

W.J. GŁUCHOWSKI*, Z.M. RDZAWSKI**, J.P. STOBRAWA**, K.J. MARSZOWSKI*

MICROSTRUCTURE AND PROPERTIES OF Cu-Nb WIRE COMPOSITES

MIKROSTRUKTURA I WŁASNOŚCI DRUTÓW KOMPOZYTOWYCH Cu-Nb

Nowadays, there is much activity all over the world in development of Cu-Nb composites for their potential use as conductors in high field magnets. This study was aimed at investigation of microstructure, mechanical and electrical properties of Cu-Nb composite wires. The investigated materials have been processed by vacuum furnace melting and casting, and then hot forging and cold drawing. Initial results of research into Cu-Nb composite material obtained using repeated iterative drawing of niobium wires compacted into copper tube, have been also presented in this article. The ultimate tensile strength versus cold deformation degree has been presented. These changes have been discussed in relation to microstructure evolution. It was assumed that repeated drawing of compacted wires is a promising method for fibrous composite production (more than 823,000 Nb fibres of nanometric diameter) characterized by high mechanical properties and electrical conductivity. Original SPD technique applied for Cu-Nb composite deformation result in initial microstructure refinement and improves effectiveness of wire production process.

Keywords: fibrous composite, copper alloy, mechanical properties, electrical conductivity, microstructure

Aktualnie obserwuje się na świecie intensywny rozwój kompozytów Cu-Nb stosowanych jako przewody nawojowe generatorów silnych pól magnetycznych. Badania miały na celu określenie mikrostruktury oraz właściwości mechanicznych i elektrycznych drutów kompozytowych Cu-Nb. Badane materiały wytworzono przez zastosowanie topienia i odlewania w piecu próżniowym, a następnie kucia na gorąco i ciągnięcia. Zaprezentowano także wstępne wyniki badań wytwarzania kompozytu Cu-Nb na drodze iteracyjnego ciągnięcia pakietu drutów niobowych w rurze miedzianej. Pokazano wyniki badań wytrzymałości na rozciąganie w zależności od stopnia odkształcenia, w powiązaniu ze zmianami mikrostruktury. Stwierdzono, że wielokrotne ciągnięcie pakietu drutów jest obiecującą metodą wytwarzania kompozytów włóknistych (ponad 823000 włókien Nb o przekroju nanometrycznym) o wysokich właściwościach mechanicznych i konduktywności elektrycznej.

1. Introduction

Nanostructured materials with a combination of good electrical and thermal conductivity and also high mechanical strength are needed for winding wires used in production of high-field magnets. Cu-Nb composites are considered as candidate materials for the production of electrical transformers, strong electromagnets for metal transportation and equipment for plastic deformation by magnetic field.

Stronger than $\mu_0 H = 45$ T magnetic fields can be generated in a form of relatively short, irregular impulses. Magnetic flux compression can reach up to $B = 2800$ T, however in such a situation the magnet becomes destroyed after emission of a single impulse [1]. Currently studies are conducted into development of resistant magnets capable to generate magnetic flux of $B = 100$ T for 10 ms. High tensile strength is necessary to resist the Lorentz force while high electrical conductivity is needed to reduce Joule heat resulting from strong magnetic current [2-3]. The materials should also show high ductility

to provide possibilities for production of rectangular wires by drawing or profile rolling and also to prevent cracking during coil winding [2,4-5].

Mutual solubility in the solid state of Cu and Nb metals is negligible. Cu-Nb alloys after crystallization are composed of two pure metallic phases. During plastic working the diameter and distance between Nb dendrites significantly decreases to produce advantageous band microstructure and to stimulate increase of mechanical properties of the alloy. At the same time the high electrical conductivity of copper phase remains unchanged [6-8].

The high niobium melting point (significantly higher than copper melting point), and very low mutual solubility of those metals (Fig. 1) [9] bring difficulties in production of Cu-Nb winding wires.

One of techniques used in preparation of Cu-Nb micro-composite wires is melting and casting in vacuum furnace and further processing by multiple drawing and annealing [4,10].

* NON-FERROUS METALS INSTITUTE, SOWIŃSKIEGO 5 STR., 44-101 GLIWICE, POLAND

** INSTITUTE OF ENGINEERING MATERIALS AND BIOMATERIALS, SILESIA UNIVERSITY OF TECHNOLOGY, KONARSKIEGO 18A STR., 44-101 GLIWICE, POLAND

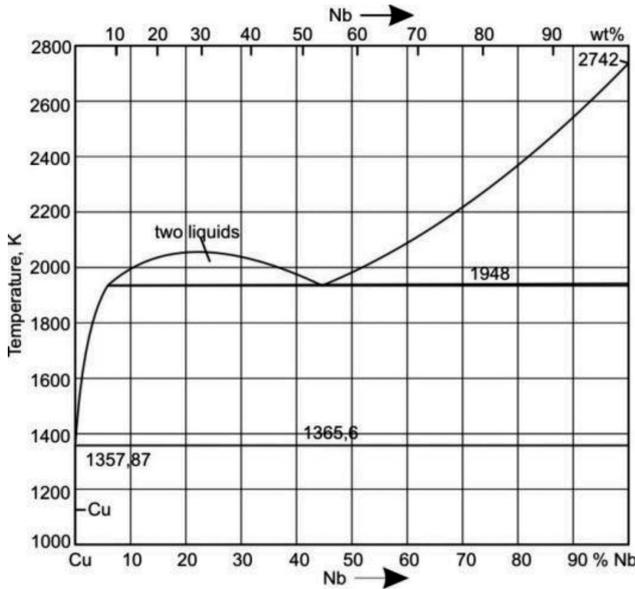


Fig. 1. Cu-Nb phase equilibrium system [7]

In the second method iterative repetition is applied and in the subsequent stages wires produced earlier from Cu-Nb microcomposite are introduced into the copper tube [10-12]. Melting of Cu-Nb alloys can also be performed in arc [13-15] or electron-beam furnace [16] and the produced billet can be processed by the described above methods. Annealing of Cu-Nb microcomposites can lead to spheroidization and growth of niobium fibers which has significant influence on mechanical and magnetic properties [17]. The next method for production of Cu-Nb microcomposite consists of mechanical alloying of powders and further plastic working and heat treatment of wires [1,18-19]. Powder metallurgy methods can also be used for production of microcomposites where metallic niobium is substituted by niobium carbide [20].

The study shows results of investigations into copper alloy with 20% niobium addition processed for production of a wire which meets the requirements for materials used in production of strong magnetic field generators. It was assumed that such wires should present tensile strength over 900 MPa and electrical conductivity over 40MS/m.

2. Methodology, material for studies

The study presents results of the examinations made with Cu-20%Nb alloy produced by classical vacuum melting and casting (Fig. 2). The produced ingots of 50 mm diameter were extruded by the press with reversibly rotating die (KOBO®) to the diameter of 6 mm or hot forged into a 10 mm rectangle rod. Further mechanical working was conducted by rolling and/or drawing.

As an alternative a fibrous Cu-Nb microcomposite was produced by multiple iterative drawing of a bundle of seven Nb wires in Cu tube. The wires with diameter of 2,5 mm obtained after drawing with intermediate heat treatment have been bundled and drawn again seven times. The processing according to this method is presented in Fig. 3. After seventh bundling operation niobium volume in the composite wire decreases from about 47 percent to about 12 percent.

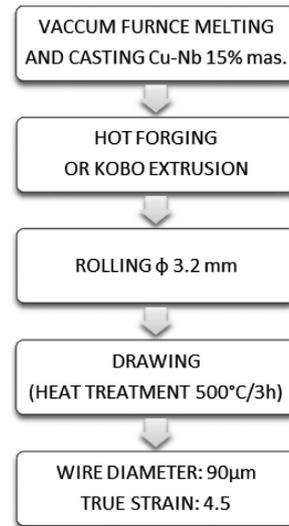


Fig. 2. Processing of CuNb microcomposites (classical method)

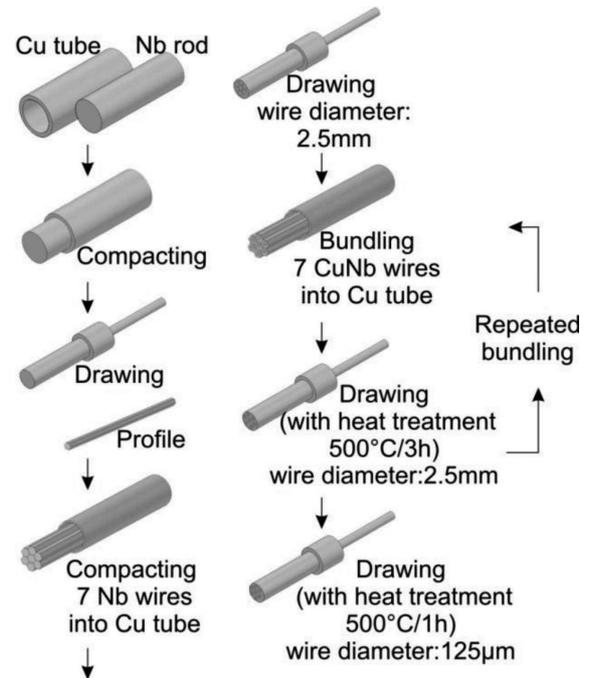


Fig. 3. Processing of CuNb microcomposites (bundling method)

Number of niobium fibres increased significantly (Table 1) so after seventh bundling a microcomposite with more than eight hundred thousand niobium fibres uniformly distributed in copper matrix was produced.

TABLE 1
Changes of niobium content and niobium fibres number during repeated bundling process

Bundling number	%Nb	Nb fibre number
1	47	7
2	36	49
3	30	343
4	24	2401
5	19	16807
6	15	117649
7	12	823543

Microstructure at individual stages of processing was examined by optical and electron scanning microscopy with EDS microanalysis. Examinations covered hardness, electrical conductivity and mechanical properties determined with testing machine.

3. Results and discussion

The microstructure after casting of vacuum melted copper-niobium alloy is presented in Fig. 4. In the copper matrix there are niobium rich dendrites. They are about several microns in size, there were also smaller particles with dimension about hundreds of nanometres placed in copper matrix grain boundaries.

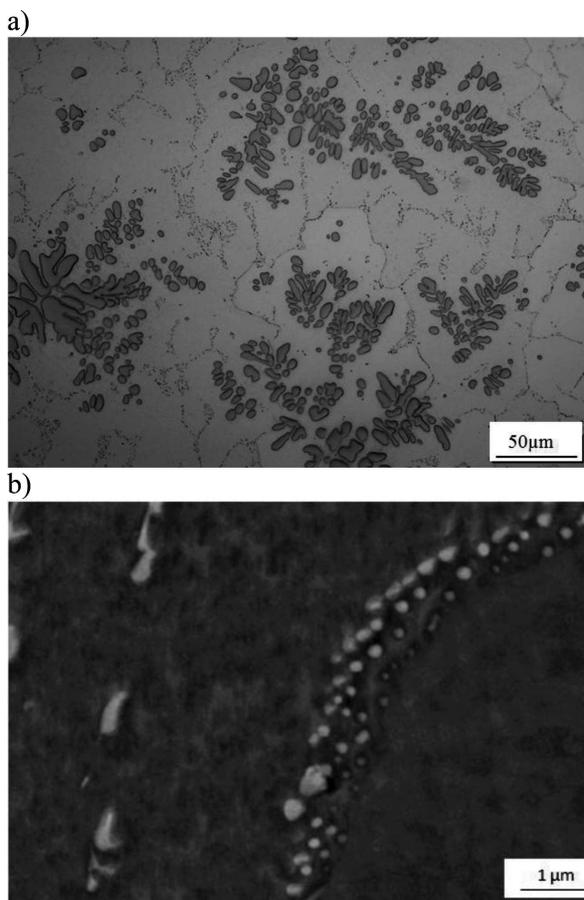


Fig. 4. Microstructure of CuNb ingot, a)optical microscopy, b) SEM

It is supposed that these particles are also of niobium rich phase. The further hot forging, rolling and drawing strongly influenced the initial microstructure. During this processing the niobium rich particles were refined and the microstructure was oriented according to the deformation direction (Fig. 5).

Finally a wire with 90 microns in diameter was produced. The true strain calculated from last annealing operation was about 4,5. The microstructure wasn't uniform. There were a thin nanometric bands of niobium rich phase and there were also bigger particles of several microns (Fig. 6). The obtained wire had tensile strength of about 900 MPa and electrical conductivity of about 44 MS/m.

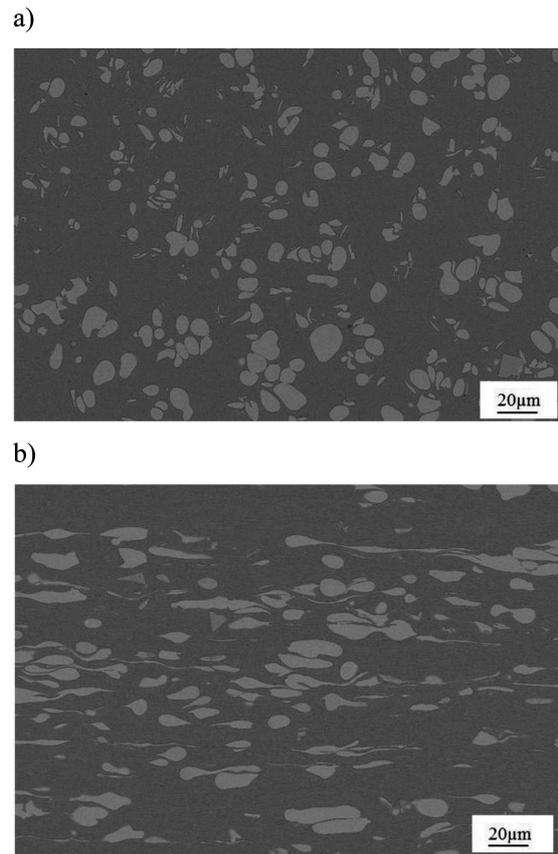


Fig. 5. Microstructure of CuNb wire after rolling and drawing $\varphi 2,8$ mm, a) cross section, b) longitudinal section

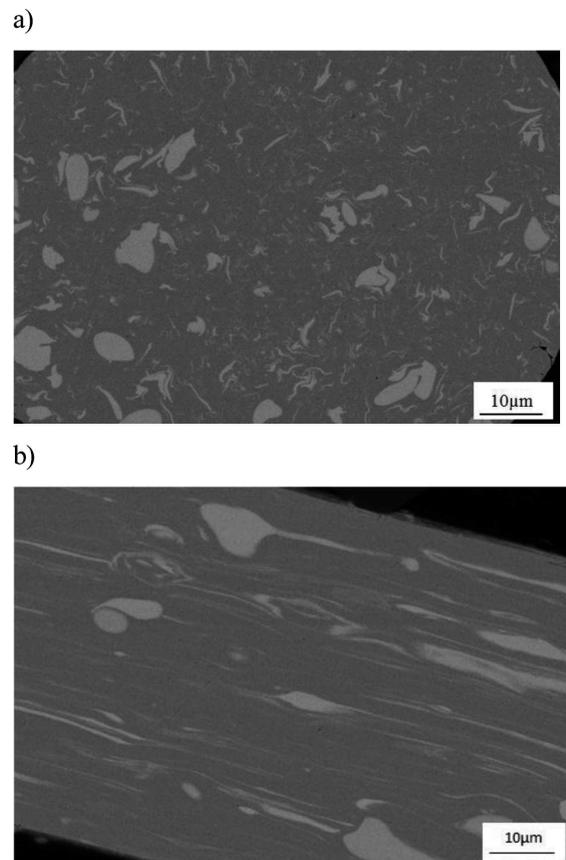


Fig. 6. Microstructure of CuNb wire after rolling and drawing $\varphi 90 \mu\text{m}$, a) cross section, b) longitudinal section

Another method for copper niobium microcomposite production is based on repetitive drawing and bundling process. We have bundled seven niobium wires into copper tube and then drawn it with intermediate annealing process to the wire 2,5 millimeters in diameter. These wires have been bundled and drawn again several times. Crosssections from the subsequent selected bundling operations are presented in Fig. 7. The microcomposite contained forty nine fibres (Fig. 7a) after second bundling and two thousand four hundred niobium fibres after fourth bundling operation (Fig. 7b).

Finally after seventh bundling the wire 2,5 mm in diameter with over eight hundred thousand niobium fibres was produced (Fig. 8).

The fibres were about one micron in diameter and grain size was about 100-200 nm (Fig. 8c). Copper matrix grain size was about 500 nm (Fig. 8d).

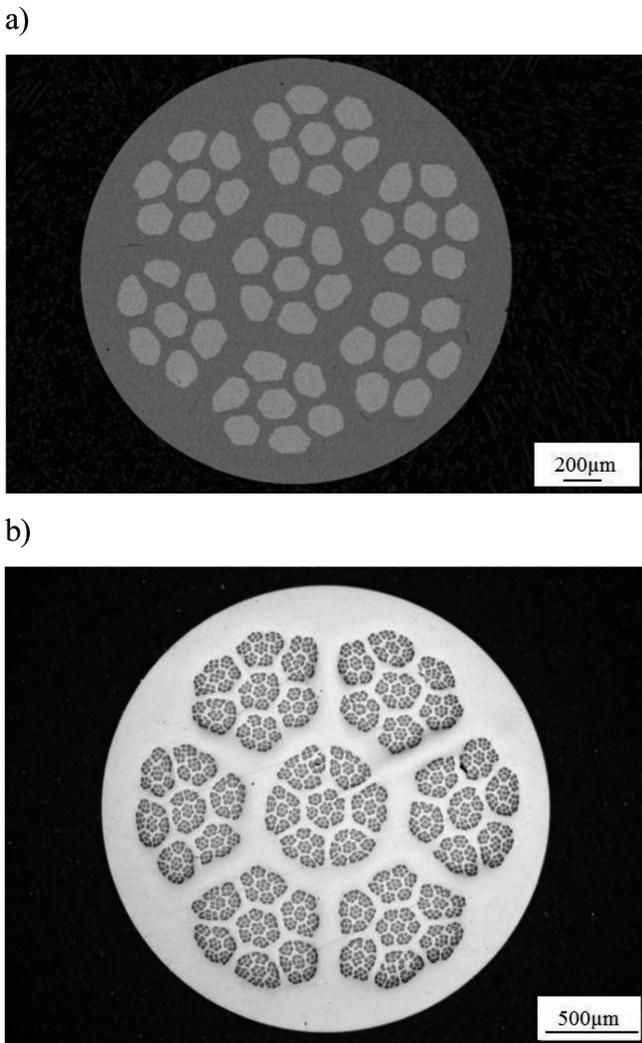


Fig. 7. Microstructure of CuNb composite wire, a) 2nd bundling, b) 4th bundling

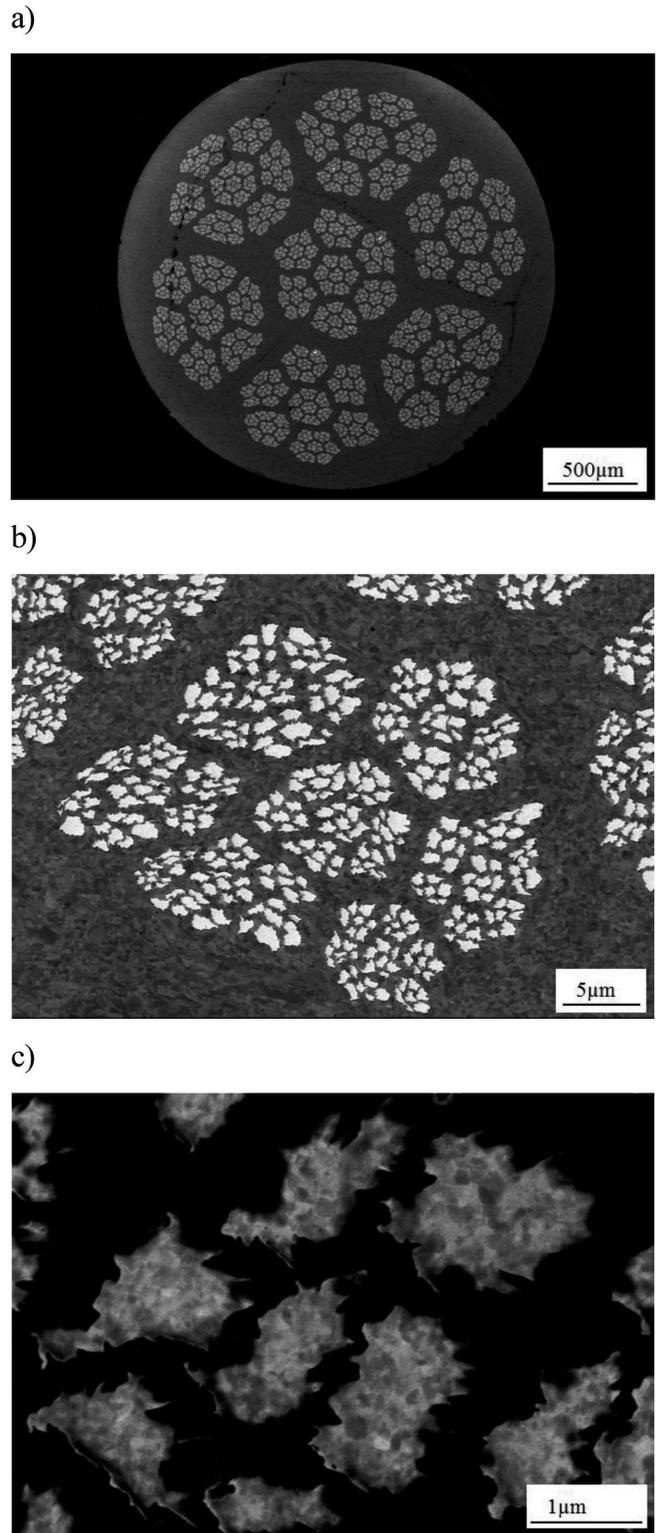


Fig. 8. Microstructure of CuNb composite wire after 7th bundling, magnification: a) 35x, b) 3 000x, c) 22 000x

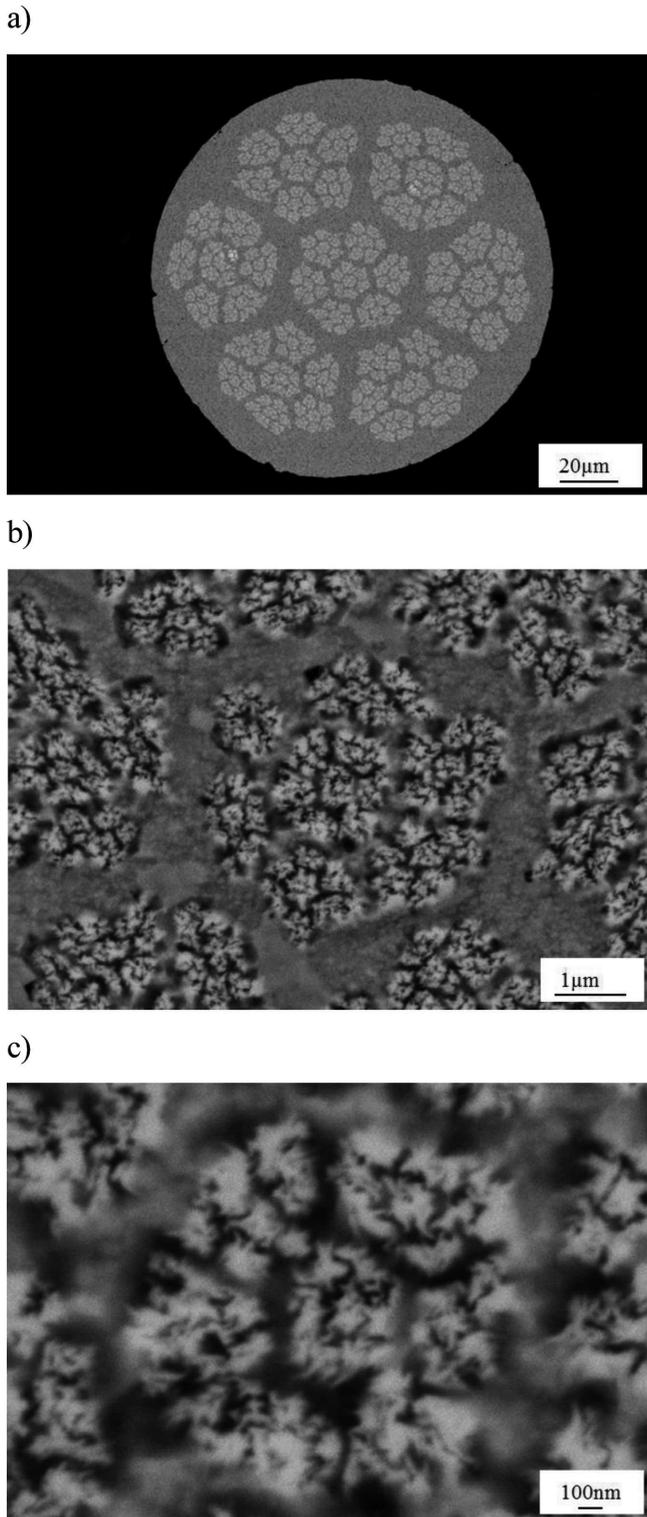


Fig. 9. Microstructure of CuNb composite wire after 7th bundling, diameter 125 μm , magnification: a) 1 300x, b) 30 000x, c) 100 000x

This wire has been drawn to the diameter of 125 microns (Fig. 9). After this huge deformation the niobium fibre diameter was about 50-100 nm. During processing the annealing temperature wasn't higher than about 500 Celsius degrees. It is supposed that niobium wires with initial diameter 2 mm have been deformed to the diameter 50-100 nm without recrystallization process, which gives a true strain of about 20. The wire had ultimate tensile strength 700 MPa. Electrical conductivity increased during repeated bundling and drawing

process from about 38MS/m to about 54 MS/m after seventh bundling (Fig. 10).

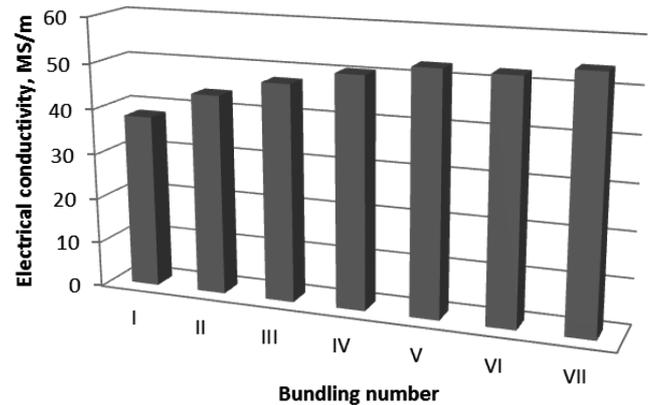


Fig. 10. Increase of electrical conductivity of composite Cu-Nb wires during repeated bundling and drawing process

4. Conclusions

The copper-niobium microcomposites were produced by classical method (including melting in vacuum furnace and cold plastic working) and by repeated bundling and drawing method. The wires produced by both processing methods met the requirements of coil wires for strong magnetic fields generators. They had high conductivity in the range of 44-54 MS/m, mainly because of the pure copper matrix. The second phase was composed of niobium rich particles (classical method) or long niobium fibres (bundling method). The niobium rich particles in wires obtained classically were of two types. There were bigger particles of globular shape and size of several microns and thin nanometric fibres of the length up to hundreds of microns. The niobium fibres in wires produced by bundling method were continuous and evenly distributed in the wire composite. Diameter of these fibres was in the range of 50-100 nm. This microstructure consisted of over 820000 continuous niobium fibres and was obtained after huge deformation with true strain of about 20. Strong refinement of the fibres cause change of mechanical properties to ultimate tensile strength of about 900 MPa for classically obtained wires and about 700 MPa for the bundled wires. This property can be improved by production of the material with fine microstructure where the second phase particle will be of very thin fibre of several nanometer in diameter.

To reach this microstructure some intensification of the processing can be considered, for example by extrusion of the wire bundle by the press with reversibly rotating die (KOB0). There is a need to define in details process conditions to reach complete plastic consolidation of the extruded material.

It is also expected that thermal stability of mechanical properties of copper niobium microcomposites will be increased because of high melting point of the niobium phase.

Acknowledgements

This work was supported by the European Union, Structural Funds Operational Program Innovative Economy – Project Number POIG.01.03.01-00-086/09.

REFERENCES

- [1] E. Botcharova, J. Freudenberger, A. Gaganov, K. Khlopkov, L. Schultz, *Mat. Sci. Eng.* **A416**, 261-268 (2006).
- [2] J. Embury, K. Han, *Solid State & Materials Science* **3**, 304-308 (1998).
- [3] J. Liu, L. Meng, Y. Zeng, *Mat. Sci. Eng.* **A435-436**, 237-244 (2006).
- [4] F. Heringhaus, D. Raabe, G. Gottstein, *Acta Metall. Mater.* **43**, 4, 1467-1476 (1995).
- [5] L. Murr, R. Flores, *Scripta Material.* **39**, 4-5 *Scripta Materialia* (1998).
- [6] W. Grunberger, M. Heilmaier, L. Schultz, *Zeitschrift für Metallkunde* **93**, 58-65 (2002).
- [7] J. Stobrawa, Z. Rdzawski, W. Głuchowski, W. Malec, *Archives of Metallurgy and Materials* **56**, 1, 171-179 (2011).
- [8] M. Woch, Z.M. Rdzawski, L. Kubica, *Archives of Metallurgy and Materials* **53**, 1, 295-299 (2008).
- [9] H. Okamoto, *J. Phase Equilibria* **12**, 614 (1991).
- [10] K. Han, J.D. Embury, J.R. Sims, L.J. Campbell, H.J. Schneider-Muntau, V.I. Patsyrny, A. Shikov, A. Nikulin, A. Vorobieva, *Mater. Sci. Eng.* **A267**, 99-114 (1999).
- [11] Y. Leprince-Wang, K. Han, Y. Huang, K. Yu-Zhang, *Mat. Sci. Eng.* **A351**, 214-223 (2003).
- [12] L. Thilly, M. Veron, O. Ludwig, F. Lecourterier, *Mat. Sci. Eng.* **A309-310**, 510-513 (2001).
- [13] J. Chung, J. Song, S. Hong, *J. Mat. Proc. Tech.* **113**, 604-609 (2001).
- [14] S. Hong, M. Hill, *Scripta Materialia* **44**, 2509-2515 (2001).
- [15] S. Hong, M. Hill, *Scripta Materialia* **42**, 737-742 (2000).
- [16] A. Shikov, V. Patsyrnyj, A. Vorobieva, N. Khlebova, A. Silaev, *Physica C* **354**, 410-414 (2001).
- [17] M.J.R. Sandim, C.A.M. dos Santos, H.R.Z. Sandim, L. Ghivelder, *Physica C* **408-410**, 207-208 (2004).
- [18] M. Dadras, D. Morris, *Scripta Materialia* **38**, 2, 199-205 (1998).
- [19] B.Z. Cui, Y. Xin, K. Han, *Scripta Materialia* **56**, 879-882 (2007).
- [20] M. Marques, V. Livramento, J. Correia, A. Almeida, R. Villar, *Mat. Sci. Eng.* **A399**, 382-386 (2005).

Received: 17 February 2013.