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# MICROSTRUCTURE AND PROPERTIES OF MULTIFIBRE COMPOSITES

In the study microstructure and properties of composite multifibre copper-base wires are presented. A decision was made to produce wires with "soft" fibres (Al) and "hard" fibres (Fe). In the study the phenomenon occurring on the border of Al-Cu was also analysed. The produced Cu-Al and Cu-Fe composites presented ordered microstructure with the fibres uniformly distributed in the copper matrix. The composites underwent plastic consolidation to the degree which provided satisfactory mechanical and electrical properties. During the drawing the fibres deformed proportionally with copper matrix therefore their content in the cross section remained unchanged.

*Keywords:* Fibrous Composite; Copper Matrix, Microstructure, Mechanical Properties; Electrical Properties.

## 1. Introduction

The produced by casting/cold working composites in Cu matrix play an important role in industry, especially because of their attractive combination of electrical conductivity and high strength. Numerous studies have been focused on materials in Cu matrix, such as Cu-Nb, Cu-Ag, Cu-Fe and Cu-Cr [1-2].

Cu-Nb alloys represent a promising group of materials for production of generators of strong and variable magnetic fields. The published papers [3-4] address production of fibrous Cu-Nb microcomposite by multiple iterative drawing of a bundle of Nb wires in Cu tube. The produced wire presented ordered composite structure, high electric conductivity and high mechanical properties. The wire of a diameter of 0.1 mm contained more than 800,000 continuous Nb fibres of sub-micron cross section [4]. The disadvantage of this solution can be seen in the process of bundling, which is difficult to be applied in industrial conditions.

The Cu-Fe alloys are subject of specific interest because of relatively low cost and availability of that alloying element. The fine crystalline microstructure of Cu-Fe alloy and high density of the phases at the interface may have some influence on the increase of mechanical properties [5]. The mechanical properties depend, among others, on the distance between the fibres in the drawn composite. For example, in the Cu-20% Fe composite which was produced by powder metallurgy methods increase of the strength according to the Hall-Petch dependence with the decrease of the distance between Fe fibres was observed. Thickness, width and distribution of Fe fibers in the fibrous structure decrease rapidly with the increase of the strain during drawing. In the study [6] an experiment and simulation were made of thermal instability of Fe fibres which were deformed during drawing (actual deformation 8.2). It was presented that when annealing temperature is below 500°C the Fe fibres still maintain the shape of fibres before the drawing, however, with the temperature increase to 600°C (temperature of iron recrystallization), the longitudinal fibres start to break and to coagulate.

Another promising group of materials can be seen in the alloys and composites of Cu-Ag and Cu-Al type. Wires made of CuAg15 alloy after cold working (drawing) were characterized by advantageous microstructure, in which numerous narrow fibres of (silver rich) $\beta$ phase were distributed parallel to the strain direction in (copper rich) $\alpha$ phase [7]. It contributed to the high tensile strength $Rm$ = 1,120 MPa and electrical conductivity 40 MS/m. Annealing in temperature of 200°C for 3 hours and for 7 hours resulted in increase of electrical conductivity to 45 MS/m, while the tensile strength remaimed at the level slightly below 1,100 MPa.

In the case of Cu-Al composites it is especially important to conduct the heat treatment process in a proper way because of the hazard of formation of brittle intermetallic compounds which reduce functional properties [8]. In the annealing temperature (200-540°C) several Al-Cu intermetallic phases develop, i.e. $Al_2Cu$ (θ), AlCu (η 2), $Al_3Cu_4$ (ζ 2), $Al_4Cu_9$ ($\gamma_1$) [9]. Because of the higher diffusivity of Cu in Al than Al in Cu, formation of $Al_2Cu$ compounds takes place before formation of $Al_4Cu_9$ compounds. The AlCu and $Al_3Cu_4$ phases become formed after generation of the two preceding phases. AlCu and $Al_3Cu_4$ present higher hardness when compared to the other phases, which also means lower resistance to brittle cracking. Al and Cu show high tendency to reactive diffusion in the temperature above 120°C, when intermetallic compounds are formed at the interphase boundary [8]. The formation and growth of the in-

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termetallic compounds has significant influence on mechanical and electrical properties of Cu-Al composites. In the study [10] and attempt was made to determine heat treatment conditions for production of CCA (copper-clad aluminium) wires of optimum mechanical properties. In the tests Al rod of diameter 20 mm was covered with copper strip of about 0.5 mm thickness and was drawn down into the form of thin wire of 300 μm diameter. The ratio of copper and aluminium volume in the drawing process remained constant (approximately 15/85). In order to control the amount and size of intermetallic compounds various annealing times were applied, from 10 minutes to 7 hours in temperature of 300°C. Additionally heat treatment operation was applied at various temperatures to determine the activation energy of diffusion process. The maximum tensile strength was 250 MPa. The heat treatment resulted in its decrease down to 135 MPa.

In this study an attempt was made to produce multifibre composite wires by continuous and easily implemented in industrial practice method. The objective of the conducted studies was to determine possibilities for production of wires of complex structure composed of fibres and matrix characterized by significant differences in their mechanical and electrical properties. In the study influence of phenomena taking place at interphase boundary on the final set of functional properties of multifibre Cu-Al composite was analysed.

## 2. Methodology, material for studies

Two Cu-based composites were selected for the studies. Wires made of "soft" matrix (Cu) and "hard" fibres (Fe) as well as of "soft" matrix (Cu) and "soft" fibres (Al) were prepared. The first one was made of bimetallic CuFe wires, where the low-carbon steel core was covered with copper layer. The copper content in the wire was at the level of 30%. 77 wires of diameter 0.4 mm were spliced to form a cable of diameter 4.45 mm. The cable was introduced into a copper tube of diameter 10 mm and wall thickness of 1mm. The produced set was drawn through a block drawing machine down to 5 mm diameter and annealed in protective atmosphere in temperature of 620°C for 2h. The wire was then drawn down to 1.3 mm diameter with application of interoperational annealing at the diameter of 4.1 mm (660°C for 3h) and 2.2 mm (650°C for 3.5h). Further drawing down to the diameter of 0.12 mm was performed with application of multi-die drawing machine with interoperational annealing at the diameter of 0.99 mm (680°C for 1 hour).

The second composite was made of copper matrix into which Al fibres were introduced. It consisted of bimetallic CuAl wires in which aluminium core was covered with copper layer. Copper content in the wire was 30%. 49 wires of diameter 1.2 mm were spliced to form a cable of diameter 9.5 mm. The cable was introduced into a copper tube of diameter 18 mm and wall thickness of 1 mm. That set was drawn with a block drawing machine down to the diameter of 8 mm and annealed in the protective atmosphere in temperature of 300°C for 1.5h. Then the wire was drawn down to 1.2 mm with interoperational annealing at the diameter of 5.1 mm (280°C for 1.5h) and 3.2 mm (500°C for 1h). Further drawing down to the diameter of 0.34 mm was conducted with application of a multi-die drawing machine. In order to examine the phenomena occurring at the interphase boundary and resulting in formation of intermetallic phases in the CuAl composite the wire of diameter 1.4 mm, was additionally annealed in 500°C for 1 hour.

Observations of microstructure of the materials was done with application of metallographic microscope on the polished section transverse and longitudinal to the extrusion direction and in the scanning electron microscope with the microanalysis of chemical composition (EDS) in microareas. The tension tests were done in the testing machine. Electrical conductivity was measured with Kelvin bridge.

## 3. Results and discussion

The microstructure of the cross-section of Cu-Al wire after drawing from the diameter of 5.1 mm as observed with light microscopy is presented in Fig. 1.

a)
b)

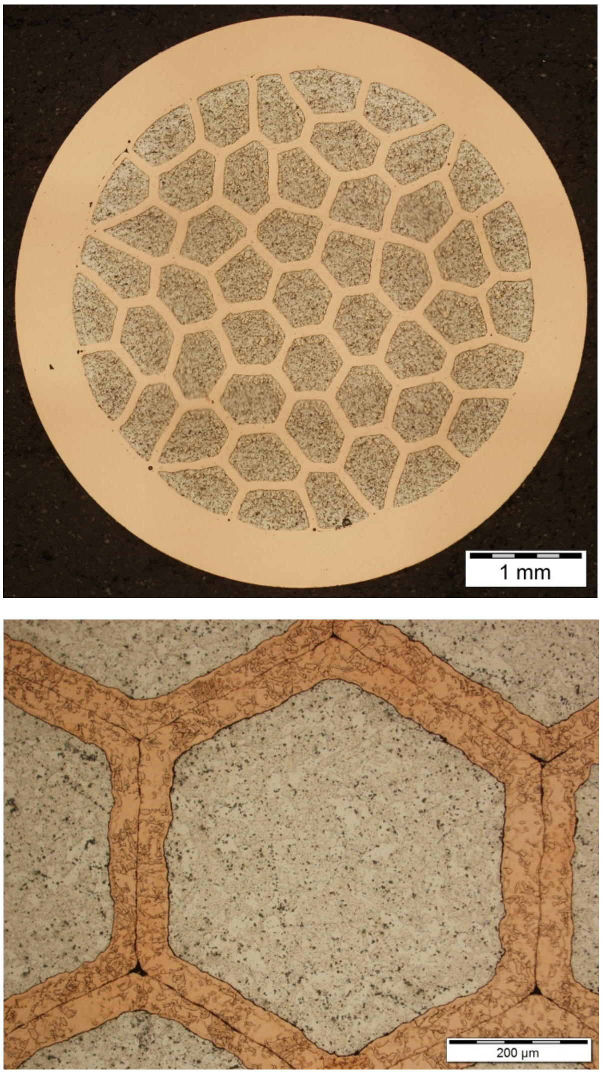



Fig. 1. Microstructure of Cu-Al wire of diameter 5.1 mm, a) magnif. 60×, b) magnif. 200×

Figures 2-3 present results of measurements of the diameter of the wire, jacket thickness and diameters of Al fibres which changed during drawing. A phenomenon of plastic consolidation with the strain increase was observed. Microstructure of both copper matrix and Al fibres became broken-up. The change of the diameter of Al fibres was directly proportional to the change of the diameter of the wire during drawing (Fig. 2). The fibres were arranged in an ordered way and parallel throughout the whole length of the wire. The content of copper jacket in the wire did not change during drawing and was slightly above 35%. During the processing the material showed sufficient reserve of plasticity.

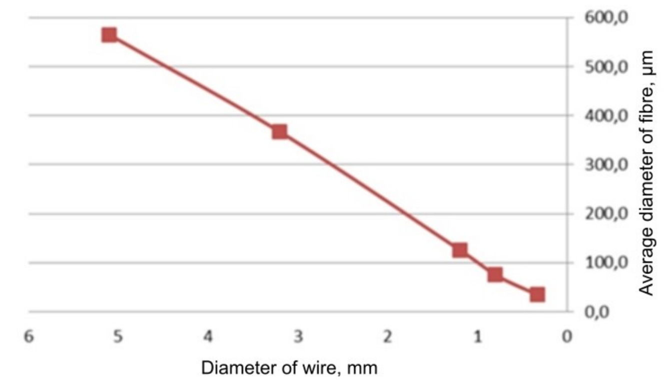



Fig. 2. Changes of average diameter of Al fibres vs outer diameter of composite wire during drawing

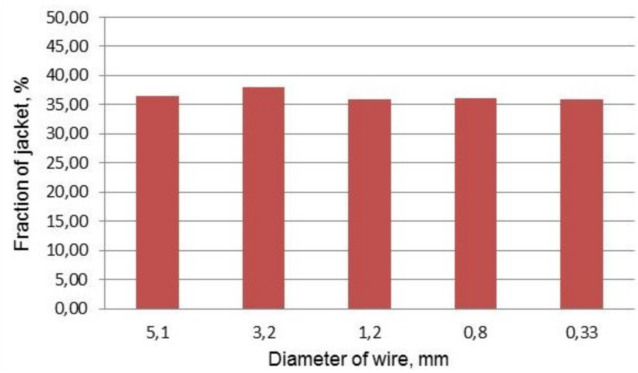



Fig. 3. Surface fraction of copper jacket in composite wire vs outer diameter during drawing

a)

b)

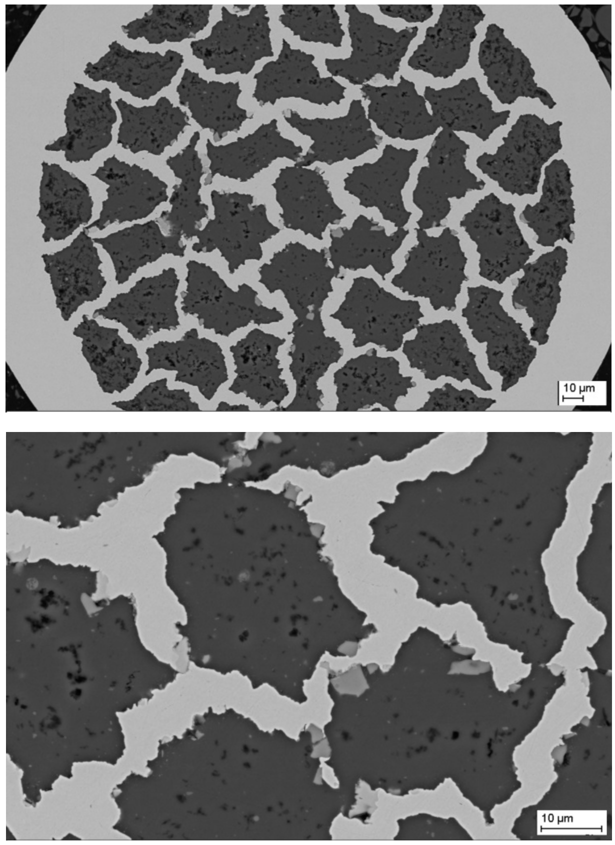



Fig. 4. Microstructure of CuAl wire of diameter 0.34, polished cross-section, SEM, a) magnif. 1000×, b) magnif. 3000×

a)

b)

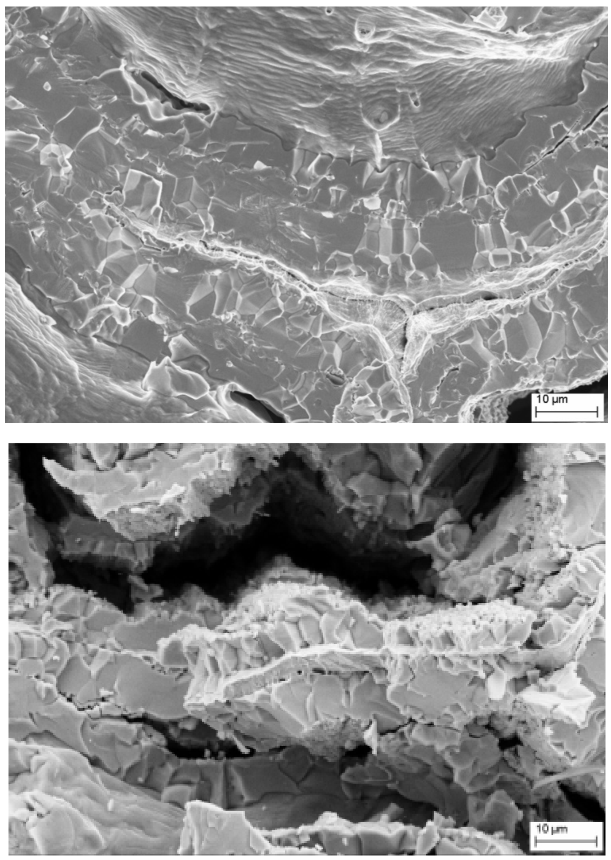



Fig. 5. Fracture of CuAl wire of diameter 1.4 additionally annealed in temperature 500°C/1h, SEM, a) magnif. 3000×, b) magnif. 3000×

In microstructure of CuAl wire of diameter 0.34 mm (Fig. 4) presence of $Al_2Cu$ intermetallic phases of several micrometres was observed. The particles most probably became formed in earlier stages of the process. Because of high hardness they were not deformed (broken-up) together with the copper matrix and Al fibres, therefore their presence in the microstructure was most distinct and had the biggest influence on properties of the wire after plastic working to the smallest diameter of 0.34 mm. Based on the results of surface and linear distribution of Al and Cu it was established that the particles represented $Al_2Cu$ intermetallic phase.

To confirm the presence of intermetallic phases the wire of 1.4 mm diameter was annealed for 1 hour in the temperature of 500°C. SEM observations of both fractures – Fig. 5 and microstructure – Fig. 6 confirmed formation of intermetallic phases in the area between copper and aluminium. In the area of intermetallic phases the fracture was of brittle character without any visible effects of plastic strain, unlike the ductile character of the fracture observed in the area of pure copper and aluminium. Three intermetallic phases can be singled out in the examined wire. The literature data [10] show that $Al_2Cu$, $Al_4Cu_9$ compounds are the first to be formed and then formation of AlCu and $Al_3Cu_4$ takes place. The observed phases can be identified as $Al_2Cu$, AlCu+$Al_3Cu_4$ and $Al_4Cu_9$. Thicknesses of the individual phases formed during additional 1 hour annealing in temperature of 500°C were 8.3-9.2 μm, 3.5-4.2 μm, 2.5-5.0 μm, respectively.

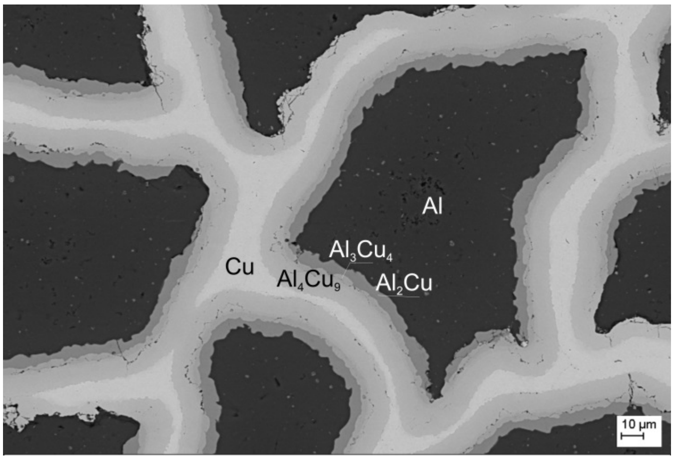



Fig. 6. Microstructure of CuAl wire of 1.4mm thickness additionally annealed in temperature of 500°C/1h

Based on the examination of mechanical properties the relations between the strain and mechanical properties of the composite wire during its drawing from the diameter of 3.2 mm (as annealed in 500°C for 1 hour) to the diameter of 0.34 mm – actual strain 3.6, were determined (Fig. 7). Increase of tensile strength from about 120 MPa to 330 MPa was observed as well as increase of the proof stress from about 50 MPa to 320 MPa. Elongation during cold working decreased vary quickly and remained at the level of 1-2%.

Additional annealing of the wire of diameter 1.4 mm for 1 hour in temperature of 500°C resulted in decrease of mechanical properties TS 130 MPa and $R_{02}$ 120 MPa, however it did not bring any wire elongation.

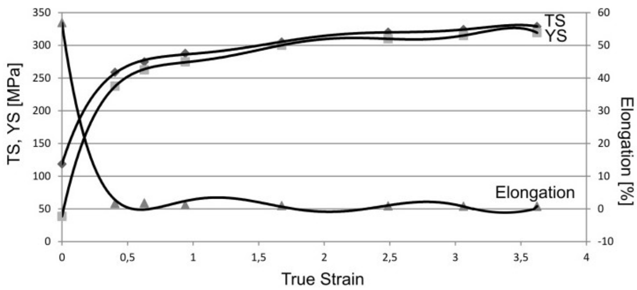



Fig. 7. Changes in Tensile Strength, Yield Strength and Elongation in relations to the deformation of the Cu-Al wire from diameter of 3.21 mm to diameter 0.34 mm.

The highest electrical conductivity (49.5 MS/m) was observed in the wire of diameter 3.2 mm. Further plastic working resulted in insignificant decrease down to 47.8 MS/m at the diameter of 0.34 mm. Additional annealing of the wire of diameter 1.4 mm in 500°C for 1 hour brought significant decrease of the electrical conductivity to 33.4 MS/m.

The second of the examined materials was in a form of a composite wire in copper matrix with Fe fibres. Figure 8 presents microstructure of Cu-Fe in the section transverse to the drawing direction. During plastic working fragmentation of microstructure was observed, especially in the cross-section of Fe fibres.

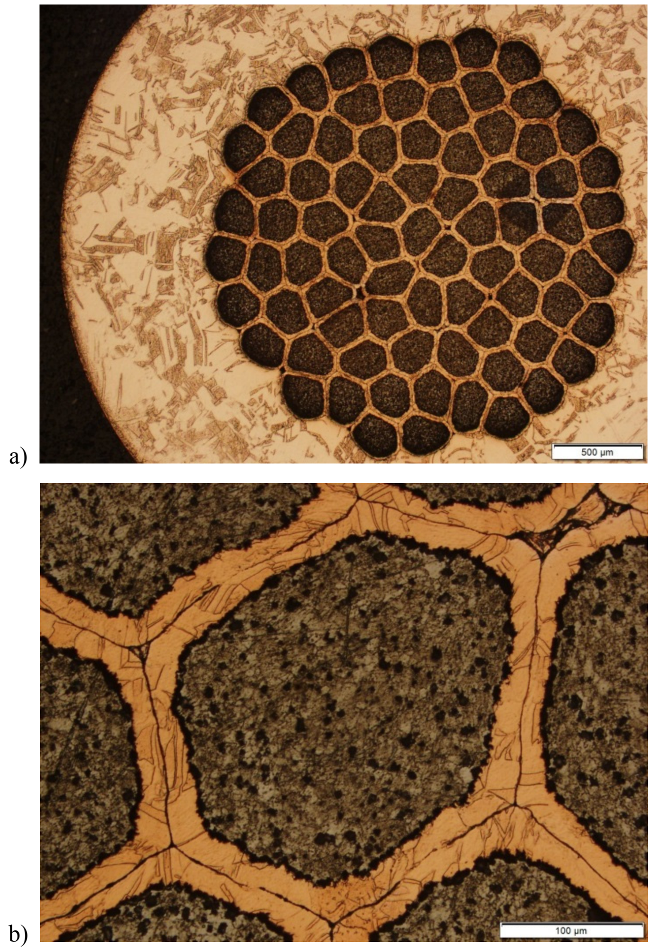



Fig. 8. Microstructure of Cu-Fe wire of diameter 4.1mm, cross-section, a) magnif. 50×, b) magnif. 400×

Figures 9-10 present additionally results of measurements of the wire diameter, jacket thickness and diameters of Fe fibres which change during the drawing. A phenomenon of plastic consolidation with the increase of the strain during drawing was observed. The change of diameter of Fe fibres was directly proportional to the change of the wire diameter during the drawing. The fibres were arranged along the axis of the wire, parallel to each other along the whole length of the wire. The fraction of the copper jacket in the wire did not change during the drawing and was at the level of about 65% (Fig. 10). No transient phases were observed at the border of copper and iron.

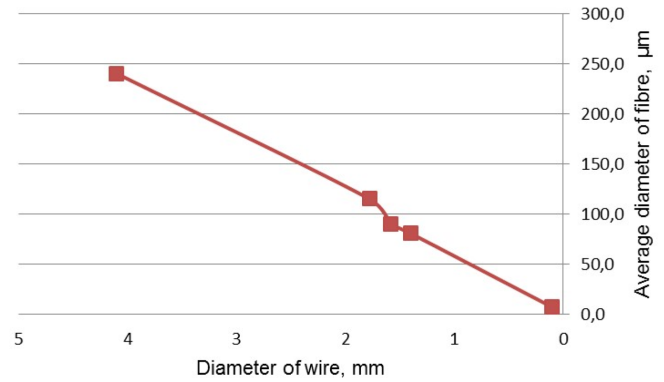



Fig. 9. Change of average diameter of Fe fibres in relations to the outer diameter of composite wire during drawing

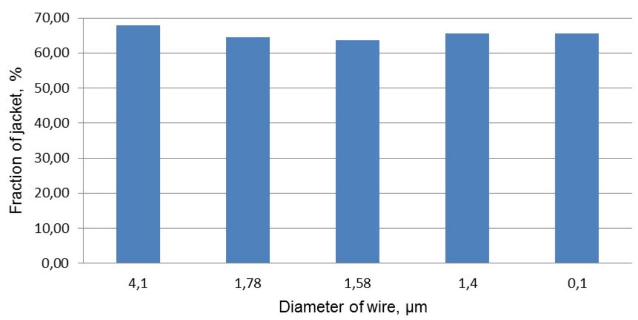
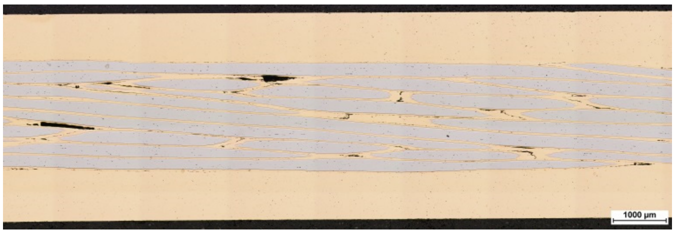
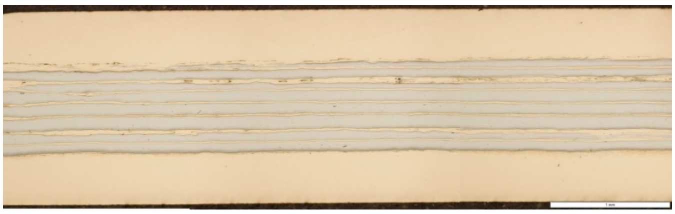



Fig. 10. Surface fraction of the copper jacket in Cu-Fe composite wire vs outer diameter during drawing

Large deformation from the diameter of 4.1 mm to diameter of 0.1 mm resulted in expansion of the shape of fibres cross-section. The fibres initially spliced to form a cable became untwisted and arranged parallel to the strain direction (Fig. 11). During drawing the composite wire presented sufficient reserve of plasticity.




a)



b)

Fig. 11. Microstructure of Cu-Fe wire of diameter a) 4.1 mm, b) 1.78 mm, longitudinal section

In the Cu-Fe microcomposite which was plastic worked by drawing from diameter of 2.08 mm to 0.12 mm (actual strain – 5.7) influence of cold plastic deformation on mechanical properties was determined from the results of a tensile test. The tensile strength of the wire of diameter 0.12 mm increased from 264 MPa to 710 MPa. The proof stress increased from 100 MPa to 670 MPa (Fig. 12).

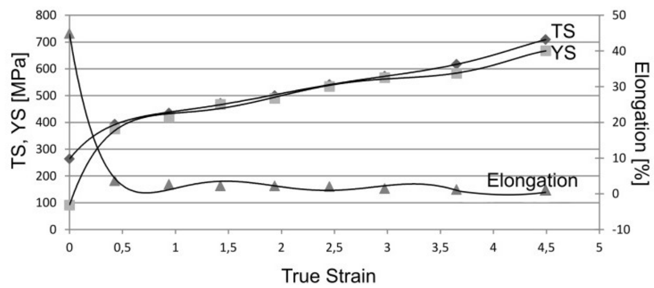



Fig. 12. Changes in Tensile Strength, Yield Strength and Elongation in relations to deformation of the Cu-Fe wire from diameter of 2.08 mm to diameter of 0.12 mm

Closing of internal spaces during plastic consolidation of the CuFe composite wire contributed to the increase of its electrical conductivity to about 41 MS/m.

## 4. Conclusions

The studied technology provided possibility to produce composite multifibre wires in pilot scale with controlled microstructure, also from the materials resistant to cold plastic deformation.

The produced Cu-Al and Cu-Fe composites were characterized by microstructure with fibres evenly distributed in the copper matrix. The composites underwent plastic consolidation to the degree sufficient to reach satisfying mechanical and electrical properties. During drawing the fibres became deformed proportionally with the copper matrix, therefore their fraction in a cross-section remained unchanged. Al fibres were deformed from the diameter of 1.2 mm down to the diameter of about 35 µm in the composite wire after drawing. In a similar way Fe fibres were deformed from the initial diameter of 0.4 mm to the diameter of 7.4 µm.

The widely used bimetallic Cu-Al wires have tensile strength TS – 280 MPa, yield point of about 140 MPa and electrical conductivity of 38 MS/m. The examined in this study multifibre CuAl composites reach higher mechanical properties (TS 330 MPa and $R_{0.2}$ 320 MPa) and significantly higher electrical conductivity – 49 MS/m.

Mechanical properties of the examined multifibre Cu-Fe wires (TS about 700 MPa) are comparable to mechanical properties of commercial bimetallic Cu-Fe wires, while electrical conductivity of the multifibre CuFe wires – 41 MS/m, is almost two times higher than in bimetallic CuFe wires (23 MS/m).

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## REFERENCES

[1] Z. Rdzawski, W. Głuchowski, J. Stobrawa, W. Kempiński, B. Andrzejewski, Microstructure and properties of Cu-Nb and Cu-Ag nanofiber composites, Archives of Civil and Mechanical Engineering **15**, 689-697 (2015).

[2] Z.W. Wu, J.J. Liu, Y. Chen, L. Meng, Microstructure, mechanical properties and electrical conductivity of Cu-12 wt.% Fe microcomposite annealed at different temperatures, Journal of Alloys and Compounds **467**, 213-218 (2009).

[3] Hong, S.I., Hill, M.A., Microstructure and conductivity of Cu-Nb microcomposites fabricated by the bundling and drawing process. Scripta Materialia **44**(10), 2509-2515 (2001).

[4] W. Głuchowski, Z. Rdzawski, J. Stobrawa, Microstructural characterization of high strength high conductivity Cu-Nb microcomposite wires, Journal of Achievements in Materials and Manufacturing Engineering **46**/1, 40-49 (2011).

[5] Y.S., Go, W.A., Spitzig, Strengthening in deformation processed Cu-20% Fe composites. Journal of Materials Science **26**(1), 163-171 (1991).

[6] Xiao-pei LU, Da-wei YAO, Yi CHEN, Li-tian WANG, An-ping DONG, Liang MENG, Jia-bin LIU, Microstructure and hardness of Cu-12% Fe composite at different drawing strains, Applied Physics & Engineering **15**/2, 149-156 (2014).

[7] Z. Rdzawski, W. Głuchowski, J. Stobrawa, J. Sobota, Effect Of Rare-Earth metals addition on microstructure and properties of selected copper alloys, Archives of Metallurgy and Materials **59**/2, 641-648 (2014).

[8] M. Abbasi, A. Karimi Taheri, M.T. Salehi, Growth rate of intermetallic compounds in Al/Cu bimetal produced by cold roll welding process, Journal of Alloys and Compounds **319**, 233-241 (2001).

[9] Chih-Yuan Chen and Weng-Sing Hwang, Effect of Annealing on the Interfacial Structure of Aluminum-Copper Joints, Materials Transactions **48**/7, 1938-1947 (2007).

[10] E. Hug, N. Bellido, Brittleness study of intermetallic (Cu, Al) layers in copper-clad aluminium thin wires, Materials Science and Engineering **A 528**, 7103-7106 (2011).