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Copper-clad thermally stable Al-Zr wire, produced via copper electrodeposition

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ABSTRACT

Producing of the copper-clad aluminium wire made of Al-0.4Zr alloy via electrodeposition was studied. In order to ensure the adhesion of the copper after deposition, the intermediate Ni layer was applied to the aluminium wire surface. Formation of the composite wire with aluminium alloy core and copper sheath resulted in the increase of the ultimate tensile strength from 175 to 233 MPa, while also slightly decreasing electrical conductivity and notably decreasing ductility from 6 to 3 %. Annealing at 300 °C for 1 hour was performed to the composite wire to increase its ductility. Annealing resulted in the recovery of the mechanical properties back to the level of initial aluminium alloy wire, while electrical conductivity increased by 3.5 % IACS, allowing the newly produced composite wire to compete with the commercially produced copper-clad aluminium wires.

KEYWORDS

aluminium alloys • Al-Zr • copper-clad aluminium • copper-clad aluminium wire • electrodeposition mechanical strength • electrical conductivity • ductility

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Introduction

Currently, the search for cheaper alternatives to copper as a material for electrical applications is a pressing issue in electrical and energy industries. The main disadvantage of copper is its high cost and high relative weight [1]. In order to avoid such complications, the choice of a substitute material is sometimes preferable.

Among the commercially available materials for the electrical purposes the aluminium and its alloys are close seconds after the copper [2–4]. The lower density and thus, lower weight of the products make aluminium attractive in terms of relative electrical conductivity being higher than that of copper alloys [5,6]. Unfortunately, aluminium is also characterized by significantly lower mechanical strength than copper, which limits the area of its application.

Attempts to increase the strength of aluminum by creating alloys based on it or by plastic deformation methods usually lead to a decrease in its electrical conductivity [7–9], and generally don't provide the level of mechanical strength similar to one of copper.

The other way to substitute the full-copper conductors is the use of layered metal composites, consisting of two or more different alloys [10–12]. Layered materials are attractive to researchers and developers since they are able to combine required properties of different alloys without their disadvantages. Layered materials can demonstrate simultaneously low density, high thermal and electrical conductivity, unique mechanical properties compare to monomaterials [13–15]. Implementing such approach to aluminium-copper composites allows to create a material with the reduced by 35–50% weight compared to copper while achieving electrical conductivity and mechanical strength surpassing even the best aluminium alloys [15,16].

Traditionally, materials with aluminum core and copper sheath are called copper-clad aluminium. They have found application in number of devices that require the use of high-frequency electrical currencies, since the main advantage of the copper-clad aluminium is the ability to navigate the electrical current along the thin copper layer, simulating the full-copper conductor with much lighter product [17–19].

The production of bimetallic aluminum-copper wires is associated with a number of difficulties, such as different thermal expansion coefficient, drastically different melting point, inclination towards both the formation of intermetallic of the contact areas and contact corrosion. One of the biggest concerns is the formation of the intermetallic Al-Cu particles that leads to a sharp decrease in the ductility and electrical conductivity of the composite. Annealing that is usually applied to cold-formed copper-clad wires to reduce the level of residual stress can amplify the negative effects mentioned above. Thus, the choice of temperature treatment is an important issue in the production of such materials [15,20].

The most common way of production the copper-clad aluminium wires (CCAW) is the joint deformation of the composite billet [21]. However, this method provides a few complications: necessity of keeping the aluminium-copper interface oxygen-free, the necessity of controlling the thickness and chemical composition of copper layer, the necessity of post-deformation annealing [18–20].

Another, a relatively new approach to obtaining copper-aluminum bimetallic wires can be the electrochemical deposition of copper onto an aluminum wire. Despite presenting a few old patents, the scientific literature is very scarce on this method of producing copper-clad aluminium wires. However, such approach may in theory provide more accurate control of the thickness of both the copper layer and the diffusion layer, as well as the purity of the copper layer.

In this study, an attempt was made to obtain a copper-clad aluminium wire by an electrochemical deposition of a copper onto aluminum alloy core. Usually, the technically pure aluminum is usually used base material [18], but in this work conductive and thermally-stable aluminum alloy Al-0.4Zr was used [22].

Materials and Methods

The Al-0.4 wt. % Zr alloy samples in a form of the rod with a diameter of 11 mm were cold-drawn to a diameter of 3.2 mm at room temperature. Before drawing, the rods were annealed at 375 °C for 120 h. Chemical composition of the aluminium alloy is presented in Table 1.

Table 1. Chemical composition of the Al-0.4Zr alloy

Element	Zr	Si	Fe	Mg	Cu	Zn	Mn	Cr	Ti	V	Al
Composition, wt. %	0.344	0.054	0.169	0.003	<0.002	0.033	0.010	0.001	0.0017	0.0145	99.37

The electrodeposition of the copper onto aluminium was performed in four steps: (i) surface preparation, (ii) surface clarification, (iii) Ni plating and (iv) Cu plating [23].

Surface preparation (i) included chemical degreasing in an alkali solution, subsequent washing in hot and then cold water. Surface clarification (ii) was carried out in 10 % nitric acid solution, also functioning as activation process. The Ni plating (iii) was performed at 40 °C in a sulfuric acid electrolyte ($NiSO_4$) for 3 min at a current density of 5 A/dm^2. The Cu plating (iv) was performed at room temperature in a sulfuric acid electrolyte ($CuSO_4$) for 2 h at a current density of 5 A/dm^2 until the copper layer obtained 90 ± 10 μm thickness. The control of the Cu layer thickness was performed by optical microscopy analysis of cross-sections at different electrodeposition time.

The choice of copper sheath thickness was based on the minimum current frequency that provides the skin effect at such skin depth. According to the skin depth calculations, the 100 μm of copper provides the skin effect at current frequences from 400 kHz and higher, which is the frequency range for the audio cables, audio equipment, high frequency current equipment, wireless charging devices etc. The introduction of the nickel layer is a necessary stage of electrochemical copper deposition onto aluminum. Copper cladding of aluminium without transition layers is possible but complicated by the reasonably high difference in the electronegativity of Cu and Al, causing corrosion on their interface [1]. The thickness of the nickel layer in the state after copper cladding was measured to be around 20–25 μm.

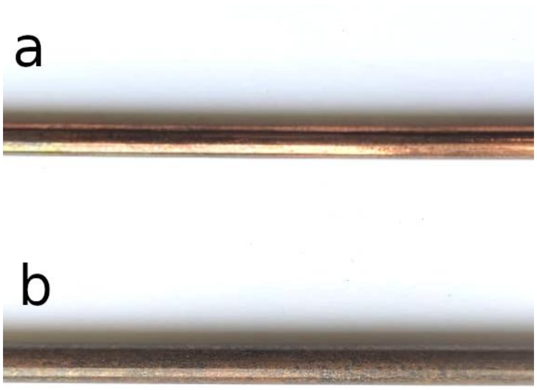

Fig. 1. Appearance of the Al-0.4Zr alloy wire after copper deposition (a) and subsequent annealing (b)

Optical microscopy was performed on an Olympus Q15OR light microscope. Scanning electron microscopy (SEM) was performed on a Tescan MIRA V microscope at an accelerating voltage of 15 kV.

Annealing was performed in atmosphere furnace Nabertherm B180 at 300 °C for 1 h. The choice of time and temperature was based on the study [21]. The samples after annealing were cooled into water to remove the dross from the surface. The appearance of the sample's surface is presented in Fig. 1.

Tensile tests were carried out on an Instron 5589 testing machine at room temperature. At least 3 samples were tested for each condition.

The specific electrical resistance of the material under study was measured in accordance with IEC 60468:1974 [24]. Straightened samples of at least 1 m in length were selected. The electrical resistivity of the studied material was measured in accordance with the IEC 62641:2023 standard [22]. The electrical conductivity value of the samples relative to annealed copper (International Annealed Copper Standard) was calculated using equation:

$IACS = \omega_{Al} / \omega_{Cu} * 100$ [%], (1)

where ω_{Al} is the experimentally determined value of the electrical conductivity of the aluminum alloy sample, ω_{Cu} is the electrical conductivity of annealed copper, equal to 58 MSm/m. Electrical conductivity measurements were taken with an instrument operating at a single frequency of 50 Hz, while skin effect measurements require an instrument operating at varying current frequencies. In addition, the skin effect at a copper layer thickness of about 100 μm begins to appear at current frequencies from 400 kHz [25]. Thus, in this study, the conductivity of the entire wire was measured; the skin effect was not directly measured.

Results and Discussion

Figure 2(a) shows the results of optical metallography of bimetallic wire before and after annealing. According to Fig. 2(a), electrochemical deposition of nickel and copper onto aluminum alloy wire is characterized by the absence of pores, large intermetallic particles or other undesirable defects at both Al-Ni and Ni-Cu interfaces. The Al-Ni interface is characterized by the flake-like particles seemingly protruding into Ni layer, while the Ni-Cu interface appears smooth and continuous.

Annealing (Fig. 2(b)) results in the overall thinning of the copper-nickel shell. Two simultaneous processes occur: Ni-Cu interdiffusion and Cu layer thinning. Ni-Cu interdiffusion is resulted in visible thickening of the Ni layer, while Cu thinning occurs due to the surface oxidation and subsequent dross removal after heat treatment. While total width of Cu+Ni layer decreases from 90.5 ± 2.5 to 73.0 ± 2.5 μm and the thickness of copper layer decreases from 73.0 ± 2.5 to 52.0 ± 2.5 μm, the thickness of the Ni layer seemingly increases from 17.5±2.5 to 21.0 ± 2.5 μm indicating the interdiffusion between Ni and Cu layers.

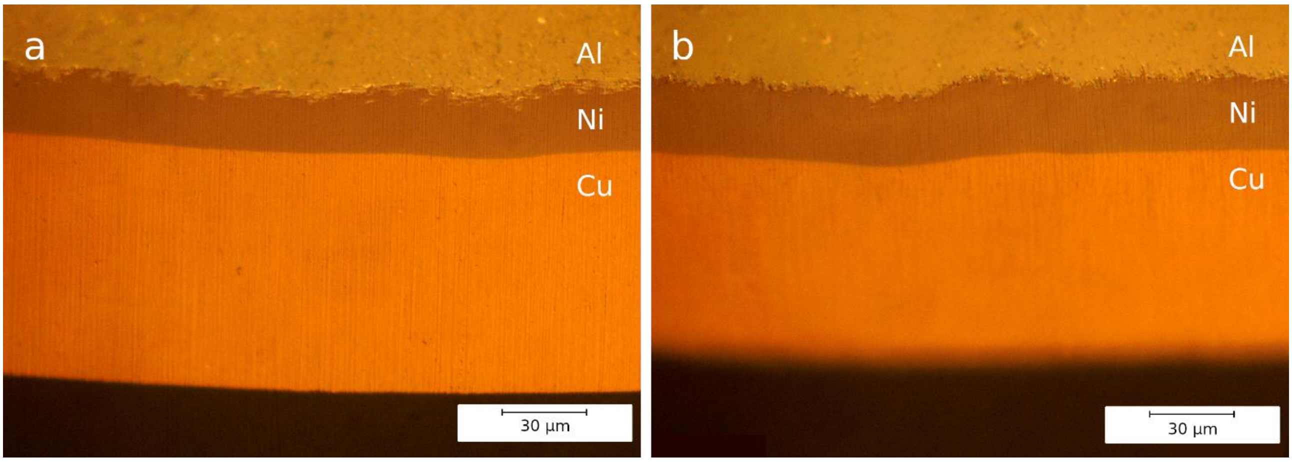

Fig. 2. Optical metallography of the cross-section of an copper-clad Al-0.4Zr alloy wire before (a) and after annealing (b)

The recommended fraction of the copper in aluminum-copper wires lies within the interval of 10–15 % [13,26]. In current study, the copper fraction are 8.4 and 6.1 % before and after annealing respectively (Table 2). The decrease in the copper content due to thinning of the copper layer occurs due to oxidation of the outer layer of the wire during annealing and the formation of carbon/scale on the copper surface. As a result of rapid cooling in water, the scale is removed from the surface, taking with it a certain amount of the copper that has entered the reaction. This effect is well known and described in literature.

Table 2. Changes in the Ni-Cu layer thickness during the annealing

State	Layer thickness, µm			Fraction in the cross-section, %		
	Ni+Cu	Ni	Cu	Ni+Cu	Ni	Cu
Al-0.4Zr copper-clad	90.5±2.5	17.5±2.5	73.0±2.5	10.3±0.3	1.9±0.3	8.4±0.3
Al-0.4Zr copper-clad and annealed	73.0±2.5	21.0±2.5	52.0±2.5	8.6±0.3	2.5±0.3	6.1±0.3

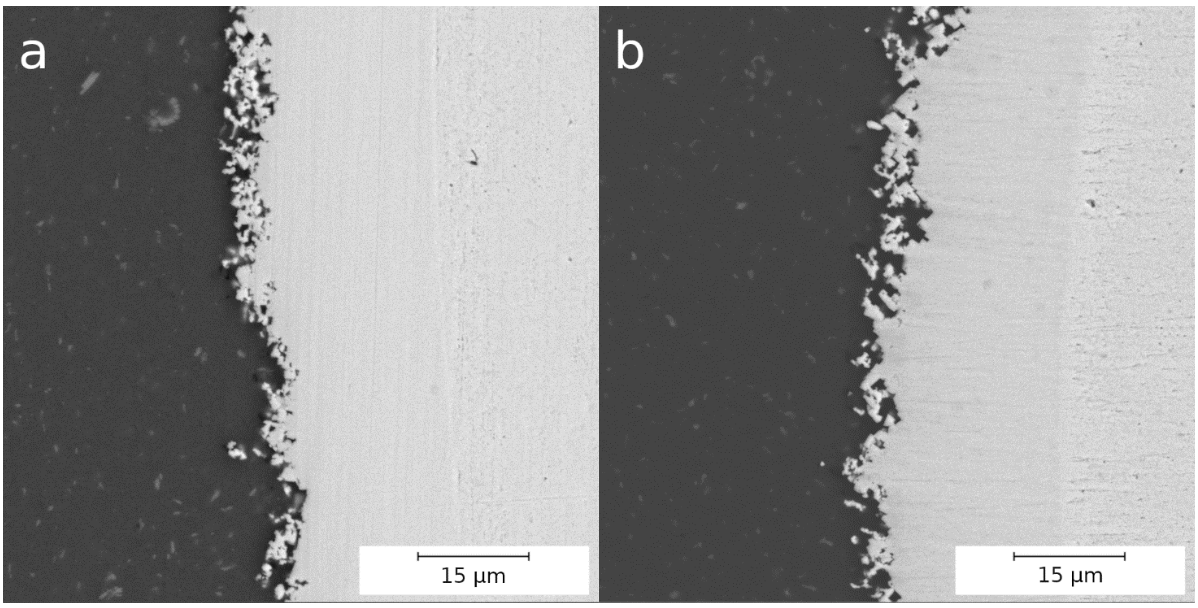

Fig. 3. Al-Ni-Cu interface in the wire after copper electrodeposition (a) and subsequent annealing (b), SEM

The results of the SEM examination of the wires' cross-section are presented in Fig. 3. The Al-Ni interface is characterized by the distinct flakes of Ni or Ni-based phase particles. The interface is also uneven which can be attributed to the initial surface of the Al alloy wire, that was not subjected to any addition surface treatment after cold drawing. Within the Al alloy wire volume intermetallic particles are clearly seen. The nature of these particles is most probable. Al_3Fe, $Al_{12}Fe3Si$ or $Al_9Fe_2Si_2$, forming in 1xxx series and Al-Zr system alloys, that conventionally have certain amount of Fe and Si in their chemical composition [4], just like in this study (Table 1). Since it was not the subject of this study, the exact phase composition of the Al wire was not established. The Ni-Cu interface is smooth and only distinguishable by the fact that copper, being softer material, was more roughly polished rather than Ni.

Annealing at 300 °C for 1 hour resulted in coagulation of the flakes at the Al-Ni interface, as well as in increased porosity of the Cu layer (Fig. 3(b)).

Table 3 presents the properties of Al-0.4Zr wire samples before and after copper deposition. The ultimate tensile strength (UTS) of Al-0.4Zr alloy wire is 175 MPa, level of electrical conductivity is 58.9 %IACS. Such combination while quite high in both parameters is not unique for aluminium alloys. The relative elongation before failure of Al-0.4Zr wire is decent – 6.1 %, allowing this composite wire to be recommended for practical applications. The formation of the Ni+Cu layer resulted in the moderate electrical conductivity decrease down to 57.1 %IACS with simultaneous increase in UTS up to 233 MPa, also significantly reducing the composite's ductility down to 3.1 %. The introducing of copper into composite material led to increase in mechanical and electrical properties, while either introducing the Ni or Al-Ni intermetallic particles resulted in the drop of ductility. In order to increase the ductility level, the annealing of the copper-clad aluminium alloy wire was performed.

As a result of thermal treatment, the ductility and the UTS of the material increased to the initial level (6.3 % and 176 MPa, respectively), electrical conductivity exceeded the original wire level (62.6 % IACS). The tensile strength of the composite wire after annealing has decreased, though remaining within the error limits relative to the original uncoated Al-0.4Zr wire.

The most probable reason for the decrease in electrical conductivity is the presence of a nickel layer between the copper and aluminum layers. In addition to the fact that the electrical conductivity of nickel itself is very low, nickel forms a number of continuous solid solutions with copper, on the one hand, and a number of intermetallic phases with aluminum, on the other. Both the solid solution and the intermetallic phases are characterized by reduced electrical conductivity relative to the base material. All of the above reasons could, both together and separately, negatively affect the electrical conductivity of copper-plated wire. The increase in electrical conductivity due to annealing occurred, most likely, due to the processes of recovery and recrystallization in the aluminum alloy, and the processes of recovery in the copper layer. Annealing at such parameters, as shown in the literature, is insufficient for noticeable changes to occur at the interface of materials, therefore, a potential decrease in electrical conductivity due to these processes was not recorded.

Similar method of copper deposition was applied to the Al-0.5 wt. % Fe alloy, produced by electromagnetic casting [27]. While having almost the same level of electrical and mechanical properties in the initial state (cold-drawn wire), Al-Zr based bimetallic wire demonstrates both higher electrical conductivity and ductility in the annealed state (Table 3).

Table 3. Physical and mechanical properties of copper-clad Al-0.4Zr alloy wires

Sample	Electrical properties		Mechanical properties		
	RER, $\Omega \cdot mm^2/m$	IACS, %	σ_{YS}, MPa	σ_{UTS}, MPa	δ, %
Al-0.4Zr wire	0.02929	58.9±0.4	157±3	175±4	6.1±0.5
Al-0.4Zr (copper-clad)	0.03020	57.1±0.5	167±19	233±11	3.0±0.5
Al-0.4Zr (copper-clad) annealed	0.02754	62.6±0.6	153±3	176±6	6.3±0.3
AW [29]	0.02952	58.4±0,4	170±12	204±14	5.3±0.2
CCAW [27]	0.02996	57.5±0.5	185±6	187±7	1.5±0.1
CCAW-A [27]	0.02831	60.9±0.3	135±5	184±6	4.3±0.6
Al-10%Cu hard-drawn [28]	-	>62.9	-	110−205	>1
Al-10%Cu annealed [28]	-	>62.9	-	135−170	>5-15
Al-15%Cu hard-drawn [28]	-	>64.4	-	110−205	>1
Al-15%Cu annealed [28]	-	>64.4	-	135−170	>5−15

According to the commercial prospects the requirements for the copper-clad aluminium wires are as follows: electrical conductivity is 62.9...64.4 %, tensile strength is 110...205 MPa, and for ductility is 1...15 % [28], depending on the state (hard-drawn or annealed) and copper content.

The copper-clad Al-0.4Zr alloy wire after annealing demonstrates the combination of UTS, electrical conductivity and ductility on par with the commercially produced Al-10 %Cu copper-clad wire in the annealed state while having almost twice as low copper content (6.1 %) (Table 3).

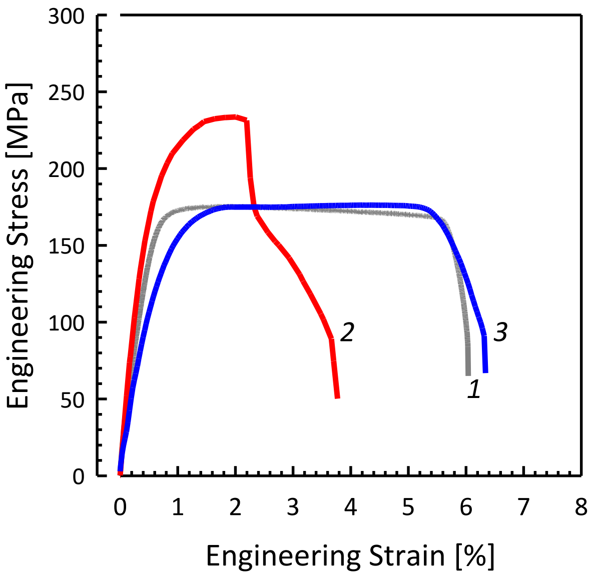

Fig. 4. Engineering stress-strain curves of initial Al-0.4Zr wire (1), copper-clad wire before (2) and after annealing (3)

Figure 4 shows the engineering stress-strain curves of copper-clad Al-0.4Zr wire samples before and after annealing, as well as the data for the original cold-drawn wire. The tensile test for the uncoated wire is represented by the curve characteristic for the aluminium alloys. Samples obtained by the electrodeposition method are characterized by the higher ultimate tensile strength and two-stage fracture – one being caused by the Cu-Ni layer fracture, and the latter by the fracture of aluminium wire itself. Such behavior during tensile tests contributed to the decrease of the elongation to failure.

Annealing at 300 °C for 1 h results in the staged character of the tensile test curve, meaning that the fracture of the wire occurs as a whole. The level of ultimate tensile strength and elongation to failure of the CCAW-A returns to the levels of initial wire, with only exception being lower level of yield stress.

The results of fractographic analysis are presented in Fig. 5. Peeling of the Ni+Cu layer from the Al alloy core could be observed in the as-deposited state. The Ni+Cu layer doesn't function as a whole having seemingly brittle cracks. Such cracks are not observed in the annealed stste (Fig. 5(b)) although the disjoinment of the Ni+Cu and Al alloy layer occurs as well.

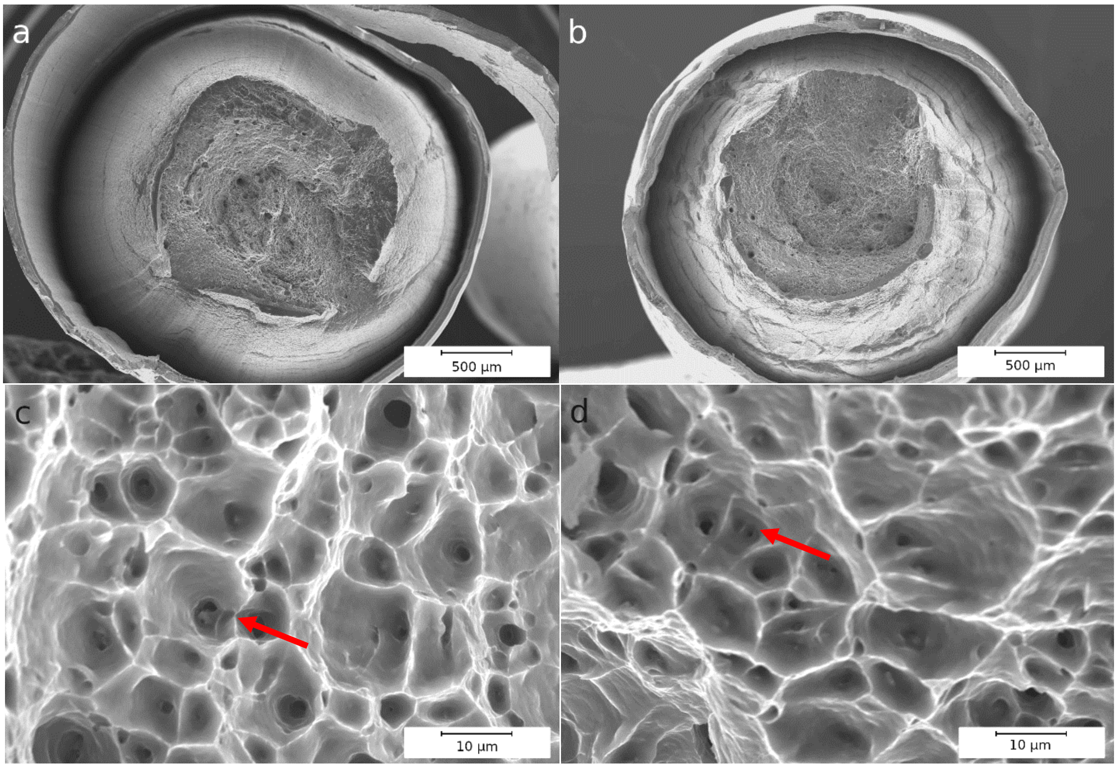

Fig. 5. Fractographic images of the copper-clad wire before (a,c) and after annealing (b,d), SEM

The detailed study of the aluminium alloy wire core demonstrats characteristic dimple fracture both in samples before (Fig. 5(c)) and after annealing (Fig. 5(d)), which along with the evidence presented before allows to establish the ductile nature of the aluminium alloy wire core fracture.

Figure 5(d) demonstrates the presence of the coarse particles in the root of the certain dimples, meaning that the fracture starts at them.

The fractographic images of the Cu-Ni alyer in the samples before and after annealing are presentyed in Fig. 6. Ni layer in sample before annealing demonstrates the clean, flat surface, indicating brittle character of the layer fracture. Cu layer fracture surface is characterized by the presence of so-called "river patterns" which also indicate the brittle nature of the fracture [30]. At the same time Cu layer is also characterized by the wave-like patterns on the fracture surface. Since Cu layer has multiple pit-like lines along the surface, it would be safe to assume that the fracrure started in the Ni layer and the transferred to the Cu layer, in which the fracture occurred in semi-brittle way.

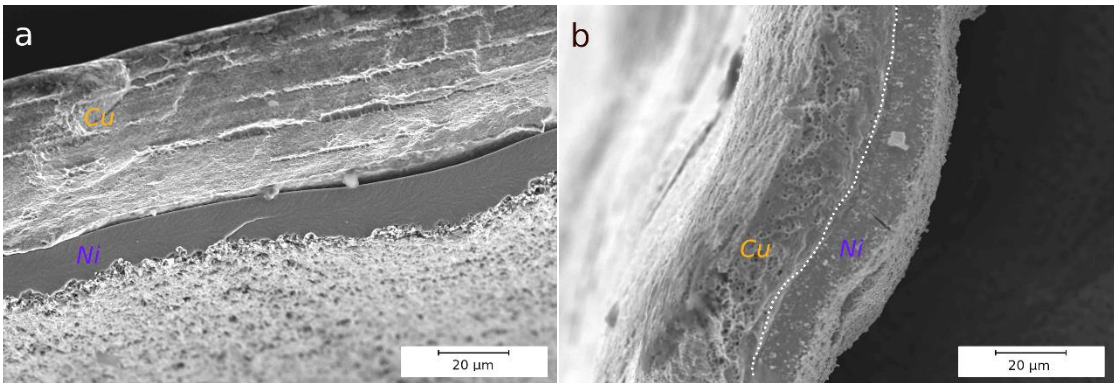

Fig. 6. Fractographic images of the Cu-Ni layer in the copper-clad wire before (a) and after annealing (b), SEM

Fracture surface of the Cu-Ni layer after annealing is presented in Fig. 6(b). Surface of both Ni and Cu layer looks spongy, most likely due to the porosity that was formed during the annealing – no protective atmosphere or vacuum was applied, thus the oxygen migration into Cu and Ni layers occurred. Despite Ni layer having flakes on the fracture surface, it also posesses the fracture, demonstrating the semi-brittle type of the fracture [31].

Conclusions

This paper studies the novel method of obtaining copper-clad aluminium wires via electrodeposition. The thermally stable Al-Zr alloy was used as a base material for the copper electrodeposition. The choice of the Al alloy was dictated by the necessity of increasing the overall strength and thermal stability of the composite wire. The proposed method is promising for producing copper-coated aluminum wires due to ability to precisely control the copper layer thickness down to tens of microns, as well as the chemical composition of this layer.

1. Samples of a copper-clad wire based on Al-0.4Zr alloy wire coated with copper using the electrodeposition method were obtained. The Ni layer was applied between aluminium and copper layers in order to eliminate electrochemical corrosion between aluminium and copper. The copper content in the cross-section is about 8 %, and there are no visible defects/intermetallic particles at the metal interfaces.

2. Electrochemical copper deposition onto Al-0.4Zr aluminum wire while having positive effect on mechanical strength and electrical conductivity, significantly (twofold) reduced the ductility of the composite wire, as well as causing the brittle nature of the

samples fracture. Annealing of composite wire at 300 °C for 1 h resulted in an increase in electrical conductivity up to 62.6 % IACS, while relative elongation before failure and UTS values recovered to the initial wire level – 6.3 % and 176 MPa, respectively.

3. The studied composite wire demonstrates the combination of UTS, electrical conductivity and ductility on par with the commercially produced Al-10%Cu copper-clad wire in the annealed state. Change of the core material from commercially pure Al to thermally stable Al-Zr alloy allowed to achieve this with almost twice as low copper content (6.1 %). Moreover, due to the use of Al-Zr alloy, the resulting wire will retain its strength properties stable even at elevated temperatures, at least up to 150 °C.

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