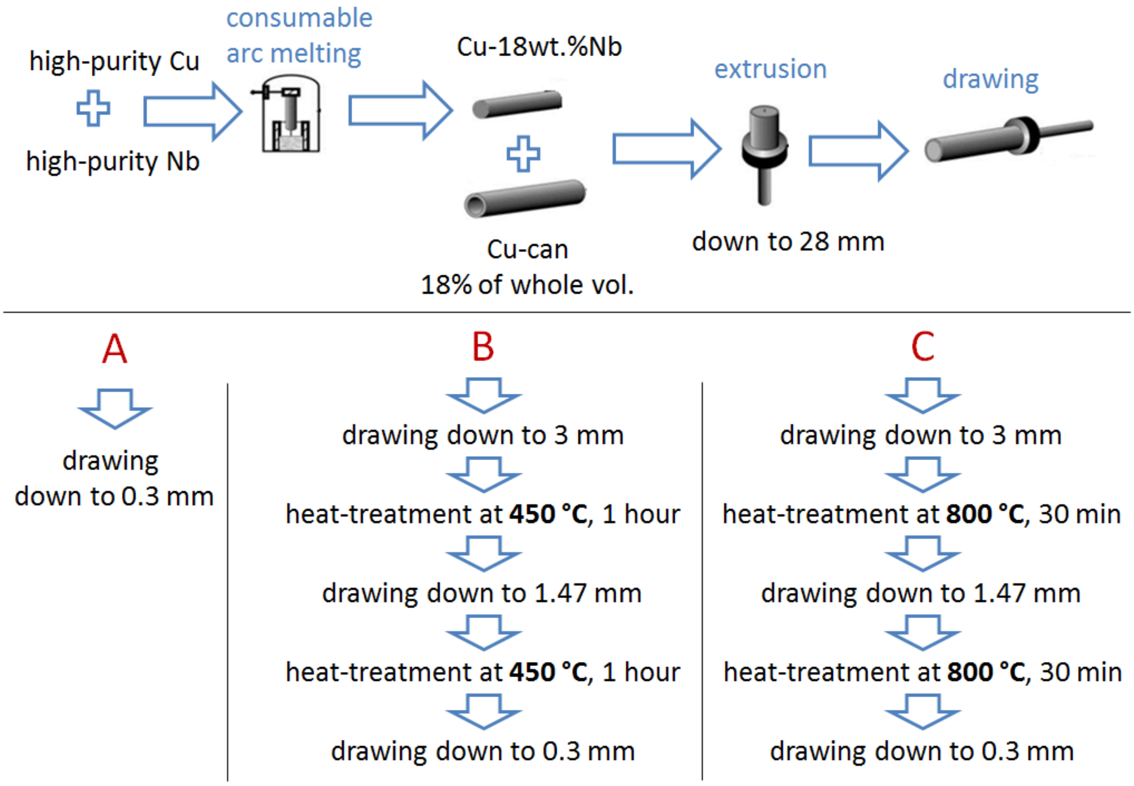


Fig. 1. Flow diagrams of manufacturing samples A, B and C

3. Results and calculations

Resistivity and filament spacing. The growth in the resistivity of Cu-Nb wires at high degrees of deformation ($\eta > 5$) is shown in Fig. 2 (at temperature 293 K) and in Fig. 3 (at temperature 77 K). During heat treatment of samples B and C (in the figures indicated by arrows), the resistivity decreases. The magnitude of this drop in resistivity is almost independent of the measurement temperature: the heat treatment at 450 °C/1 hour leads to the drop in the resistivity of about 0.03 $\mu\Omega \times$ cm both for room temperature and 77 K (Sample B), the heat treatment at 800 °C/30 min ~ 0.08 $\mu\Omega \times$ cm (Sample C). Such a significant decrease in resistivity could be attributed to the decrease of dislocations density in the copper channels, rearrangements of partial dislocations in the interphase Cu-Nb boundaries and to the changes in the size of the structural constituents due to the thermally activated coagulation process of Nb filaments [16,17]. Further, we will assume only the latter mechanism, keeping in mind that in determining the spacing after the heat treatments, this approximation gives an overestimated value.

Based on the measured values of the resistivity, assuming that the growth in the resistivity at $\eta>5$ is related only to the scattering processes at the Cu/Nb interfaces, it is possible to estimate the Nb-filament spacing. The algorithm for calculation of the growth in resistivity due to the size effect is described in detail in [14,15]. The numerical solution of the inverse problem allows us to calculate the filament spacing from the growth in resistivity. With this purpose, we used a number of fitting parameters presented in Table 1. For our calculations, the values of these parameters varied near the values known from the literature, so as to obtain the best agreement between the data obtained for room and liquid nitrogen temperatures.

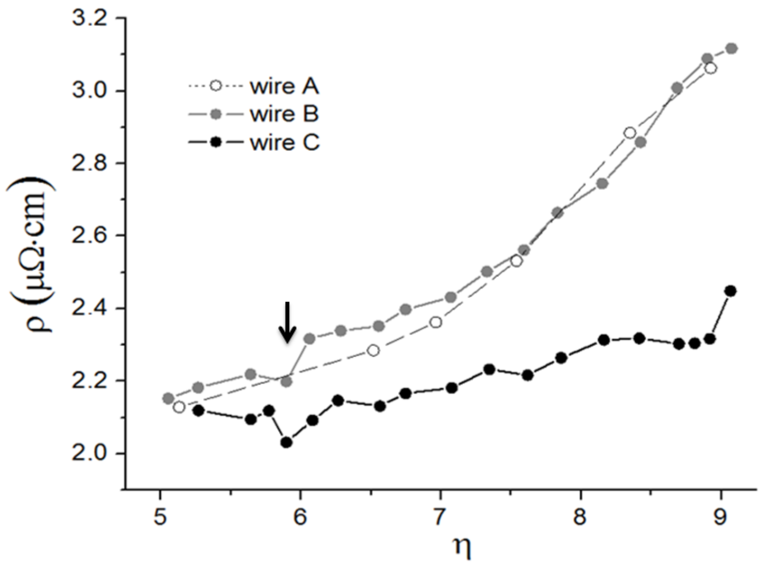


Fig. 2. Deformation dependence of resistivity at 293 K for different modes of thermo mechanical processing of Cu/Cu-18%wtNb wires. Arrow shows the second heat treatment of samples B (450 °C, 1 h) and C (800 °C, 30 min)

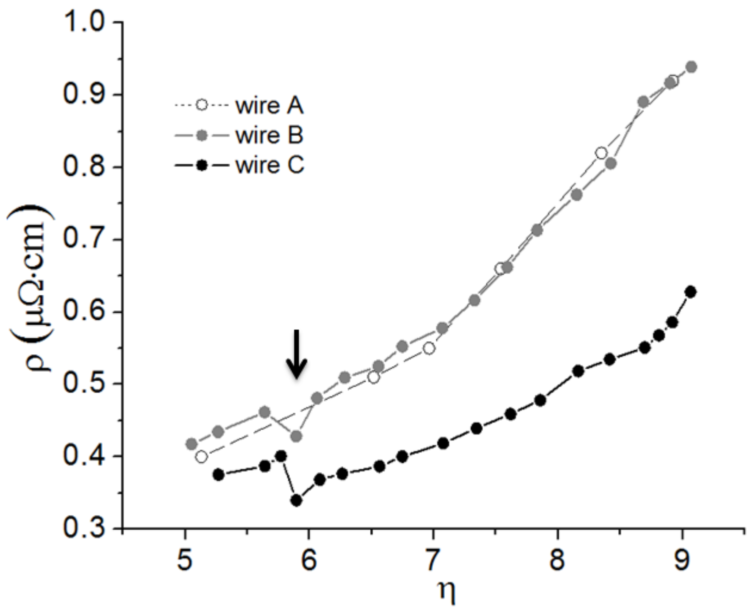


Fig. 3. Deformation dependence of resistivity at 77.4 K for different modes of thermo-mechanical processing of Cu/Cu-18%wtNb wires. Arrow shows the second heat treatment of samples B (450 °C, 1 h) and C (800 °C, 30 min)

According to [14,15], when the distance between the Nb-filaments is much larger than the mean free path of bulk Cu, the size effect is negligible. Therefore, at 293 K, correct determination of spacing for large diameters (and a small degree of deformation) is difficult. However, lowering the test temperature down to 77 K leads to an increase in the mean free path, and thus, the accuracy of the spacing estimation increases. On the other hand, it is well known that, at liquid nitrogen temperature, the nature of the scattering at the Cu / Nb interface changes, which leads to a difference in the scattering coefficient from 0 [13], and so it adds one more fitting parameter at low temperature (Table 1).

Table 1. The values of the fitting parameters for the d_{Cu} calculation: bulk resistivity at room temperature (ρ_0^{room}), temperature independent product of resistivity and mean free path $\rho_0 \times l_0$, resistance ratio at nitrogen temperature and at room temperature ($\rho_0^{77K}/\rho_0^{room}$) and the scattering parameter of the interface at 77 K in the Dingle's model (p)

	ρ_0^{room}, μΩ×cm	$\rho_0 \times l_0$, nΩ×mm^2	$\rho_0^{77K}/\rho_0^{room}$	p (at 77K)
Cu	1.69	0.66	7	0.1
Nb	13	0.92	4	0.1

The results of the d_{Cu} calculation are shown in Fig. 4. For comparison, the values of the spacing determined by the direct SEM method are also given. The obtained dependence of the exponential size reduction is in good agreement with the empirical dependence proposed in [18]. The results obtained from the numerical inversion of the size effect of resistivity give smaller, but close to direct SEM measurements, the spacing values. At the same time, it should be noted that the spacing estimation by resistivity is a fast integral method, while the determination of the average size of copper channels by the method of random secants, that requires collecting relevant statistics on several hundred local SEM images, is much more labor-intensive.

Figure 5 presents the results of a similar calculation of the Nb-filament spacing depending on the degree of deformation for samples B and C.

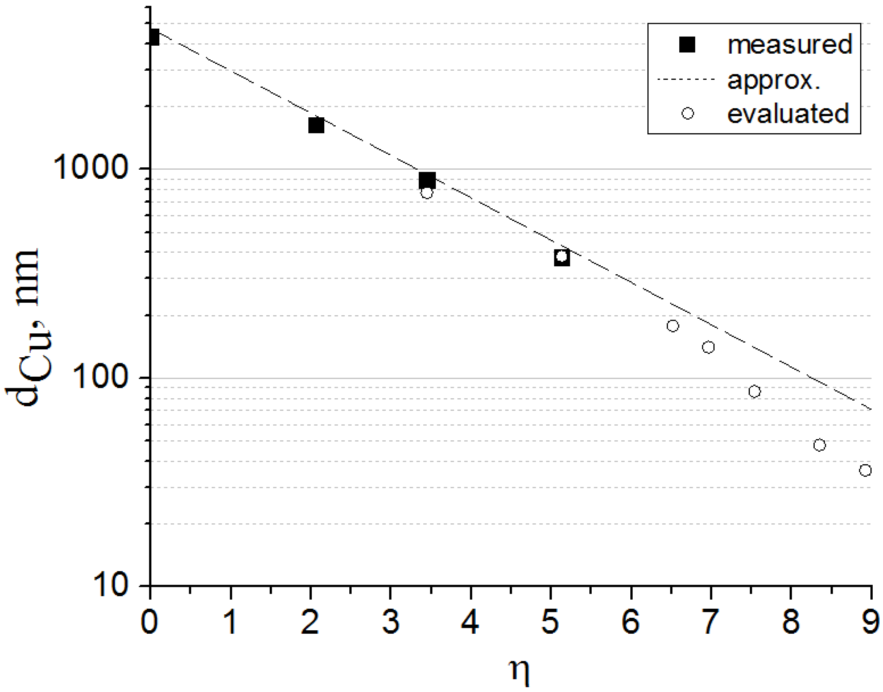


Fig. 4. Measured and calculated values of Nb-filament spacing d_{Cu} in Cu-18% Nb nanostructured wires, depending on the deformation degree for sample A

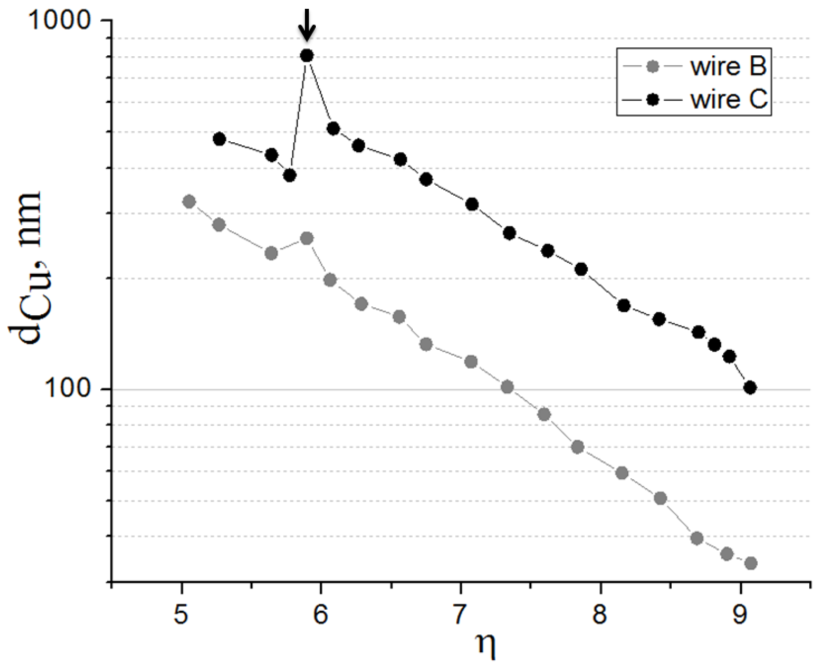


Fig. 5. The Nb-filament spacing, depending on the degree of deformation for wires B and C. The arrow shows the increase in spacing caused by heat treatment

Strength of Cu-Nb wires. The evolution of tensile strength of Cu-Nb composites at room temperature depending on the degree of accumulated drawing deformation for samples A, B, and C is shown in Fig. 6.

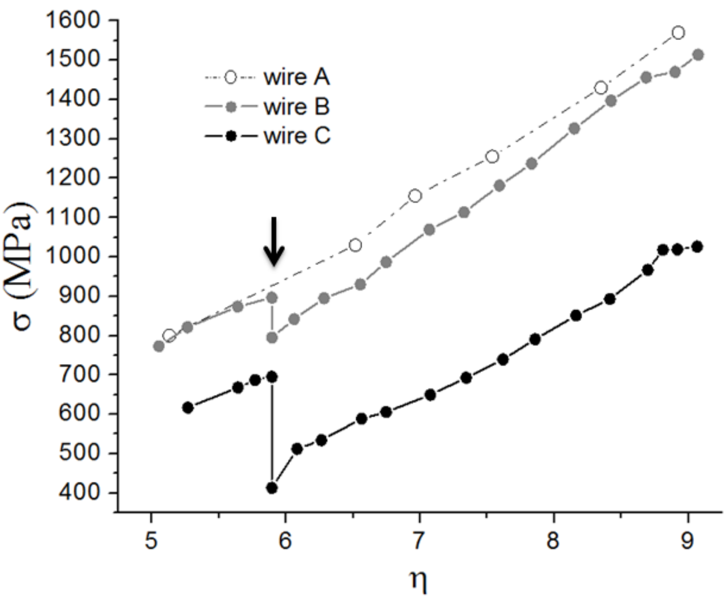


Fig. 6. Deformation dependence of tensile strength for samples A, B, and C with various heat treatment modes. Arrow shows the second heat treatment of samples B (450 °C, 1 h) and C (800 °C, 30 min)

A description of the strengthening effect of Cu-Nb composites with an increase in the degree of deformation is usually given in terms of the Embury-Hirth model or the Strain Gradient Theory.

Embury-Hirth model. It was shown [19] that the mechanical stresses, arising during drawing, lead to the appearance of dislocation loops in Cu, but they are not sufficient for generation of plastic deformation of Nb in the adjacent area of interphase surface and, therefore, the dislocation mechanism of plastic deformation in small thickness Nb filaments cannot be activated. The conglomeration of dislocation loops in Cu at the Cu/Nb interface leads to the accumulation of a uniform load on Nb. As a result, with some critical accumulation of the load, Nb is evenly deformed, taking the ribbon-like shape. Recently, the described discrete deformation process of Nb-filaments was confirmed by the stepwise increase in resistivity of Cu-Nb at liquid helium temperature [13].

In the Embury-Hirth (EH) model [20], this scheme of joint deformation of Cu and Nb is used to explain the anomalous strengthening of the Cu–Nb composites when the nano-sizes of the structural components are reached. Copper strength in this model is calculated as:

$$\sigma(\mathrm{Cu}) = \sigma_{\mathrm{Cu}} + M_{\mathrm{Cu}}\tau_{Orowan}(\mathrm{Cu}), \tag{1}$$

where $\sigma_{\mathrm{Cu}} = 450\ MPa$ – strength of cold worked copper, $M_{\mathrm{Cu}} = 3$ – the Taylor factor, τ_{Orowan} – dislocation shear stress in copper (Orowan stress):

$$\tau_{Orowan} = \frac{\mu_{\mathrm{Cu}} b_{\mathrm{Cu}}}{\pi(1-\upsilon_{\mathrm{Cu}})d_{\mathrm{Cu}}} \ln\left(\frac{\lambda}{2\pi b_{\mathrm{Cu}}}\right), \tag{2}$$

where μ_{Cu} – shear modulus of Cu (μ_{Cu}=45 GPa), b_{Cu} – the Burgers vector of Cu (b_{Cu}=0.26 nm), ν_{Cu} – Poisson ratio (ν_{Cu}=0.35), d_{Cu} – spacing , λ – mean inter dislocation distance on the Cu/Nb interface.

Due to the specific boundary conditions of elastically deformable Nb and plastically deformed Cu, niobium strengthening can be expressed through the characteristics of copper:

$$\sigma(\mathrm{Nb}) = \sigma_{\mathrm{Cu}} + M_{\mathrm{Cu}}\tau_{Orowan}(\mathrm{Cu}) + \frac{1}{X_{\mathrm{Cu}}} \cdot \frac{0.8 \cdot \mu_{\mathrm{Cu}} b_{\mathrm{Cu}}}{\lambda(1-\upsilon_{\mathrm{Cu}})}, \tag{3}$$

where $X_{\mathrm{Cu}} = 0.82$ – the volume fraction of copper in the Cu-Nb composite.

Thus, the strengthening of the entire Cu-Nb composite can be expressed through the characteristics of copper only:

$$\sigma_{\mathrm{Cu-Nb}} = X_{\mathrm{Nb}}\sigma(\mathrm{Nb}) + X_{\mathrm{Cu}}\sigma(\mathrm{Cu}) = \sigma_{\mathrm{Cu}} + M_{\mathrm{Cu}}\tau_{Orowan}(\mathrm{Cu}) + \frac{1-X_{\mathrm{Cu}}}{X_{\mathrm{Cu}}} \cdot \frac{0.8 \cdot \mu_{\mathrm{Cu}} b_{\mathrm{Cu}}}{\lambda(1-\upsilon_{\mathrm{Cu}})}. \tag{4}$$

According to [19] for Cu-17vol.% Nb at high degrees of deformation, the following empirical relation between d_{Cu} and λ can be offered:

$$\lambda = 0.294 \cdot d_{Cu} + 9.857, \tag{5}$$

where λ and d_{Cu} are measured in nm.

Strain gradient theory. In the strain gradient theory (SGT) each metal phase obeys Taylor's dislocation hardening law with an additional density term from the Cu/Nb interface [21,22]:

$$\sigma = \sigma_0 + M\alpha\mu b\sqrt{\rho_S + \rho_G}, \tag{6}$$

where σ_0 is the extrapolated yield strength at zero dislocation density, M – Taylor factor, α – a material constant ranging from 0.1 to 0.5, μ – is the shear modulus, b – is the Burgers vector length, ρ_S is the density of dislocations inside metal phases and ρ_G is the density dislocations on Cu/Nb interface.

The density of dislocations inside metal phases is determined as:

$$\rho_S = \left(\frac{(\sigma_Y - \sigma_0) f(\varepsilon)}{M\alpha C b}\right)^2, \tag{7}$$

where σ_Y is the yield strength without any strain hardening (ε = 0), $f(\varepsilon)$ is a function that can be obtained from uniaxial tension testing and chosen as $f(\varepsilon) = 1 + \varepsilon^n$, where n is the work hardening exponent for the metal.

It is assumed that the effective dislocation density on the Cu/Nb interface is proportional to the deformation true strain ε and inversely proportional to the characteristic length of the microstructure d:

$$\rho_G = k\frac{\varepsilon}{bd}, \tag{8}$$

where k - the material-dependent parameters introduced for metal phases.

Thus, Taylor's original law can be rewritten as:

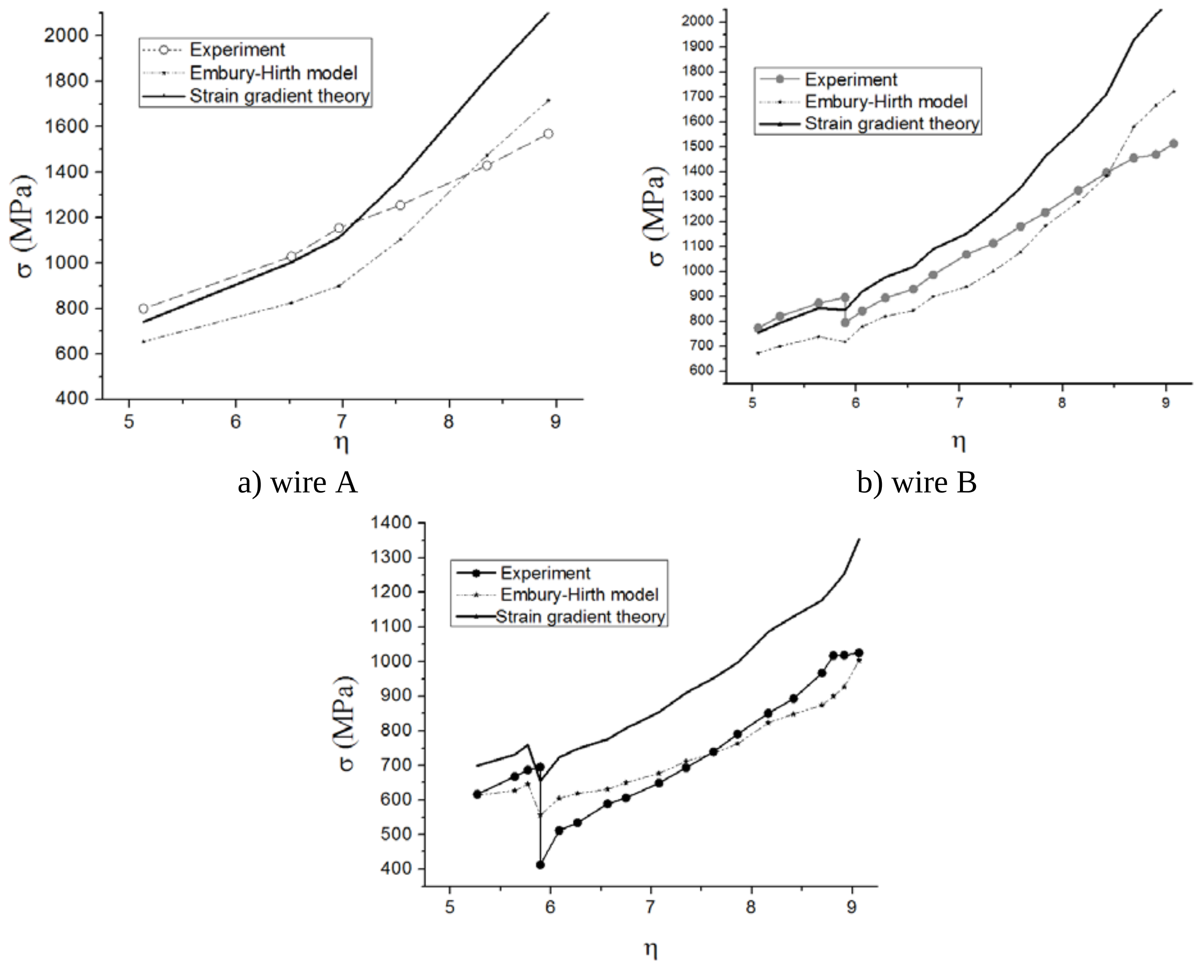
$$\sigma = \sigma_0 + (\sigma_Y - \sigma_0)\sqrt{f(\varepsilon)^2 + k\varepsilon\frac{l}{d}}, \tag{9}$$

where $l = M^2\alpha^2(\frac{\mu}{\sigma_Y - \sigma_0})^2 b$ is identified as the intrinsic material length scale [21].

Strength of the entire composite follows a modified rule of mixture relation:

$$\sigma_{\mathrm{Cu-Nb}} = X_{\mathrm{Cu}}\sigma(Cu) + X_{Nb}\sigma(\mathrm{Nb}). \tag{10}$$

Comparison with experiment. Figure 7 shows a comparison of the experimental dependences of tensile strength on the degree of accumulated deformation and calculated using Embury-Hirth model (EH) according (4) and the Strain Gradient Theory (SGT) (10). For these calculations, the values obtained from the size effect of resistivity (Figs. 4 and 5) were used as estimates of the Nb-filaments spacing.



a) wire A b) wire B

c) wire C

Fig. 7. Comparison of experimental data (from Fig. 6) and calculated using the Embury-Hirth model (dotted line) and strain gradient theory (solid line)

4. Discussions

The correlation between the behavior of electrical resistivity (Figs. 2 and 3) and strength (Fig. 6) is noteworthy. Indeed, from deformation 5 to 9, resistivity and strength are almost doubled. The heat treatments naturally lead to the drop of both resistivity and strength. The magnitude of the drop in both cases increases with increasing heat treatment temperature. Similar direct correlations between the deformation dependencies of strength and resistivity have been observed by many other authors (see for example [23]).

As it is shown above, the size effect of resistivity is a fairly well-developed theory that even allows for inverse calculations. There is a characteristic length – the electron mean free path, in comparison with which one can determine the intensity of the size effect of resistivity. When the temperature decreases from room temperature to 77 K, the mean free path increases from about 40 nm to about 270 nm. Thus, the size effect of the resistivity growth is more pronounced at liquid nitrogen temperature.

Although the strengthening mechanism is determined by completely different mechanisms than the increase in resistivity, the observed direct correlation may indicate that strengthening is also a size effect, as it is often assumed. Moreover, since the pronounced deviation from the mixture rule is observed when the deformation is greater than 5, the characteristic length of the size effect should be comparable to the size of the structural elements for this level of deformations. For Cu it is about 200 nm, for Nb ~ 40 nm.

In the Embury-Hirth model, parameter "λ" in (4) has the closest physical sense to a characteristic length. However, λ itself depends on the deformation (5), and therefore it cannot be attributed to an intrinsic material scale.

In the Strain Gradient Theory parameter "l" in (9) hinges only on the structure and properties of the metals. However estimates of this value give values for copper $l_{Cu} = 43\ \mu m$, for niobium $l_{Nb} = 47\ \mu m$ [22], i.e. several orders of magnitude higher than the characteristic sizes of the structure.

Thus, neither the Embury-Hirth model nor the Strain Gradient Theory gave a correct description of the size effect of strength. The discrepancy between the experimental data and the EH and SGT model predictions shown in Fig. 7 may be caused by an incorrect estimate of the sizes of the structural components, since for these estimations a rough approximation is used. Namely, the resistivity growth is completely determined by the decrease in size of components with constant scattering ability of the interface. However, the good agreement between the results of direct SEM measurements and the estimates obtained for sample A suggests that the error in determining the spacing cannot be so significant.

Apparently, for a correct description of the size effect of strength, it is necessary to take into account the peculiarities of the interface of metals with different types of crystallographic lattices (BCC - Nb and FCC - Cu) that are jointly deformed to high degrees. For example, it is known that the formation of a semi-coherent Cu-Nb phase boundary may be the cause of abnormal strengthening [9]. In addition, semi-coherent interface has violations in the form of sections of amorphized "islands" of the border. Such sections arise as a result of another mechanism of plastic deformation associated with the realization of rotational modes of plastic deformation, in which diffusion processes dominate over dislocation processes [24]. These areas are a several atomic layers thick mixture of Cu and Nb phases (a random mixture of Cu and Nb atoms) and their contribution to hardening is not entirely clear.

5. Conclusions

Investigations of strength and resistivity have been carried out on heavily deformed Cu-Nb nanostructured wires with different modes of thermo mechanical processing. It is shown that the dependences of resistivity and tensile strength on accumulated deformation have a direct correlation.

The transverse dimensions of the structural components in the Cu-Nb nanocomposite wire were investigated using the SEM method. It was found that the estimates of the spacing extracted from the experimental values of the growth in electrical resistivity due to the size effect are in good agreement with the results of direct SEM-measurements. This confirms the validity of the size effect model for describing the increase in resistance with deformation.

The application of the Embury-Hirth model and the Strain Gradient Theory for description of the strengthening of Cu-Nb nanocomposite wires has been analyzed. It is demonstrated that the model calculations do not correspond to the experimental data for the extremely high deformations. For a correct description of the experimental data in the range of high deformations, new models of strengthening of Cu-Nb composites are required. For new model the considering of the interface conditions, which characterized by the appearance of the amorphization regions in interphase boundaries may be proposed.

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