Journal of Achievements in Materials and Manufacturing Engineering
VOLUME 46
ISSUE 1
May
2011

Microstructural characterization of high strength high conductivity Cu-Nb microcomposite wires

W. Głuchowski [a], J.P. Stobrawa [a,b], Z.M. Rdzawski [a,b,*], K. Marszowski [a]
[a] Non-Ferrous Metals Institute, ul. Sowińskiego 5, 44-100 Gliwice, Poland
[b] Institute of Engineering Materials and Biomaterials, Silesian University of Technology, ul. Konarskiego 18a, 44-100 Gliwice, Poland
* Corresponding author: E-mail address: zbigniew.rdzawski@polsl.pl

Received 12.02.2011; published in revised form 01.05.2011

Properties

ABSTRACT

Purpose: The properties and the microstructure of cold drown Cu-Nb composites have been investigated for their potential use as conductors in high field magnets. Nowadays, there is much activity in the development of such conductors all over the world.
Design/methodology/approach: This study was aimed to investigate microstructure, mechanical and electrical properties of Cu-Nb15 wires. The investigated materials have been processed by vacuum furnace melting and casting, further hot forging and cold drawing. Alternatively material has been processed by one of the SPD (severe plastic deformation) method using oscillatory turning die pressing. Microstructure has been observed by optical and electron microscopy technics.
Findings: The ultimate tensile strength versus cold deformation degree have been presented. These changes have been discussed in relation to the microstructure evolution.
Practical implications: The obtained mechanical and electrical properties (UTS over 900 MPa and electrical conductivity over 40 MS/m) correspond to requirements for production of long pulsed 60T magnets.
Originality/value: Original SPD technic applied for Cu-Nb microcomposite deformation cause initial microstructure refinement and improves effectiveness of wire production process.
Keywords: Metallic alloys; Mechanical properties; Electron microscopy; Electrical properties

Reference to this paper should be given in the following way:
W. Głuchowski, J.P. Stobrawa, Z.M. Rdzawski, K. Marszowski, Microstructural characterization of high strength high conductivity Cu-Nb microcomposite wires, Journal of Achievements in Materials and Manufacturing Engineering 46/1 (2011) 40-49.

1. Introduction

The The materials for magnet winding wires used in generation of strong magnetic fields (operating in the range of 5-100 T) are required to present high strength and high electrical conductivity [1-3]. Stronger than µ0H=45 T magnetic fields can be generated in a form of relatively short, irregular impulses.

There is a possibility for magnetic flux compression to reach up to B=2800 T, however in such a situation the magnet becomes destroyed after emission of a single impulse [4]. Currently studies are conducted into development of resistant magnets able to generate magnetic flux of B=100T for 10 ms. High strength is necessary to resist Lorentz force while high electrical conductivity is needed to minimize Joule heat resulting from strong magnetic current [3,5]. The materials should also show high plasticity to provide possibilities for production of rectangular wires by drawing or profile rolling. High plasticity also prevents cracking of the material during coil winding [3,6-7]. The materials are used in production of electrical transformers, strong electromagnets for metal transportation and equipment for plastic deformation by electric field.

High mechanical properties and high electrical conductivity is observed in copper or silver based materials hardened with fine particles of other phase [8-12]. Mechanical working is a very good method for production of highly fragmentized microstructure of matrix and hardening particles [13-19].

The attractive properties for those applications are ultimate tensile strength over 750 MPa, at electrical conductivity above 65% IACS. Properties of the up till now used alloys, such as Cu-Be, Cu-Cr, Cu-Al_2O_3 and Cu-0.2%Ag make further development of techniques for generation of strong magnetic fields impossible. Cu-Be alloys show too low electrical conductivity while Cu-Cr, Cu-Al_2O_3 and Cu-0.2%Ag alloys present too low tensile strength [20].

There are great expectations related to application of Cu-Nb materials of fibrous composite microstructure. According to the Cu-Nb phase system, during crystallization of the alloys the monotectic reaction is dominating, and niobium dendrites form in copper enriched liquid. Mutual solubility of those metals is insignificantly small. Therefore it can be assumed that Cu-Nb alloys after crystallization are composed of two pure metal 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 [21].

Difficulties in production of Cu-Nb winding wires results mainly from high niobium melting point, significantly higher than copper melting point, and very low mutual solubility of those elements. Phase equilibrium system is presented in Fig. 1 [22]. The maximum solubility of niobium in copper in solid state is about 1.3 wt% in temperature of 1365K (Fig. 2 [23]). During crystallization of Cu-Nb alloys, which are produced from exceptionally pure components, niobium dendrites in copper matrix are observed in the microstructure, and when there are oxide impurities in the alloys of niobium content 5-35% in the microstructure niobium dendrites are observed, as well as Nb rich spheroids distributed in copper matrix [24].

The most popular methods for production of Cu – Nb microcomposites cover classical melting and casting in vacuum furnace and further processing by multiple drawing and annealing [2,6] and by introduction of a bundle of niobium wires into a copper tube and multiple drawing and annealing. 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 [2,25-26]. Melting of Cu-Nb alloys can also be performed in arc [27-29] or electron-beam furnace [30] 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 change of mechanical and magnetic properties [31]. The next method for production of Cu-Nb microcomposite consists of mechanical alloying of powders and further plastic working and heat treatment of wires [4,32,33].

Powder metallurgy methods can also be used for production of microcomposites where metallic niobium is substituted by niobium carbide [34]. Fig. 3 shows simulated relation between cold deformation and yield point in Cu-Nb alloys for various niobium contents [35], the values are close to the simulated in the study [36] for the Cu 20% Nb alloy and comparable to the experimental data (Fig. 4).

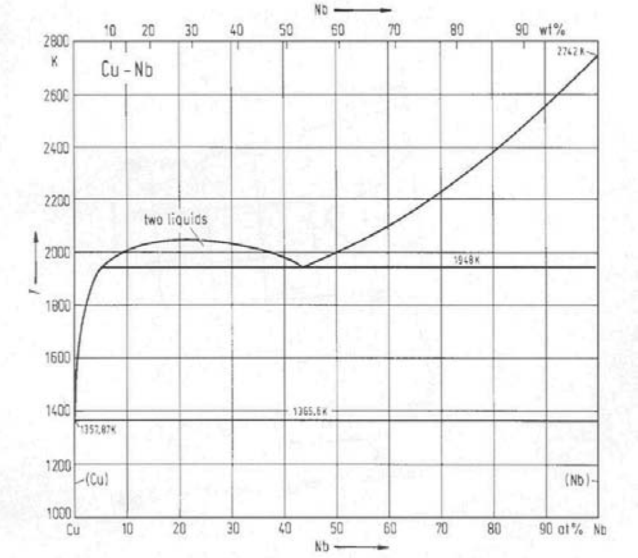


Fig. 1. Cu-Nb phase equilibrium system [22]

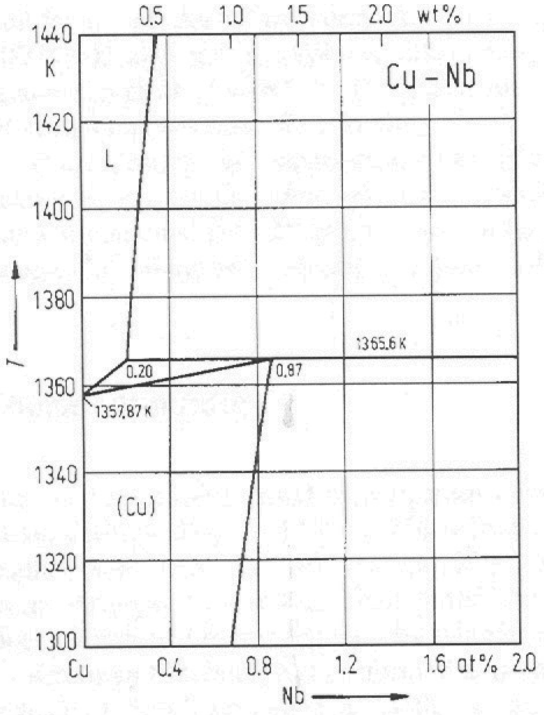


Fig. 2. Part of Cu-Nb phase equilibrium from copper side [23]

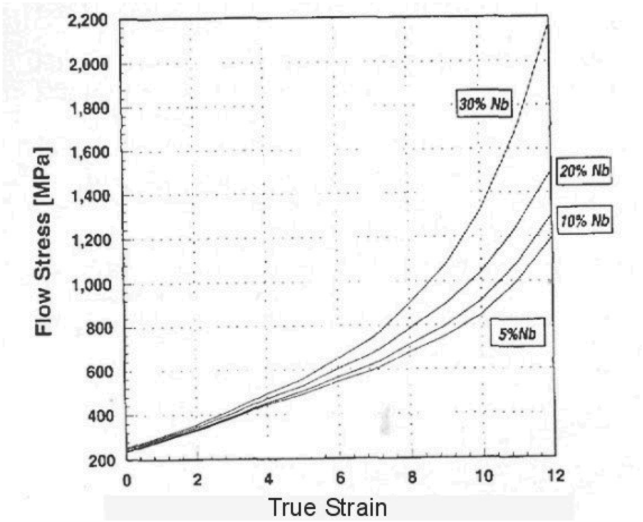


Fig. 3. Simulated relation between cold deformation and yield point in Cu-Nb alloys [37]

The study shows results of investigations into copper alloy with 20% niobium addition which was 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 40 MS/m.

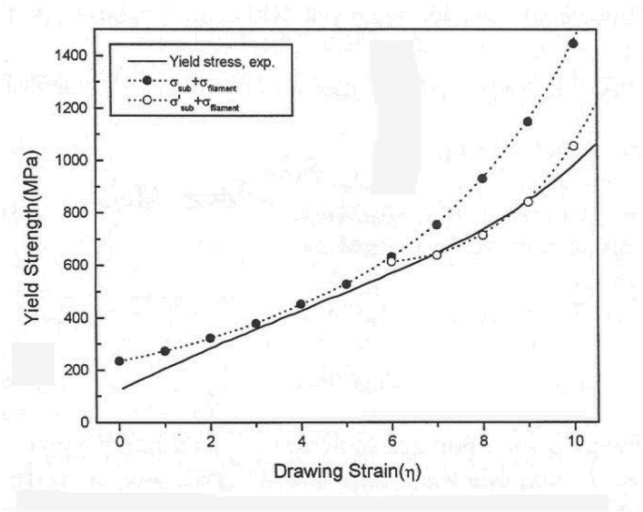


Fig. 4. Simulated relation between cold deformation and yield point in Cu-Nb alloys compared to experimental data [38]

2. Material and methodology

The study present results of the examinations made with Cu-20%Nb alloy produced by classical melting and casting. 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. A strand of wires produced from hot forged rod was extruded by KOBO press to a rod of 10mm diameter. Compression test was performed on rolls of dimensions φ10x15 mm made from the alloys after casting, after hot forging and after extrusion of strand of wires by KOBO press. As an alternative also a fibrous Cu-Nb microcomposite was produced by multiple iterative drawing of a bundle of Nb wires in Cu tube.

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

3. Results

Microstructure of Cu-Nb alloy presented typical character of the billets made by static casting (Fig. 5). Niobium (in which about 3-4% of copper was dissolved) crystallized in a form of dendrites of grain size at the level of several micrometers. Pure copper was used as matrix. Also second phase fine particles of dimensions at the level of 100 nm were observed on the borders of matrix grains (Fig. 6). Their small size did not allow their identification by EDS technique. It can be expected that they also represented niobium particles.

Microstructure of hot forged alloy when compared to the after casting state was less uniform, the size of niobium (in which about 3-4% of copper was dissolved) particles still remained at the level of several micrometers (Fig. 7).

The microstructure after extrusion by KOBO press presented similar picture. A part of niobium particles was distributed in bands along the direction of extrusion (Fig. 8).

The hot forged wire was drawn down to the diameter of φ2 mm and bundled into a hollow copper cylinder of diameter φ50 mm and wall thickness of 3 mm (Fig. 9).

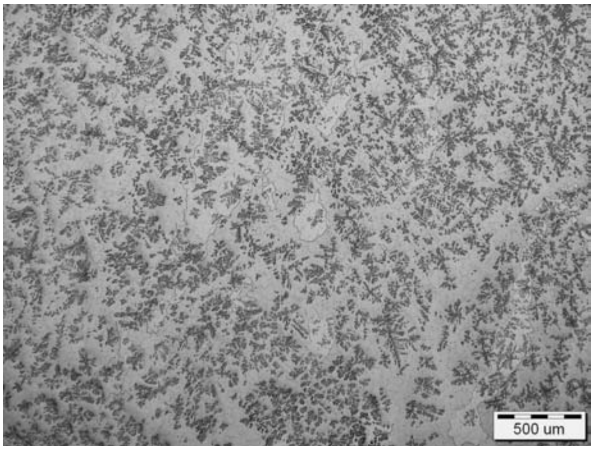
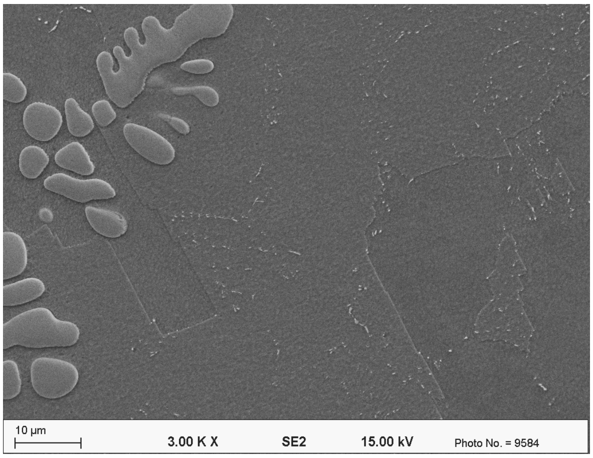


Fig. 5. Microstructure of CuNb alloy after casting, optical microscope

a) Magnif. 3000x etched microsection



b) Magnif. 10000x polished microsection

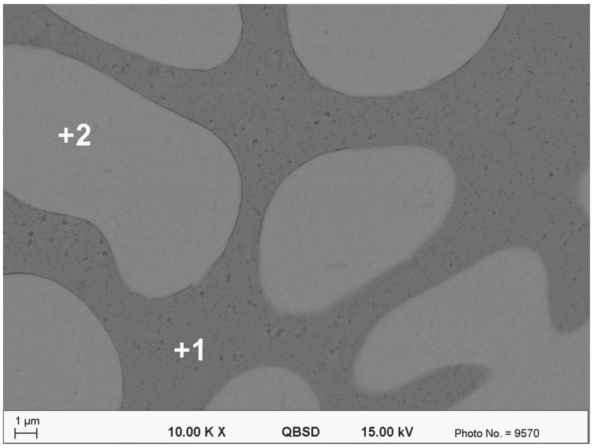


Fig. 6. Microstructure of CuNb alloy after casting, SEM with results of EDS analysis in microsections: 1:Cu=100, 2:Nb=96.2% Cu=3.8%

The bundle was annealed in temperature of 500^{o}C for 15 minutes and extruded by KOBO press into a wire of diameter 10 mm. During the process complete integration of CuNb wires with copper wall took place (Fig. 10) while the connections between the individual wires in the bundle were only partial (Fig. 11a).

The originally rectangle wires after extrusion took approximately hexagonal cross-section. The particles of rich in niobium phase became slightly deformed and also changed their shape in the result of complex state of stress during extrusion (Fig. 11b).

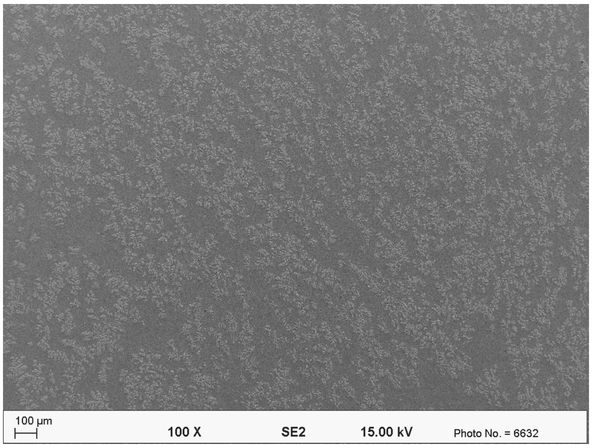
To determine deformability of the examined materials in elevated temperature compression test were condensed on testing machine in room temperature (20^{o}C) and in temperatures of 300 and 500^{o}C.

It was established that during compression of the alloy, which was melted and cast in vacuum furnace, copper matrix grains became deformed while particles of the rich in niobium phase were not significantly different to the ones observed in the material after casting. Microstructure of the alloy which was deformed in 300^{o}C was similar, only growth of copper matrix grains was observed. When the material was compressed in temperature of 500^{o}C it was found out that there was a change in arrangement of particles of rich in niobium phase (when compared to the original dendritic structure, also dynamic recrystallization of alloy matrix was observed (Fig. 12).

Microstructure of the cast alloy and the hot forged one did not differ significantly during compression at various temperatures. It was only for the highest value (500^{o}C) when the copper matrix microstructure was more coarse grained (Fig. 13).

Microstructure of the bundled and extruded by KOBO press materials which were then compressed on the testing machine was similar, regardless of the temperature (Fig. 14). Lack of full connection of the material during extrusion resulted in local loss of cohesion during the compression thus showing imperfectness of the material (Fig. 15). Process of extrusion of the wire bundle, although promising, needs improvement - increase of plastic working degree and process temperature - to produce complete plastic consolidation of the material.

a) Magnif. 100x etched microsection



b) Magnif. 1000x etched microsection

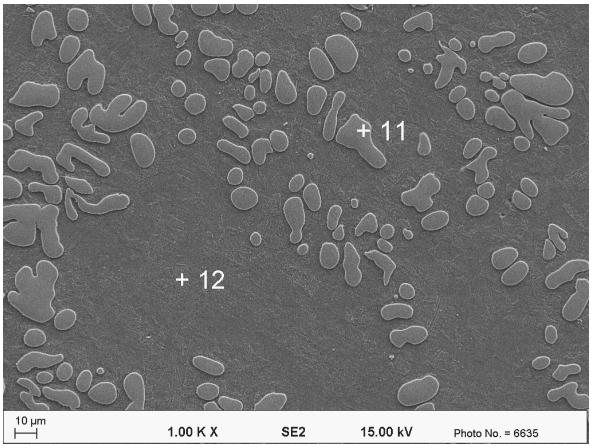
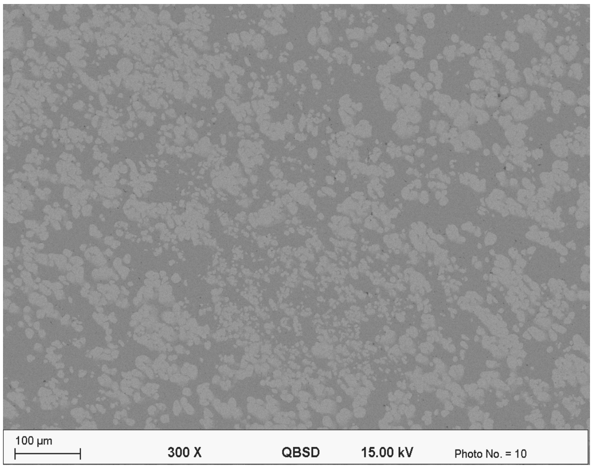


Fig. 7. Microstructure of CuNb alloy after casting and hot forging, SEM with results of EDS analysis in microsections: 11: Nb=96.9 Cu=3.1, 12: Cu=100

a) crossection



b) longitudinal section

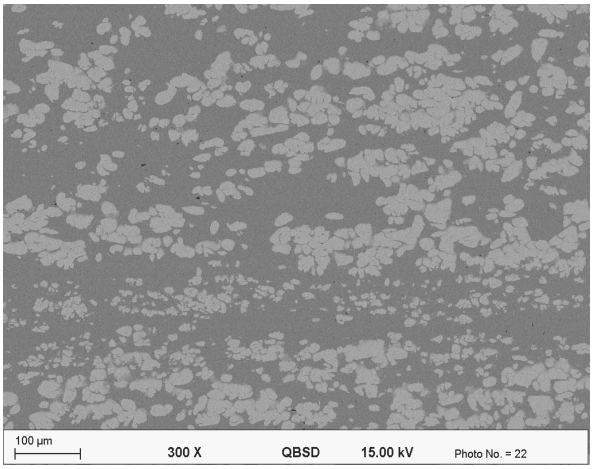


Fig. 8. Microstructure of CuNb alloy after casting and extrusion by KOBO press, SEM

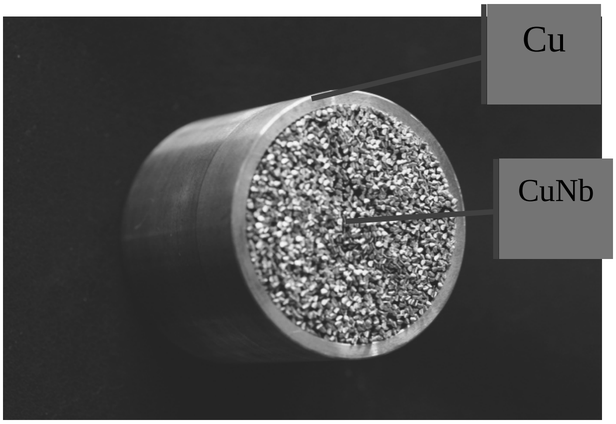


Fig. 9. Bundle of CuNb wires in a hollow copper cylinder of diameter 50 mm

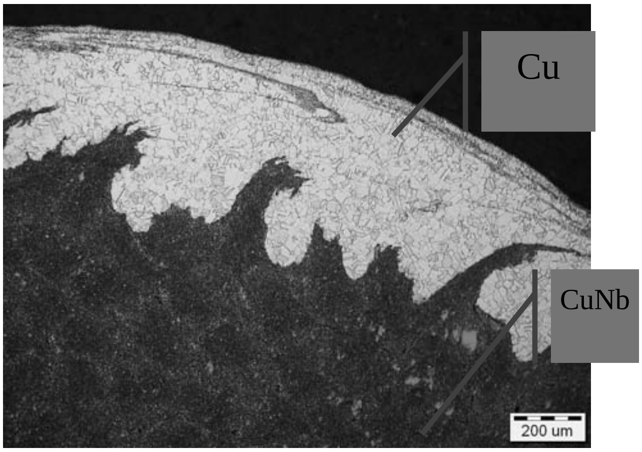
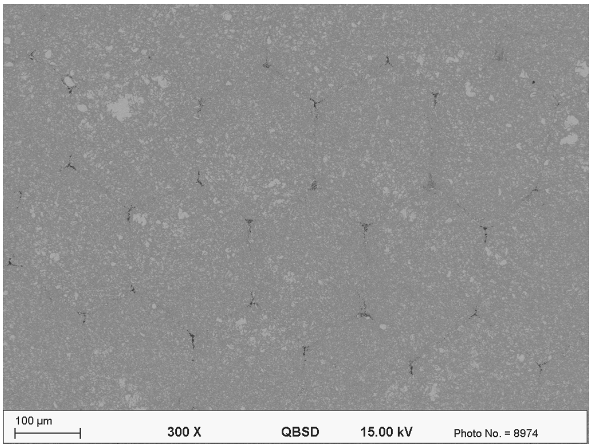


Fig. 10. Cross-section of the wire after bundle extrusion, area close to the surface

a) Magnif. 300x



b) Magnif. 3000x

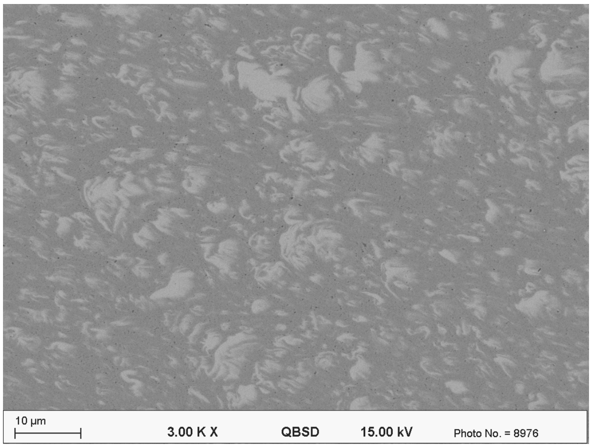
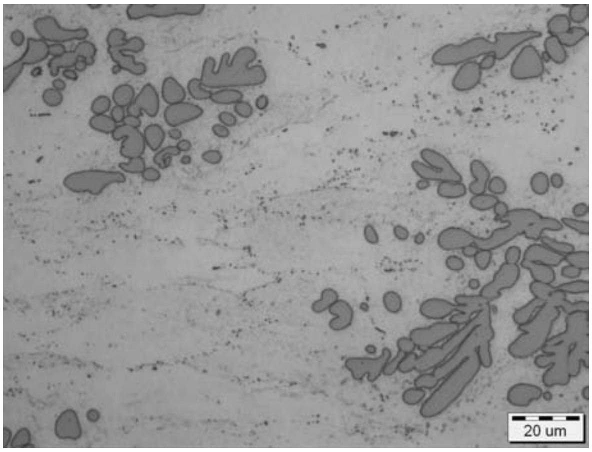
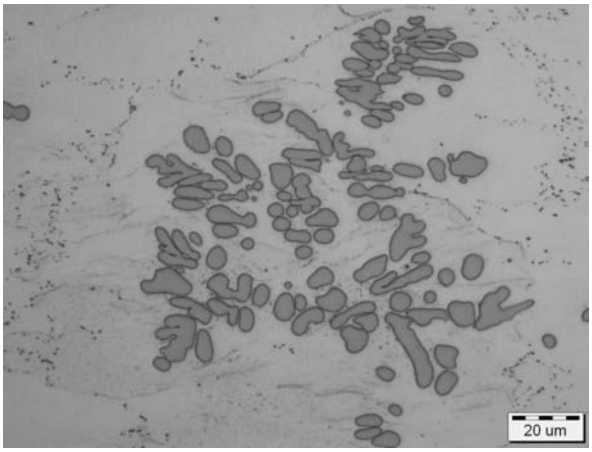


Fig. 11. Cross-section of the wire after bundle extrusion

a) temperature of compression 20ºC



b) temperature of compression 300ºC



c) temperature of compression 500ºC

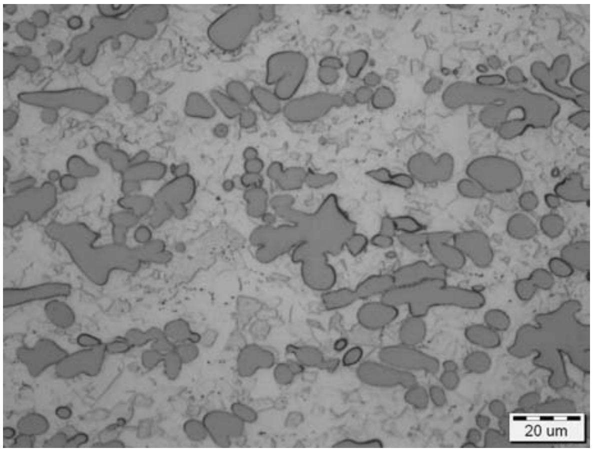
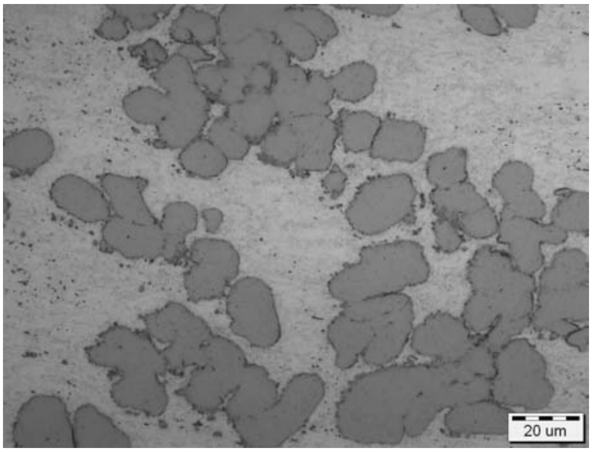
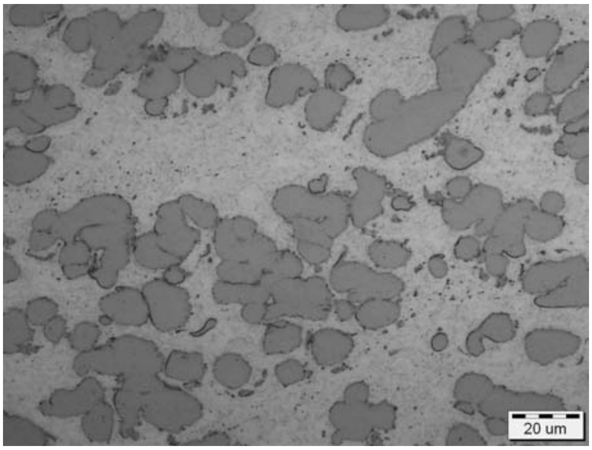


Fig. 12. Microstructure of CuNb15 alloy after casting samples subjected to compression in various temperatures

a) temperature of compression 20ºC



b) temperature of compression 300ºC



c) temperature of compression 500ºC

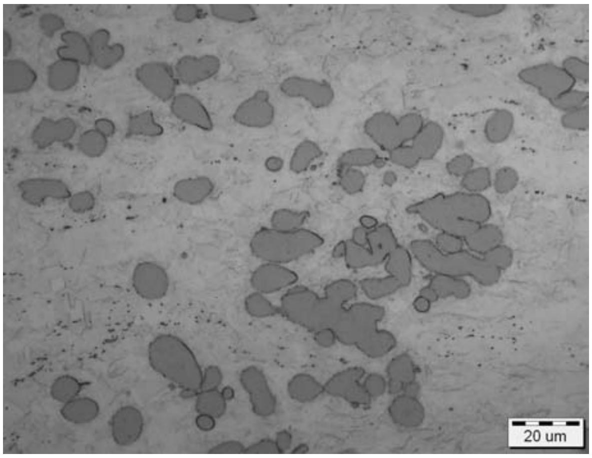
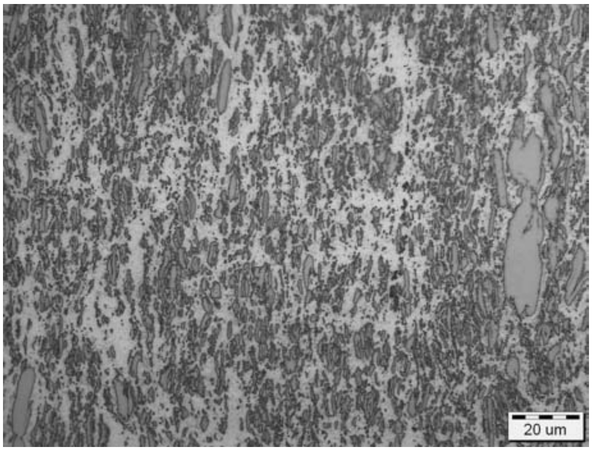
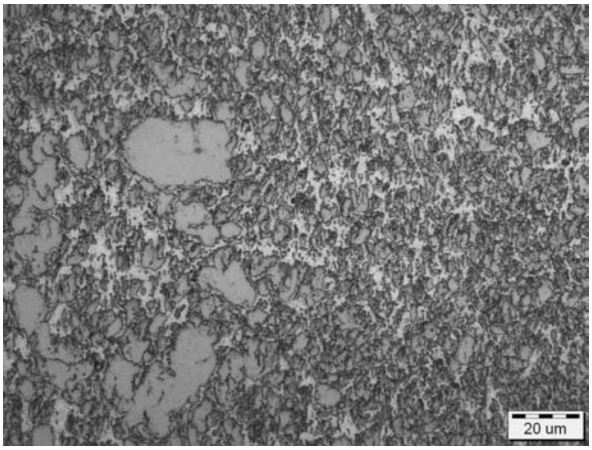


Fig. 13. Microstructure of CuNb15 alloy after casting and hot forging samples subjected to compression in various temperatures

a) temperature of compression 20°C



b) temperature of compression 300°C



c) temperature of compression 500°C

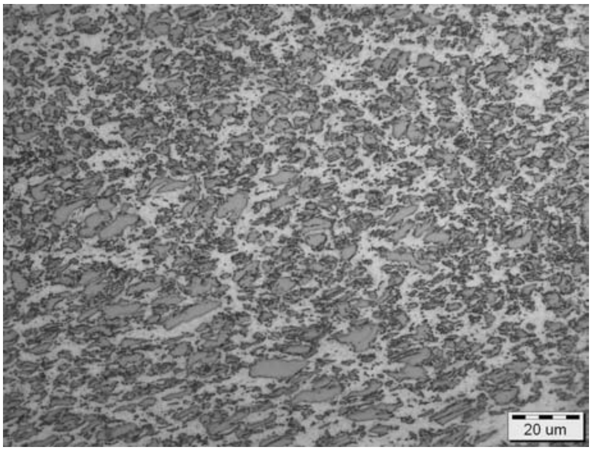


Fig. 14. Microstructure of CuNb15 wire samples extruded from wire bundle by KOBO press subjected to compression in various temperatures

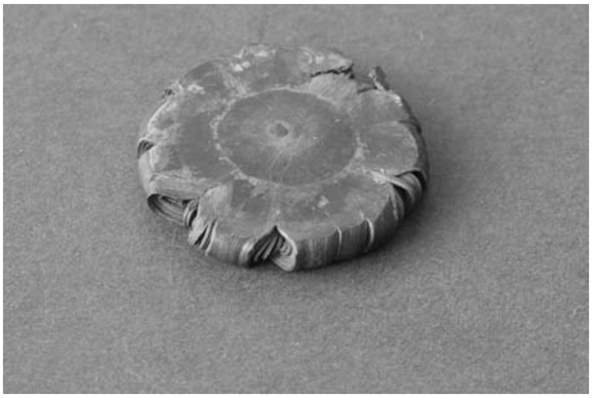


Fig. 15. Sample extruded by KOBO press from wire bundle, after compression in 500°C

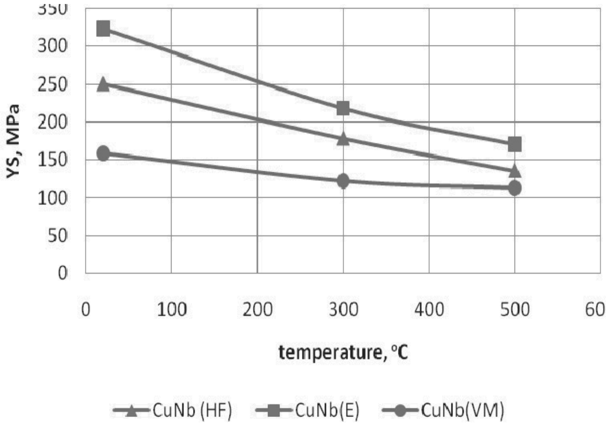


Fig. 16. Change of the proof stress during compression at various temperatures of samples after: Casting - CuNb(VM), Hot forging - CuNb(HF), Extrusion of wire bundle - CuNb(E)

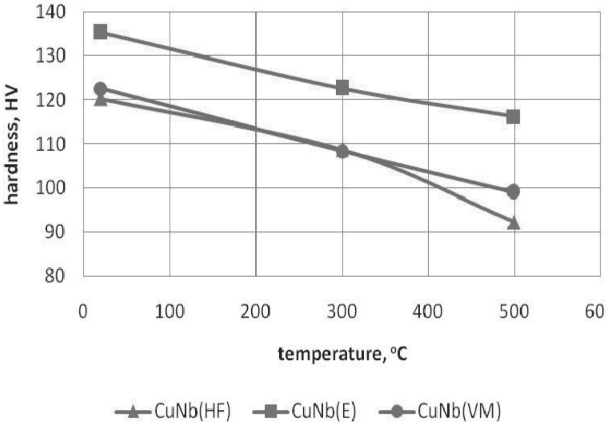


Fig. 17. Change in hardness during compression at various temperatures of samples after: Casting - CuNb(VM), Hot forging - CuNb(HF), Extrusion of wire bundle - CuNb(E)

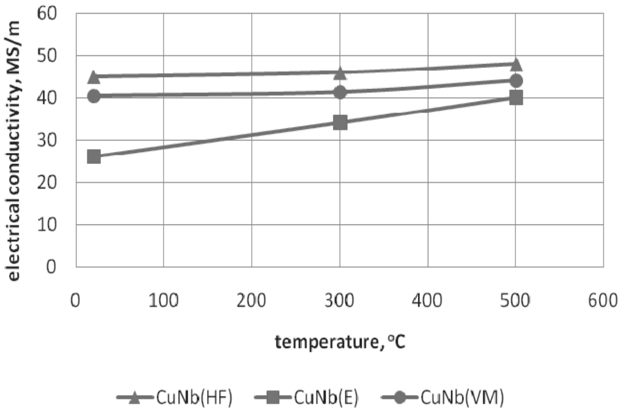


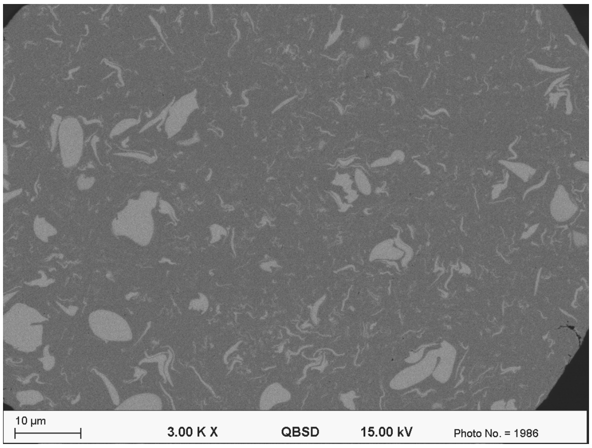
Fig. 18. Change in electrical conductivity during compression at various temperatures of samples after: Casting - CuNb(VM), Hot forging - CuNb(HF), Extrusion of wire bundle - CuNb(E)

Change of the proof stress during compression tests at various temperatures is presented in Fig 16. With the increase of deformation temperature the value of proof stress in all the examined materials decreased. The lowest values were observed in the material after casting while the highest in the material after most extensive plastic working by hot forging, drawing, bundling and extrusion by KOBO press. Similar changes were observed during hardness examination (Fig. 17). The highest values were registered in the material after most extensive plastic working while the alloys after casting and hot forging presented similar level of hardness. Electrical conductivity (Fig. 18) increased with the increase of deformation temperature, especially in the bundle extruded by KOBO press, however the highest electrical conductivity was registered in the material after hot forging.

The material after hot forging was rolled down to the diameter of 3.4 mm and then drawn to the diameter of 2 mm. The wire was annealed in 450°C for 1 hour. Further drawing was conducted until diameter of 0.09 mm was reached and in the result a wire of tensile strength above 900 MPa and electrical conductivity of 44 MS/m was produced. The produced microstructure (Fig. 19) was heterogeneous. The rich in niobium phase was present in the copper matrix in a form of particles of the size of several micrometers and bands of thickness of several hundred nanometers stretched along the direction of deformation.

There is an alternative method for production of Cu-Nb microcomposites by multiple drawing of Nb wire bundle in copper matrix (jacket). In the result of five times performed bundling and drawing of 7 Nb wires (of original diameter 2mm) according to the adopted method (Fig. 20) a microcomposite with copper matrix was produced with evenly distributed 2401 Nb bands, continuous along the whole length of the wire. In the further tests, after 7 bundling a wire was produced with 823543 continuous Nb bands. The produced wires are further examined.

a) cross-section



b) longitudinal section

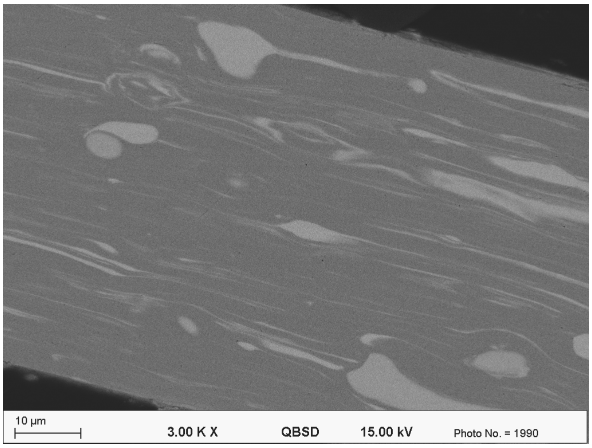


Fig. 19. Microstructure of CuNb wire after hot forging, rolling and drawing down to diameter φ 0.09 mm

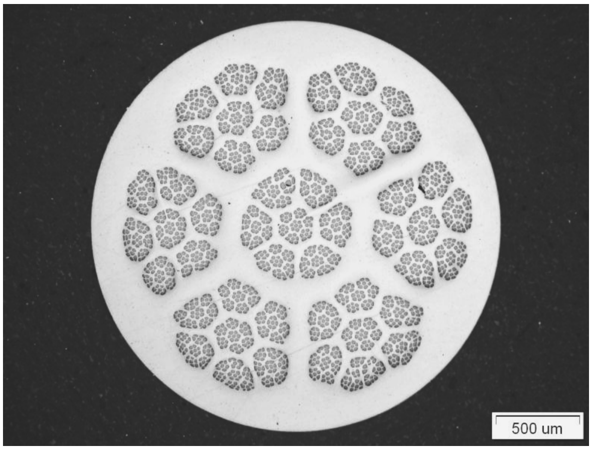


Fig. 20. Microstructure of CuNb microcomposite produced after four times performed bundling of 7 Nb wires in copper matrix

4. Summary

Cu-Nb alloys represent a promising material for production of coil wires for generators of strong magnetic fields. After application of classical melting in vacuum furnace and cold plastic working wires of satisfactory mechanical properties (Rm over 900 MPa) and high electrical conductivity (over 40 MS/m) can be produced. To reach better uniformity of microstructure intensification of the processing can be considered, for example by extrusion of wire bundle by the press with reversibly rotating die (KOBO). There is a need to determine in details process coditions to reach complete plastic consolidation of the extruded material.

Multiple drawing of Nb wire bundle in copper jacket is a promising method for production of Cu-Nb microcomposites. The number of wires which increases in geometric progression during subsequent bundling and accompanying reduction of Nb band cross-section provide possibilities for production of a microcomposite of homogenous microstructure and with Nb microbands evenly distributed in pure copper matrix. In the result it can be expected that the produced wire will have high mechanical properties and high electrical conductivity (above 50MS/m), and will be stable in elevated temperature.

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] S. Bednarek, Project of system for non-destructive production of high magnetic fields by explosive methods, Physica B: Condensed Matter 293 (2000) 67-74.

[2] K. Han, J. Embury, The fabrication, properties and microstructure of Cu-Ag and Cu-Nb composite conductors, Materials Science and Engineering A 267 (1999) 99-114.

[3] J. Embury, K. Han, Conductor materials for high magnetic fields, Solid State and Materials Science 3 (1998) 304-308.

[4] E. Botcharova, J. Freudenberger, A. Gaganov, K. Khlopkov, L. Schultz, Novel Cu-Nb wires: Processing and characterization, Materials Science and Engineering A 416 (2006) 261-268.

[5] J. Liu, L. Meng, Y. Zeng, Microstructure evolution and properties of Cu-Ag microcompositeswith different Ag content, Materials Science and Engineering A 435-436 (2006) 237-244.

[6] F. Heringhaus, D. Raabe, G. Gottstein, On the correlation of microstructure and electromagnetic properties of heavily cold worked Cu-20wt% Nb wires, Acta Metallurgica Materiala 43/4 (1995) 1467-1476.

[7] L. Murr, R. Flores, Defects and failure in ultra-fine copper magnet wire, Scripta Materialia 39/4/5 (1998) 527-532.

[8] J. Stobrawa, Z. Rdzawski, W. Głuchowski, Structure and properties of dispersion hardened submicron grained copper, Journal of Achievements in Materials and Manufacturing Engineering 20 (2007) 195-198.

[9] J. Stobrawa, Z. Rdzawski, W. Głuchowski, Microstructure and properties of nanocrystalline copper - yttriamicrocomposite, Journal of Achievements in Materials and Manufacturing Engineering 24/2 (2007) 83-86.

[10] J. Stobrawa, Z. Rdzawski, Deformation behavior of dispersion hardened nanocrystalline copper, Journal of Achievements in Materials and Manufacturing Engineering 17 (2006) 153-156.

[11] J. Stobrawa, Z. Rdzawski, Dispersion - strengthened nanocrystalline copper, Journal of Achievements in Materials and Manufacturing Engineering 24/2 (2007) 35-42.

[12] Z. Rdzawski, J. Stobrawa, W. Głuchowski, Structure and properties of CuFe2 alloy, Journal of Achievements in Materials and Manufacturing Engineering 33/ 1 (2009) 7-18.

[13] J. Stobrawa, Z. Rdzawski, W. Głuchowski, W. Malec, Ultrafine grained strip od CuCr0,6 alloy prepared by CRCS metod, Journal of Achievements in Materials and Manufacturing Engineering 33/2 (2009) 166-172

[14] J. Stobrawa, Z. Rdzawski, W. Głuchowski, W. Malec, Microstructure and properties of CuNi2Si1 alloy processed by continuous RCS method, Journal of Achievements in Materials and Manufacturing Engineering 37/2 (2009) 466-479.

[15] J. Stobrawa, Z. Rdzawski, W. Głuchowski, W. Malec, Microstructure evolution in CRCS processed strips of CuCr0,6 alloy, Journal of Achievements in Materials and Manufacturing Engineering 38 /2 (2010) 195-202.

[16] J. Stobrawa, Z. Rdzawski, W. Głuchowski, W. Malec, Ultrafine grained strips of precipitation hardened copper alloys, Archives of Metallurgy and Materials 56/1 (2011).

[17] W. Głuchowski, Z. Rdzawski, Silver - mishmetal alloy for application at elevated temperature, Journal of Achievements in Materials and Manufacturing Engineering 26/2 (2008) 123-126.

[18] W. Głuchowski, Z. Rdzawski, Thermal stability of properties in silver-rare earth metals alloys, Journal of Achievements in Materials and Manufacturing Engineering 28/2 (2008) 143-150.

[19] W. Głuchowski, Z. Rdzawski, Stabilization of mechanical properties in silver alloys by addition of Lanthanides, Journal of Achievements in Materials and Manufacturing Engineering 30/2 (2008) 129-134.

[20] Y. Sakai, K. Inoue, H. Maeda, New high-strength, high-conductivity Cu-Ag alloy sheets, Acta Metallurgica Materiala 43/4 (1995) 1517-1522.

[21] W. Grunberger, M. Heilmaier, L. Schultz, Microstructure and mechanical properties of Cu-Ag microcopmosites for conductor wires in pulsed high-field magnets, Zeitschrift für Metallkunde 93 (2002) 58-65.

[22] H. Okamoto, Journal of Phase Equilibria 12 (1991) 614.

[23] M. Hämäläinen J. Jäaskeläinen, R. Luoma M. Nuotio P. Taskinen O. Teppo CALPHAD 14 (1990) 125.

[24] D. Li, M. Robinson, T. Rathz, G. Wiliams, Liquidus temperatures and solofification behavior in the copper-niobium system, Acta Materailia 46/11 (1998) 3849-3855.

[25] Y. Leprince-Wang, K. Han Y. Huang , K. Yu-Zhang, Microstructure in Cu-Nbmicrocomposites, Materials Science and Engineering A 351 (2003) 214-223.

[26] L. Thilly, M. Veron, O. Ludwig, F. Lecourterier, Deformation mechanism in high strength Cu/Nb nanocomposites, Materials Science and Engineering A 309-310 (2001) 510-513.

[27] J. Chung J, Song S. Hong, Bundling and drawing processing of Cu-Nbmicrocopmosites with various Nb content, Journal of Matrerials Processing Technology 113 (2001) 604-609.

[28] S. Hong M. Hill, Microstructure and conductivity of Cu-Nbmicrocomposites fabricated by the bundling and drawing process, Scripta Materialia 44 (2001) 2509-2515.

[29] S. Hong M. Hill Mechanical properties of Cu-Nb microcomposites fabricated by the bundling and drawing process, Scripta Materialia 42 (2000) 737-742.

[30] A.Shikov, V. Pantsyrnyj, A. Vorobieva, N. Khlebova, A. Silaev, High strength, high conductivity Cu-Nb based conductors with nanoscaled microstructure, Physica C 354 (2001) 410-414.

[31] M.J.R. Sandim C.A.M. dos Santos H.R.Z. Sandim, L. Ghivelder, Influence of the filament morphology on the magnetic properties of a Cu-Nb composite Physica C 408-410 (2004) 207-208.

[32] M. Dadras D. Morris, Examination of some high-strength, high-conductivity copper alloys for high-temperature applications, Scripta Materialia 38/2 (1998) 199-205.

[33] B.Z. Cui, Y. Xin and K. Han, Structure and transport properties of nanolaminate Cu–Nb composite foils by a simple fabrication route, Scripta Materialia 56 (2007) 879-882.

[34] M. Marques, V. Livramento, J. Correia, A. Almeida, R. Villar, Production of copper-niobium carbide nanocomposite powders via mechanical alloying, Materials Science and Engineering A 399 (2005) 382-386.

[35] D. Raabe, U. Hangen, Simulation of the yield strength of wire drawn Cu-based in-situ composites, Computational Materials Science 5 (1996) 195-202.

[36] S. Ig Hong, Yield strength of a heavily drawn Cu-20% Nb filamentary microcomposite, Scripta Materialia 39/2 (1998) 1685-1691.