

A. KAWECKI*, T. KNYCH*, E. SIEJA-SMAGA*, A. MAMALA*,
P. KWAŚNIEWSKI*, G. KIESIEWICZ*, B. SMYRAK*, A. PACEWICZ*

FABRICATION, PROPERTIES AND MICROSTRUCTURES OF HIGH STRENGTH AND HIGH CONDUCTIVITY COPPER-SILVER WIRES

OTRZYMYWANIE ORAZ WŁASNOŚCI I MIKROSTRUKTURA WYSOKOWYTRZYMAŁYCH I WYSOKO PRZEWODZĄCYCH DRUTÓW ZE STOPÓW Cu-Ag

Research results of manufacturing composite filamentary nanostructure Cu-Ag alloys with silver addition from 5 to 15% wt. are presented in the paper. Manufacturing technology of these composites and variable solubility of silver in copper and copper in silver in the range of solid solutions. Suitable quantity and processing sequences of high deformation plastic working and heat treatment allows to obtain wires constituted from Cu and Ag fibres with nanometric transverse dimensions and in consequence provide to optimum superposition of high mechanical strength, high electrical conductivity and sufficient ductility of Cu-Ag alloys.

The paper presents the method of continuous casting of alloys, selected physico-chemical properties and degree of deformation. Influence of chosen heat treatment method over electrical and mechanical properties of both casts and micro wires on mechanical and electrical properties of cast materials during converting them into micro wires with tensile strength higher than 1200 MPa and electrical conductivity higher than 40 MS/m are presented too.

Research results of optical and scanning microscopy structure analysis were presented for casts and wires submitted to various thermo-mechanical strengthening.

Keywords: silver-copper alloys, continuous casting, drawing, micro-wires, filamentary micro-composite, nanostructure, high conductivity, high strength

Praca dotyczy badań nad kształtowaniem zespołu bardzo wysokich własności wytrzymałościowych i elektrycznych drutów i mikro-drutów ze stopów CuAg5 i CuAg15. Technologia wytwarzania drutów ze stopów Cu-Ag wykorzystuje zjawisko obustronnej zmiennej rozpuszczalności składników stopów w stanie stałym. Jak wykazały przeprowadzone badania, odpowiednie połączenie przeróbki plastycznej materiałów o strukturze odlewniczej z międzyoperacyjną obróbką cieplną umożliwia uzyskanie korzystnej kompozytowej mikrostruktury silnie wydłużonych włókien Cu i Ag o nanometrycznych wymiarach poprzecznych. Optymalizacja parametrów technologicznych pozwala na uzyskanie drutów i mikro-drutów Cu-Ag o wytrzymałości na rozciąganie w zakresie 1000÷1300 MPa przy równocześnie wysokiej przewodności elektrycznej wynoszącej 70÷85% w skali IACS.

W artykule pokazano metodę uzyskania stopów Cu-Ag oraz wyniki badań wybranych własności fizykochemicznych, schemat odkształcenia oraz badania wpływu wstępnej obróbki cieplnej materiałów w stanie odlanym na zmianę własności elektrycznych i mechanicznych zarówno odlewów jak i drutów po przeróbce plastycznej. Zamieszczono także wyniki obserwacji strukturalnych przy zastosowaniu mikroskopii optycznej i skaningowej odlewów oraz ewolucję struktury po przeróbce plastycznej oraz po różnych etapach międzyoperacyjnej obróbki cieplnej.

1. Introduction

Conductive copper alloys of extra-standard high set of mechanical and electrical properties can be used as important elements of electrical conductivity bearing considerable mechanical loads. It is difficult to achieve concurrently very high mechanical and electrical properties of copper and its alloys. To do so, one must choose

and use appropriate alloy components as well as apply proper thermo-mechanical treatment.

In case of copper alloys of increased content of other elements, (such as arsenic, beryllium, chromium, tin, zirconium, silicon manganese, manganese, nickel, magnesium, tellurium alloys, etc.), practical UTS of wires rarely exceeds 700 MPa, and, at the same time, their conductivity is relatively low and runs at a range of

* AGH UNIVERSITY OF SCIENCE AND TECHNOLOGY, FACULTY OF NON-FERROUS METALS, 30-059 KRAKÓW, 30 MICKIEWICZA AV., POLAND

20÷40 MS/m. However, the values of above-mentioned set of parameters may turn out to be insufficient in specific applications.

The branch of bundles, wires and sections of a special intended use (mainly in the area of electric energy distribution, power systems of strong magnetic field, system generators, bundles supplying wind generators, etc.) requires significantly higher strength and electrical properties ($UTS > 1000$ MPa, $\gamma > 45$ MS/m) with the mass reduced. The latest research conducted in the field showed that those parameters can be obtained using Cu-Ag alloys with increased Ag content.

First patent applications in the field of obtaining and processing of Cu-Ag alloys with increased Ag content date back to 1940s and 50s (e.g. US Patent No. 2,567,560, 1948, *Heat Treatment of Copper-Silver Binary Alloys*). However, rapid development of research on those alloys took place only at the beginning of 1990s, when a range of new application methods for this kind of materials appeared. The precursors of obtaining and processing of silver copper were mostly scientists from Japan (such as Sakai, Maeda, and Inoue), and from the US (Schneider-Muntau, Sohn) [4].

Unquestionable development of global research on Cu-Ag alloys can be observed in recent 10 years, particularly in Asia. The largest number of research results were published in Japan (Oshaki, Yamazaki, Hono, Shibata, Arai, Watanabe, Asano, Kiyoshi), China (Zhang, Ning, Meng, Liu, Tian), and South Korea (Hoon Cho, Byoung-Soo Lee, Bok-Hyun Kang, Ki-Young Kim, Choi, Kwon), where virtually 80% of all available works come from. The other research centres are located mainly in the US (Han, Vasquez, Kalu, Hong, Ceylan, Sheng, Campbell), Canada (Hill, Wood, Embury), Germany (Mattissen, Heringhaus, Grunberger, Heilmaier, Schultz), Belgium (Frings, Bockstal), Switzerland (Benghalem, Morris), Russia (Gaganov, Nikulin, Vorobieva, Shokov, Pansyrnyj), and Sweden (Fredrikson, Haddad-Sabsevar) [1-8]. The latest publications are from the previous two months of the current year. The crucial reason for research was rapid technological development in energy, automobile, and medical equipment industry, forcing constructors and producers to use materials with higher operating parameters.

2. Obtaining alloys and their properties

Research materials in the form of Cu-5%wt. Ag and Cu-15%wt. Ag alloy rods were obtained using a laboratory installation for continuous casting. The installation consists of an inductive furnace, a generator, an electronic system for casting control, and system which pulls out cast rods (two rotating mills). The oven con-

tains two independent systems of initial (in the area of crystallizer) and secondary (in the area of rod cast) cooling, which, combined with an appropriate temperature of metal in a melting pot and with proper casting speed, gives an opportunity for controlling the number, size, and arrangement of grains in the rod being cast. Owing to the possibility of inductive forcing of movement of liquid metal in the melting pot, the repeatability of its chemical composition can be obtained along the entire length of a rod. Crystallizers were made from a proper type of graphite shaped in a way, which makes possible a certain connection with a graphite melting pot and the system of initial cooling. The 9,5 mm diameter rods were cast in nitrogen protective atmosphere in $1180 \div 1200^\circ\text{C}$ with the speed of 10 mm/s. In order to maximally deoxidize the alloy, a special graphite casting powder was used to cover the liquid metal.

Copper and silver form together a solid solution with mutual limited solubility in a solid state (cf. Fig. 1). Cu-Ag alloys are characterized by eutectic transformation in 779°C , with eutectic Ag content of 71,9 %wt. As alloy temperature is decreased, a drop in mutual solubility of both elements occurs, according to the limit of alloy components solubility.

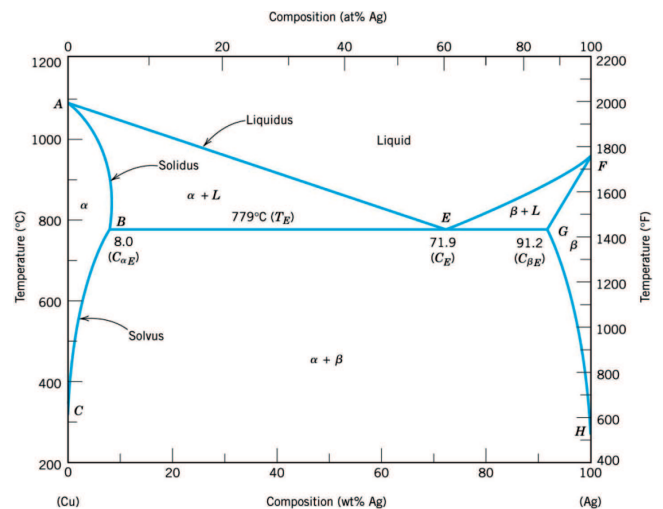


Fig. 1. Phase diagram of Cu-Ag

The Cu-5%wt. Ag alloy solidifies beyond the temperature of eutectic transformation. After solidification, mainly β -phase appears (the solution of silver in copper, Cu-rich phase), during very slow cooling, however, α -phase can also be emitted (the solution of copper in silver, Ag-rich phase).

In case of Cu-Ag alloy eutectic composition (15%wt. Ag), during the solidification, α -phase precipitation results in the change of chemical composition of liquid in accordance with the liquids line. In eutectic temperature, the liquid attains eutectic composition, but after cooling, the alloys consist of α - and β -phase mix-

ture of composition depending on the amount of silver in the alloy, the limit of solubility, and alloy's crystallization conditions.

The microscopic observation of rods after casting in their different plastic deformation level was performed with the Hitachi SU70 scanning microscope.

Fig. 2 shows CuAg5 and CuAg15 alloys' microstructure. Casting structure of the CuAg5 alloy contains Ag-rich areas (white) precipitated from the matrix which consists mostly of copper (dark background). The CuAg15 alloy's microstructure contains eutectic structure appearing within dendritic structures in conglomer-

ates of large grains that allocates spatially, depending on their crystallization conditions. Owing to mutual limited solubility of Ag in Cu and *vice versa*, casting structure consists of the matrix (Cu with some Ag), and precipitations enriched with Ag containing a little amount of Cu.

Fig. 3 presents sample results of an analysis of distribution of elements constituting the Cu-15%wt. Ag alloy. The analysis of chemical composition shows Cu- and Ag-rich areas, which confirms the effect of mutual precipitation of both elements.

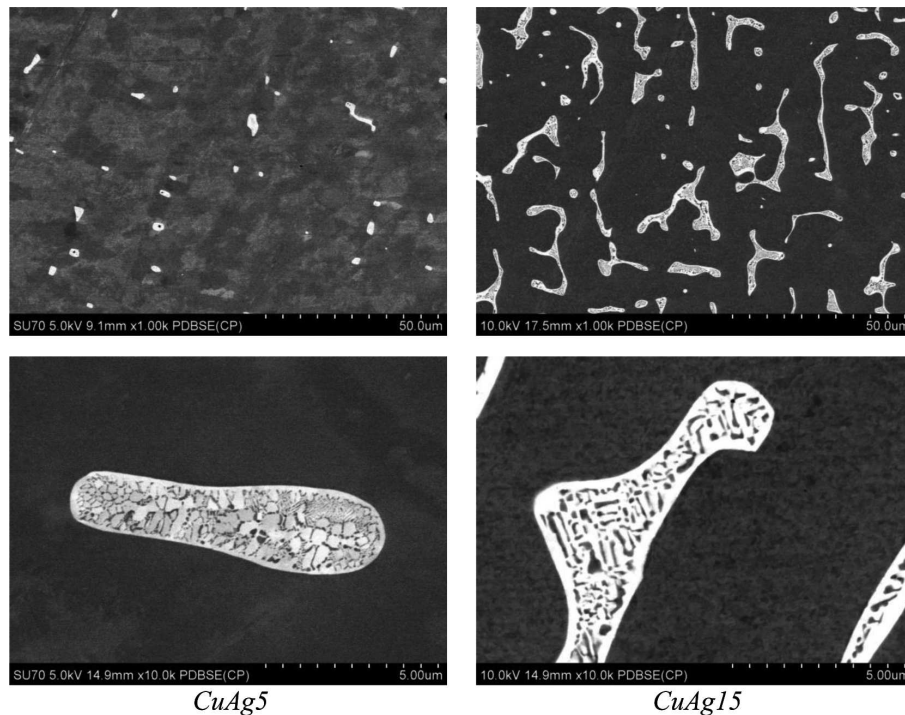


Fig. 2. SEM microstructures in longitudinal section of rods made from CuAg5 and CuAg15

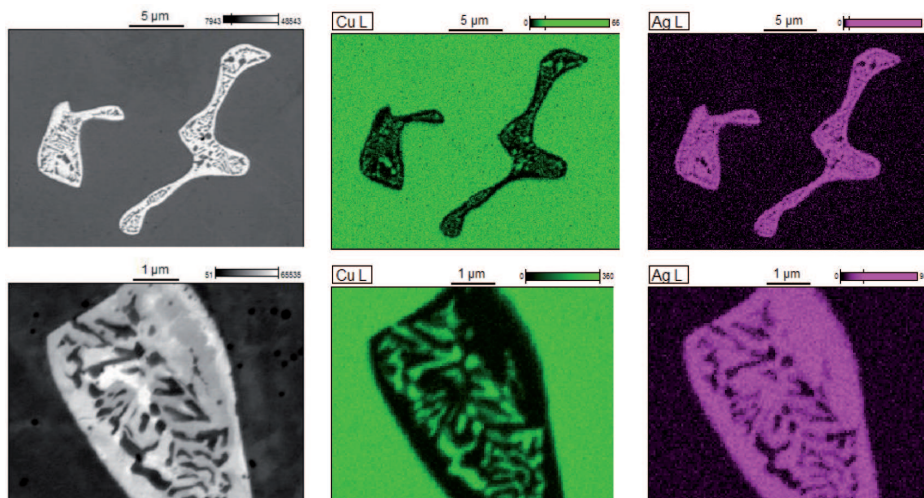


Fig. 3. EDS microanalysis of Cu and Ag concentrations in Cu-15%wt. Ag alloy

In alloying process, an oxygen-free copper produced by KGHM Polska Miedź S. A. was used. The content of silver in copper was conducted in Centrum Badań Jakości Ltd "Legnica" which is a Qualitative Analysis Department localized in Lubin.

In Table 1, detailed chemical compositions of casts is presented. As can be seen, the actual content of silver in both alloys is close to the assumed amount. The analysis showed an insignificant number of pollution with other elements, whose summary number does not exceed 20 ppm.

TABLE 1
Chemical composition of Cu-Ag casts

Alloy elements	CuAg5	CuAg15
	Content, %wt	
Ag	4,84	14,91
Cu	in balance	in balance
Impurities	Content, ppm	
Bi	0,52	0,23
Pb	0,6	0,38
Sb	0,19	0,15
As	0,27	0,25
Fe	1,9	1,23
Ni	0,4	0,27
Sn	0,42	0,34
Zn	1,77	1,02
Cd	1,41	1,36
Co	0,05	0,04
Cr	0,44	0,24
Mn	0,38	0,35
P	1,8	1,34
Se	0,1	0,09
Si	1,7	0,97
Te	0,012	0,014
S	5,5	4,1
Total amount of impurities	17,46	12,37

The density of the materials tested was determined taking into consideration the air and liquid density in the temperature in which the samples were weighed. The measured density was at the level of 8,962 g/cm³ for CuAg5, and 9,118 g/cm³ for CuAg15 alloy.

The examination of castings' hardness in their cross-section was conducted using Brinell's method according to the requirements of PN-EN ISO 6506-1:2006 norm. In the Table 2, the mechanical and electrical properties of the alloys examined are presented.

TABLE 2
Comparison of the mechanical properties of tested alloys after casting

Material	ρ g/cm ³	HB	UTS, MPa	YS, MPa	γ , MS/m	%IACS
CuAg5	8,96	71	222	98	50,5	87,2
CuAg15	9,09	82	287	130	46,6	80,4

3. Research on shaping of mechanical and electrical properties of Cu-Ag alloy drawn wires

Produced wires with casting structure were drawn repeatedly in order to obtain micro-wires with total strain of 6 in the logarithmic scale. During the drawing process, researchers measured strength parameters which enabled them to determine the mechanical properties growth trend. Drawing process was conducted with the use of laboratory drawing machine fitted with force sensor for measuring drawing force, in the range of 1 to 100 kN. Hottinger Spider 8 amplifier was used and along with computer system it allowed to record and process all the measured data. Wires with diameters lower than 0,5mm were drawn on special laboratory micro-drawing machine. Carbide and diamond wire drawing dies were used with a rapeseed oil as a lubricant. Fig. 4 presents characteristic of drawing tension value of specific stages of drawing of CuAg5 and CuAg15 alloys wires.

Fig. 5 presents diagrams showing the comparison of ultimate tensile strength changes in the function of total true strain of CuAg5 and CuAg15 alloy wires. The graph of technological curves of alloys' hardening indicated considerable potential of hardening during cold plastic working. When comparing both alloys, researchers noticed a significant influence of silver addition to copper on mechanical properties of cast as well as to the dynamics of its hardening during deformation. In case of the CuAg5 alloy, the cast shows UTS=207 MPa and YS=77 MPa, and it hardens during drawing to UTS=706 MPa and YS=685 MPa (final wire with 0,5mm diameter). It is nearly 3.5-times increase in drawing stress value, and almost 9-times increase of yield stress.

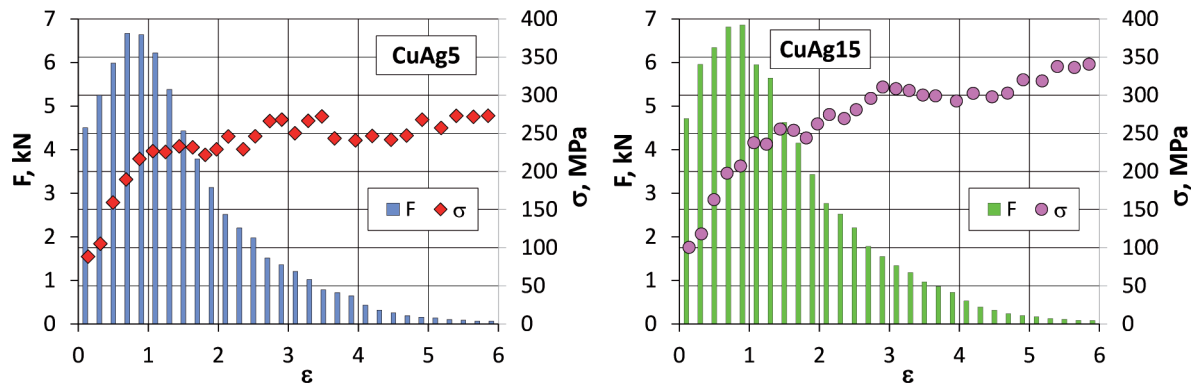


Fig. 4. Characteristics of drawing tension value vs. total true strain after each drawing pass for CuAg5 and CuAg15 alloys wires

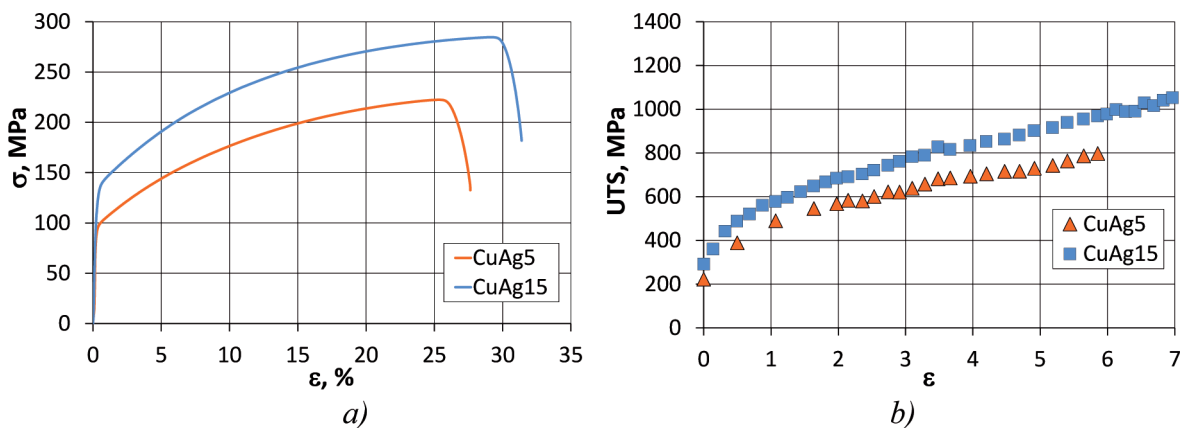


Fig. 5. Stress strain graph during tensile test of rods (a) and comparison of ultimate tensile strength vs. total true strain during drawing of CuAg5 and CuAg15 alloy wires (b)

Similarly, for the CuAg15 alloy, the cast show UTS=289 MPa and YS=122 MPa. After 6 total true strain properties increased up to UTS=927 MPa and YS=905 MPa, which is over 3-times strengthening increase, and over 7-times increase of yield stress of the alloy.

When comparing both alloys, it can be noticed that the initial difference between casts' UTS and YS (82 and 45 MPa respectively), after 6 total true strain increased up to 225 and 220 MPa respectively. The value of total elongation (measured in the tensile test) was at a similar level of 33÷34% and changed similarly in the process of their deformation.

The intensive increase in mechanical properties was accompanied by substantial drop in electrical conductivity of the alloys examined. Resistance of wires was measured after each strain according to the guidelines of PN-IEC 61089:1994 norm. The measurements were performed with a Burster RESISTOMAT®Kelvin bridge, model 2304.

In order to convert correctly the resistance of the alloys into a standard temperature of 20°C, the examination of their temperature coefficient of resistance was

conducted. In order to do that, researchers carried out a series of examinations consisting in determining the impact of temperature on the dynamics of alloys' resistance change. The rate α_R was calculated on the basis of conducted measurements in the temperature range up to 100°C, and amounted to 0,00318 1/K for CuAg5 alloy, and 0,002937 1/K for CuAg15 alloy.

In Fig. 6 the comparative diagrams of electrical properties of alloys examined are presented: graphs concern the influence of strain on conductivity changes, and show the influence of strain on conductivity changes with reference to the percent of pure copper electrical conductivity (according to the international IACS standard).

Plastic working of the CuAg5 alloy with the total amount of true strain at the level of 6 brought the increase in its resistivity by over 11%. In case of the CuAg15 alloy, the increase in resistivity was at the level of 23%. When comparing electrical conductivity of casts of both alloys (CuAg5: 50,68 MS/m and Cu-15%wt. Ag: 46,3 MS/m), and wires 6 total true strain (CuAg5: 45,41 MS/m and CuAg15: 37,4 MS/m), it can be noticed that the initial difference in their electrical conductivity (9%) increased to 21% at the end of drawing.

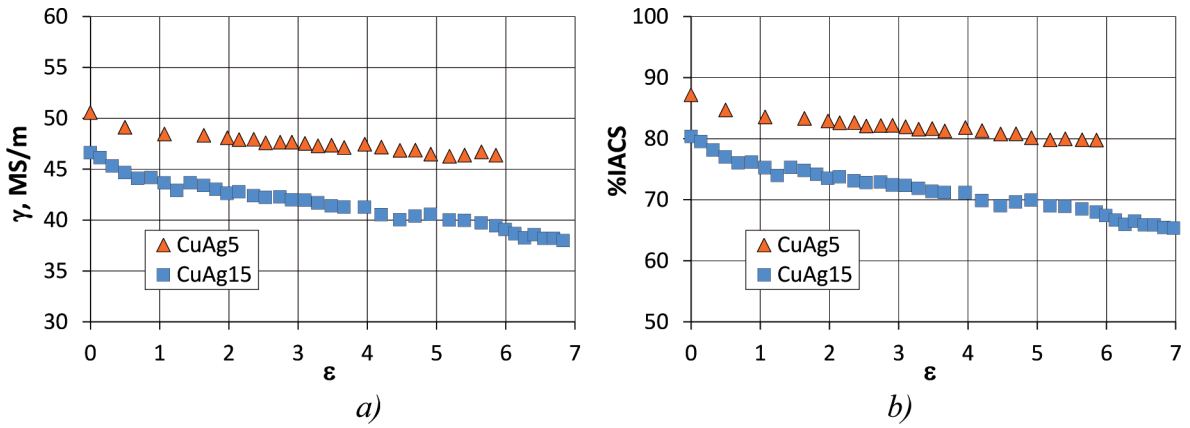


Fig. 6. Comparison of electrical conductivity changes (a) and the international IACS standard (b) in the function of total strain of CuAg5 and CuAg15

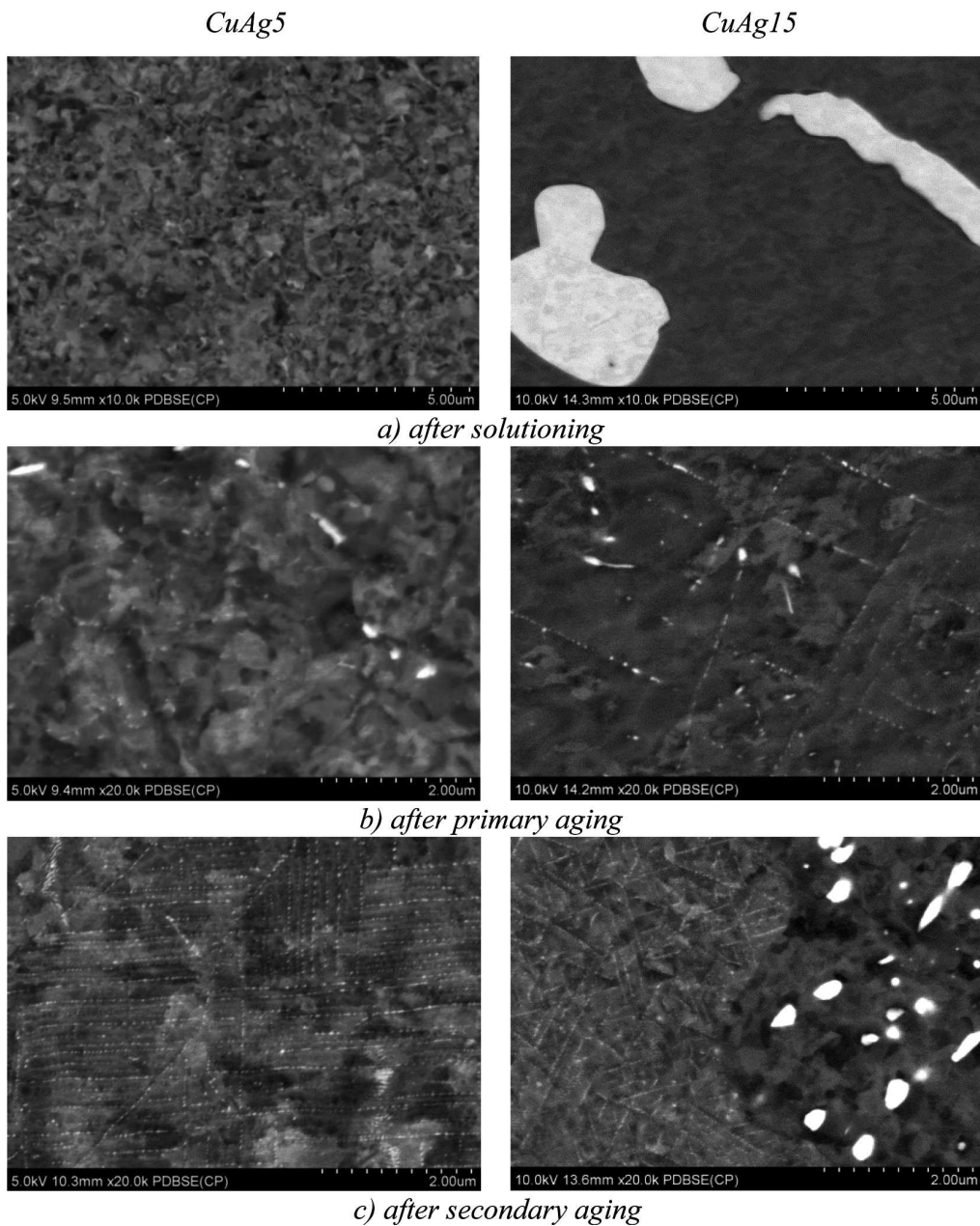


Fig. 7. SEM microstructures of CuAg5 and CuAg15 rods after another steps (a, b, c) of intermediate heat treatment (IHT)

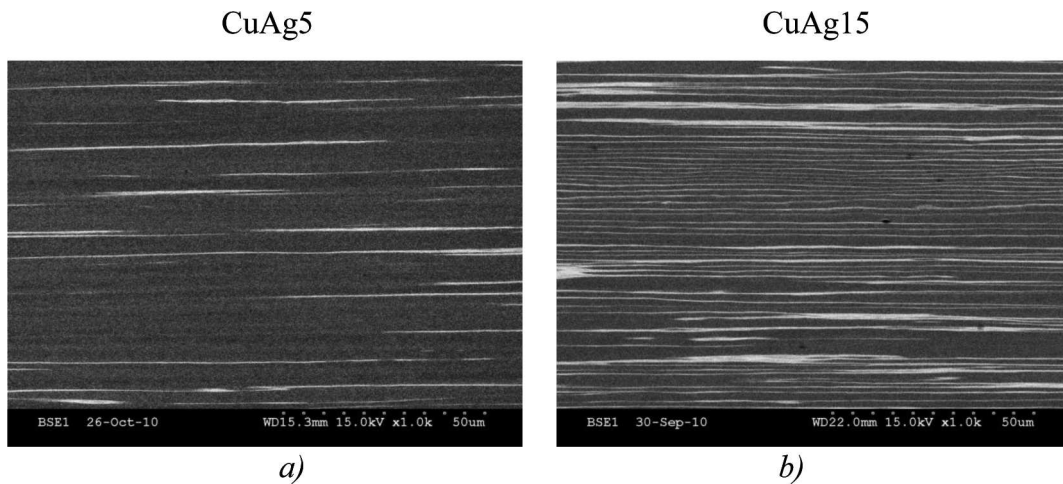


Fig. 8. SEM microstructures in wires made of CuAg5 (a) and CuAg15 (b) alloys in longitudinal section, obtained at the total strain $\varepsilon = 6$

Such extremely high drops in electrical conductivity of examined Cu-Ag alloys can be explained by particularly intensive generation of structural defects, crystal structure deformations, and the increase in dislocation accumulation during plastic working of the materials.

In order to limit the negative influence of strain on electrical properties of wires, as well as to improve their mechanical properties, researchers conducted tests of the influence of preliminary heat treatment of casts before their further plastic working.

On the basis of conducted research, it has been determined that both mechanical and electrical properties can be significantly increased using gradual heat treatment that consisted of water quenching and ageing processes. It is a result of Ag-rich α -phase precipitation from the Cu-rich matrix, and, contrarily, the Cu-rich β -phase precipitation from the Ag-rich areas, as well as the increase in α - and β -phases already emitted.

CuAg5 and CuAg15 alloys' microstructures shown in the Fig. 7 present structural state after each of heat treatment processes used. The initial cast structure is the image of β -phase precipitations (CuAg5), and eutectic precipitations compared to $\alpha + \beta$ phase matrix (CuAg15). The conducted procedure of super-saturation resulted in complete (in case of the CuAg5 alloy), or partial (in case of the CuAg15) solution of silver in copper matrix (cf. Fig. 7). Properly chosen temperature and duration of aging procedure allowed precipitation of numerous and small Ag particles in the volume of supersaturated solution. It resulted in precipitation hardening enabling alloys to obtain improved hardness. An additional procedure of annealing the alloys resulted in growth and coagulation of already initiated Ag precipitation. In the final result, heat treatment of alloys brought about a positive modification of CuAg5 and CuAg15 microstructure. The maximal precipitation of Ag from Cu matrix contributed

to a distinct improvement in electrical conductivity of the alloy. The numerous and small Ag particles which precipitated from Cu matrix caused an increase in mechanical properties of the cast. As it can be observed, the final durability and electrical properties of the cast increased by more than 15% after the heat treatment procedure.

The use of a considerable plastic deformation resulted in growth of the eutectic precipitations length resulting from thermo-mechanical treatment of the alloy. The longitudinal section structure of micro-wires of the 6 approximating total true strain consists of numerous, thin, strongly elongated fibres of silver (almost completely consisting in Ag, the remainder is Cu) in comparison with the matrix almost completely consisting in Cu (cf. Fig. 8). The diameter of those fibres amounts to the value from a few nm to ca 100 nm. The microstructure obtained is the reason for strong hardening of the alloy resulting from a large number of grain boundaries and their deformation level. A composite alloy structure has been generated with a strengthening phase of numerous Ag fibres which allow to obtain high mechanical properties. On the other hand, intensive precipitation of silver contributed to the purification of the solution in which almost only pure copper remained. This resulted in the situation that strongly elongated fibres of silver, together with copper, create a characteristic parallel connection of perfect electrical conductors. In the end, CuAg15 alloy micro-wires attain very high electrical properties.

During Cu-Ag rods' drawing strength parameters of the process increased by several per cents, and the increment in mechanical properties of the alloys deformed as well as somewhat poorer dynamics of the drop in electrical properties of the wires drawn were observed.

Table 3 compares the tensile strength and electrical parameters of CuAg5 and CuAg15 alloy wires obtained

from alloys after casting and exposing them to the intermediate heat treatment.

TABLE 3
Comparison of the strength and electrical parameters of CuAg5 and CuAg15 alloy wires obtained from alloys after casting and exposing them to the intermediate heat treatment (IHT)

Material	UTS, MPa	γ , MS/m	%IACS
CuAg5 as cast	222	50,5	87,2
CuAg5 after IHT	252	54,1	93,1
CuAg15 as cast	287	46,6	80,4
CuAg15 after IHT	376	54,6	94,1

When comparing the characteristics of mechanical and electrical properties of wires obtained from materials after casting and exposing them to the intermedi-

ate heat treatment, a considerable increase in drawing stress of wires of the same total strain can be observed. Fig. 9 and 10 shows the characteristics of changes of tensile strength and electrical properties in the function of the total true strain of alloys after the intermediate heat treatment in comparison with the characteristics of the alloys drawn without heat treatment. It can be seen that the wires after drawing with total true strain at the level of 6 have more than 3-time higher drawing stress along with the same electrical conductivity which was displayed by the rods after casting.

Fig. 11 presents characteristic of electrical conductivity in the function of wires' tensile strength without and with intermediate heat treatment. On this basis, researchers can assess the range of an obtainable set of mechanical and electrical properties of specifically deformed wires.

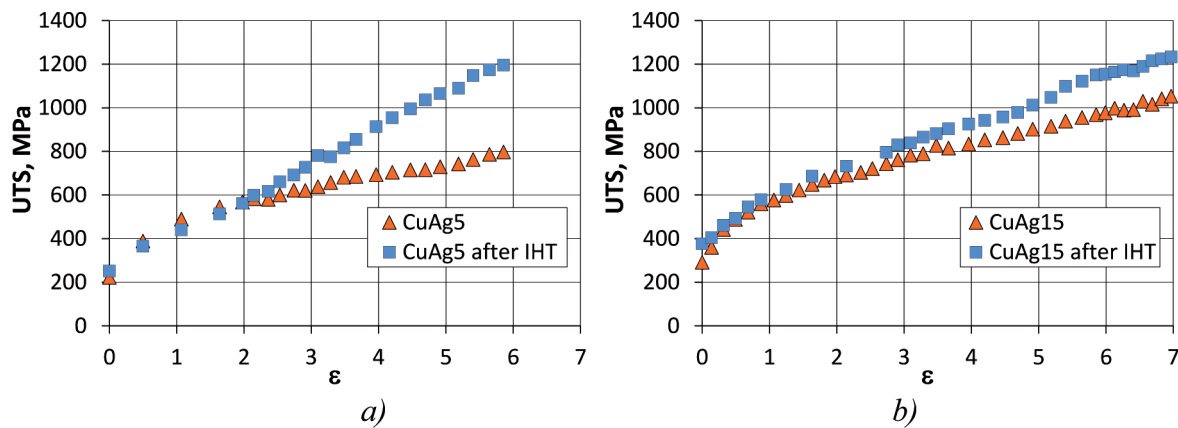


Fig. 9. Influence of total true strain on tensile strength of wires made from CuAg5 (a) and CuAg15 (b) alloys without and with intermediate heat treatment (IHT)

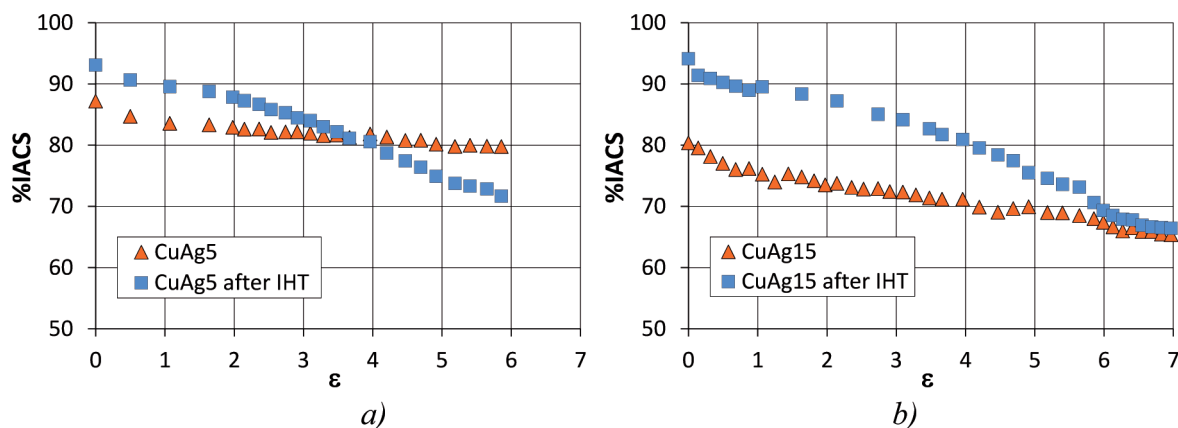


Fig. 10. Influence of total true strain on the change of electric properties of wires made from CuAg5 (a) and CuAg15 (b) alloys without and with intermediate heat treatment (IHT)

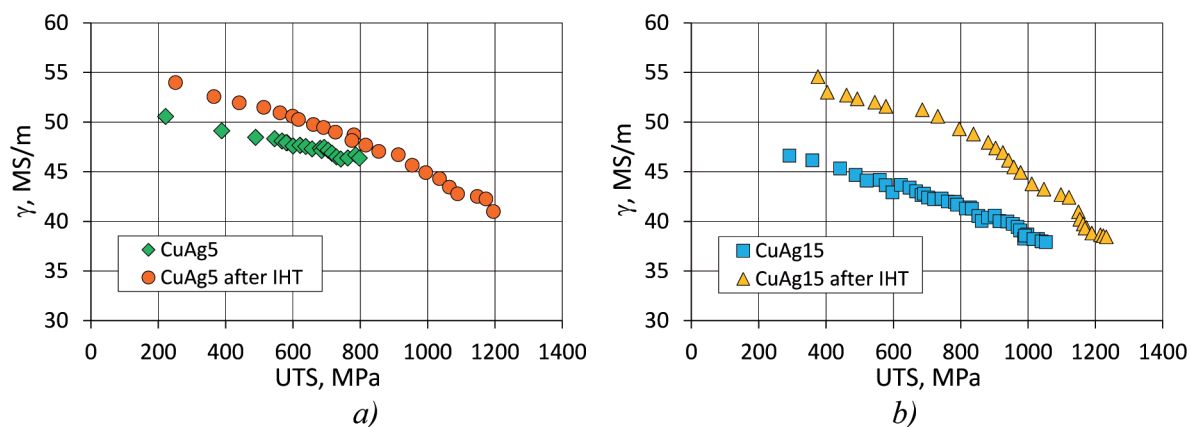


Fig. 11. Characteristic of changes in electrical conductivity in the function of ultimate tensile strength wires made from CuAg5 (a) and CuAg15 (b) alloys drawing without and with intermediate heat treatment (IHT)

Wires of the final diameter of 0,2 mm, and true strain at the level of 6, depending on the production parameters applied, obtained ultimate tensile strength of above 1250 MPa with a simultaneous electrical conductivity above 40 MS/m (CuAg15), or UTS >1100 MPa with γ >46 MS/m (CuAg5).

As the conducted research has shown, a mutual, variable Ag solubility in Cu matrix, as well as Cu solubility in Ag matrix, together with temperature change, gives practical possibilities for improving mechanical and electrical properties of Cu-Ag alloys through mutual reactions of precipitation and drawing.

Currently, further research has been in progress on improving chemical composition of Cu-Ag alloys in order to limit the Ag content (economical factor), and to select most favourable parameters of plastic working and heat treatment, enabling researchers to obtain wires with the set of highest possible mechanical and electrical properties.

4. Final conclusions

In the laboratory line for continuous casting of non-ferrous metals and their alloys, a research material in the form of CuAg5 and CuAg15 alloy rods was attained. The researchers carried out the basic physico-chemical properties of the alloys (chemical composition analysis, density measurement, determining the temperature coefficient of resistance in the range of 20÷100°C).

The research on drawing stress of rods into wires (from the initial state after casting, and after different variants of heat treatment of the cast), and the examination of mechanical properties (UTS, YS, A_{100} , HB) of wires of different level of strain were conducted. The researchers prepared the curves of alloy's hardening after different variants of thermo-mechanical treatment, and

the characteristics of the changes in their electrical conductivity in the function of material total strain.

For the alloys in the initial state and for selected variants of thermo-mechanical treatment, the researchers conducted macro- and micro-observations using a scanning electron microscope (SEM). Furthermore, maps of Cu and Ag distribution in the alloy were created with the aid of energy-dispersive X-ray (EDX) analyzer.

The influence of multi-operational thermo-mechanical treatment on the change in structure, and on the mechanical and electrical properties of the wires has been presented. The modification of the initial casting structure contributed to the generating of composite structure of numerous, strongly elongated silver fibres in the copper matrix. In the presence of the material's 6 total true strain of the numerous, evenly distributed along the silver fibres volume, those fibres (whose final diameter does not exceed 50÷100 nm – the thinnest fibres are several÷dozen or so nanometres wide) created, through numerous grains' boundaries and dislocation accumulation, strengthening phase markedly improving mechanical properties of the alloy. Simultaneously, the proper thermo-mechanical treatment of the alloy provided the optimal Ag precipitation from the Cu matrix. The system of parallel Cu-Ag connections which was created at that time also contributed to attaining relatively high electrical conductivity of wires.

5. Conclusions

1. CuAg5 and CuAg15 alloy rods, obtained as the result of laboratory installation to continuous casting, are characterized by a homogenous chemical composition along their entire length.
2. The examined Cu-Ag alloys show considerable potential for hardening by plastic deformation. At the same time, there is an unexpected high drop in elec-

trical conductivity resulting from a large number of grain limits and dislocation accumulation in the alloy structure. This negative effect of conductivity drop can be reduced by a properly selected heat treatment scheme during plastic working of the alloy.

3. The modification of casting structure resulted in intensification of strain hardening and enabled researchers to obtain wires of particularly high set of mechanical and electrical properties. As it has been proved in this paper, researchers obtained wires that (with total true strain above 6), depending on thermo-mechanical treatment of alloy, show UTS=1050 MPa and $\gamma = 46,4$ MS/m (80% IACS), or UTS=1300 MPa and $\gamma = 37,7$ MS/m (65% IACS).
4. The microstructural result of properly selected thermo-mechanical treatment of CuAg5 and CuAg15 alloys is a structure of numerous, strongly elongated fibres of almost pure silver compared to the matrix consisting almost only of copper. Silver fibres, depending on heat treatment parameters and the degree of deformation, have a diameter of between several÷dozen and so nm to 50÷100 nm, and, by creating composite strengthening alloy phase, are simultaneously an electrical system of parallel links with copper of very high electrical conductivity.

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