Structure, thermal stability and mechanical properties of composite wires made of conducting microalloyed aluminum alloys

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Abstract. Thermal stability of the structure and mechanical properties of composite wires made of microalloyed aluminum alloys were studied. The wire was produced by co-rolling of the copper-coated aluminum alloy. It has been demonstrated that annealing at 200 °C for 200 hours leads to the onset of recrystallization, reduced hardness and strength of the wire as per the Hall–Petch equation. The grain size in the annealed wire was less than 1-2 μm, manifesting the stabilized effect of precipitates of Al₃(Sc,Zr) phases. At that the rate of diffusion of copper into the aluminum was quite low. The formation of a uniform fine-grained structure and zero copper-induced embrittlement resulted in increased by up to ~10% plasticity of the wire.

1. Introduction
Small-sized (with a diameter of less than 0.5 mm) composite wires are one of the key elements of modern aircraft and spacecraft electric systems, they are thereby subject to stricter requirements concerning electric conductivity, strength, and thermal stability [1-5].

Today, the Russian aircraft industry uses small-sized composite wires made of 01417 aluminum alloy (developed by All-Russian Institute of Light Alloys) with the addition of 7-9% cerium, lanthanum, and praseodymium, the surface of which has a protective copper coating that ensures better corrosion resistance and facilitates sealing of the wire into connectors. Working on similar tasks, the French manufacturer Filatex uses 1%Mg alloys (similar to the Russian-produced AMg1 alloy) [6].

Low strength of 01417 coarse-grained aluminum alloy, low resistance to wire twisting, characterized by fatigue resistance of the material, modest electric conductivity, and low thermal stability pose the key scientific and technical challenges in the development of new ultralight fine composite "coated aluminum" wires.

One of the biggest challenges of modern electrical engineering is to replace copper wires with aluminum ones while ensuring the same level of thermal stability, high electrical conductance and strength at elevated temperatures. It is particularly challenging to ensure this behavior in small composite "coated aluminum" wires, where prolonged exposure to elevated temperatures leads to an intensive diffusion of copper or silver from the coating into the aluminum wire, rendering it brittle. In
some cases, in order to prevent the diffusion of copper or silver atoms into the aluminum wire, various buffer subcoatings are required.

The purpose of this study is to investigate the thermal stability of fine-grained microalloyed aluminum alloys and small-sized composite wires made of such alloys.

2. Methods and materials

The research focused on the structure of small-sized composite "coated aluminum" wires with a diameter of 0.2-0.5 mm, produced by co-rolling in grooves at room temperature. Microalloyed magnesium-free aluminum alloys with a varying content of scandium, zirconium, hafnium, and ytterbium were used as base materials. Samples of alloys measuring 22×22×200 mm were produced by induction casting, using an INDUTHERM VTC-200 casting machine. High-purity copper was used for coating. Small composite wires were produced by co-drawing at room temperature.

Industrially produced by FSP-One Inc. (France), a small aluminum copper-coated composite wire (with a diameter of 0.126-0.129 mm) with a silver subcoating was used for benchmark studies. This wire is used in electric systems of modern European civil aircraft (A380, etc.). As per the supplier certificate, this wire has a silver coating (over 2 μm thick) and a copper buffer subcoating, high electrical conductance (of at least 60% IACS, which corresponds to 2.676 μΩ·cm), and satisfactory strength (tensile strength of 138 MPa, elongation of at least 6%). There are no thermostability requirements. Coatings are electrodeposited while wires are drawn.

A Lloyd Instruments LR5KPlus universal materials testing machine was used to study rupture strength of the wire. Special grips were used to stretch-test the wire. NEXYGEN MT software was used to control the test machine and to process test results. All tests were performed at a crosshead speed of 10 mm/min. For the purposes of statistical validity, at least three samples in each batch were tested. Microhardness measurements were taken with an HVS-1000 microhardness tester. Microstructure was analyzed using a Jeol JSM-6490 scanning electron microscope (SEM) with an Oxford Instruments INCA 350 EDS microanalyzer. Specific electrical resistivity (SER) was measured using the eddy current method with a SIGMATEST 2.069 instrument. The wires were annealed in an air furnace.

In order to study the thermal stability of the structure and mechanical properties of the wires as well as the kinetics of the copper diffusion into the surface of the fine-grained aluminum wire, the samples were subjected to annealing in sealed glass capsules.

3. Experimental results

It is found that, in its initial state, the commercial wire manifests intense interdiffusion of copper into the silver buffer subcoating as well as interdiffusion of aluminum into the silver subcoating, and silver diffusion into the surface of the aluminum wire. The wire has a fine-grained homogeneous microstructure with 2-3 μm grain size in the central part of the wire. Microhardness of the surface layer was 20-25% higher than that of the central part (see figure 1c). Besides, energy-dispersive spectroscopy detected intermetallic particles of Al-Cu and Al-Fe in the aluminum matrix. All these factors, in our opinion, suggest that the wire was manufactured by hot deformation.

It has been demonstrated that long-time annealing at 200 °C leads to the diffusion of copper and silver into the surface of the commercial aluminum wire. The impact of the annealing temperature and time on its microhardness, strength, and plasticity was as follows. It has been found that annealing for up to 500 hours leads to a sharp decrease in the composite wire plasticity with an increase in tensile strength and a decrease in microhardness in the central part of the wire by 15-20%. The results of energy-dispersive spectroscopy suggest that such a behavior was caused by the intense diffusion of copper and silver into the aluminum wire surface.

Induction casting was used to obtain aluminum alloys with a total content of rare earth elements (REE) and transient metals (TM) of less than 0.4 wt%. Al-0.4%Zr alloy, where the zirconium part (0.1, 0.2, 0.3%Zr) was being progressively substituted by 0.1%Sc, 0.1%Hf, and 0.1%Yb microadditives, was used as the base material. The selection of the casting modes and the chemical
composition of the alloy was determined in a way which minimizes the volume of \( \text{Al}_3(\text{Zr,REE,TM}) \) primary phases, which could cause the wire to break during cold rolling. In order to define the maximum acceptable value, the concentration of the selected alloying agent in pure aluminum was varied in increments of 0.03-0.05 wt% under the selected casting modes, while the presence and size of particles was recorded for three sections throughout the height of the bar – bottom, central, and upper part (near the head). Wires made of A99 aluminum were also used for comparative purposes.

![Figure 1](image1.png)

**Figure 1.** EDS results (a), microstructure image (b) and microhardness measurements (c) of the surface layers of the composite wire produced by FSP-One Inc. (France)

The aggregated findings suggest that for most alloys the presence of primary phases is a consequence of partial decomposition of a supersaturated solid solution under crystallization. This leads to a decrease in the specific electrical resistivity (SER) of the alloy – the SER value appears to be below the theoretical value \( (\rho_{th}) \), calculated using the additivity rule.

![Figure 2](image2.png)

**Figure 2.** Photo of an Al-0.3Zr-0.1Yb bar (a), typical macrostructure of the cross-section after annealing (b), and after cogging with subsequent annealing (c)

The bars were subjected to severe plastic deformation followed by recrystallization annealing in order to eliminate the heterogeneity of the chemical composition and microstructure, resulting in a decrease in the technological plasticity of alloys (figure 2c). The annealing conditions were selected individually for each alloy to ensure a homogeneous recrystallized structure with a specific electrical resistivity setpoint (no more than 2.8-2.9 \( \mu \Omega \cdot \text{cm} \)). The dependencies presented in figure 3 demonstrate that annealing at 320 °C leads to a fairly noticeable increase in electrical conductance for most of alloys. This goes to prove that the solid solution decomposition begins, and \( \text{Al}_3(\text{REE,TM}) \) precipitation starts. Most significant changes in electrical conductivity are observed after annealing cast scandium-containing alloys: \( \mu \text{Al}-0.3\text{Zr}-0.1\text{Sc}, \mu \text{Al}-0.2\text{Zr}-0.1\text{Sc}-0.1\text{Yb}, \mu \text{Al}-0.2\text{Zr}-0.1\text{Sc}-0.1\text{Hf}, \) and \( \mu \text{Al}-0.1\text{Zr}-0.1\text{Sc}-0.1\text{Hf}-0.1\text{Yb} \). Similar electrical conductance behavior at this stage for all the alloys with an addition of 0.1 wt.% Sc allows an assumption that during low-temperature annealing (320 °C, 2 h), \( \text{Al}_3\text{Sc} \) precipitates are formed. This assumption is further corroborated by a decrease in electrical
conductance after heating these alloys to temperatures above 450°C. In our opinion, the lower electrical conductance can be caused by the partial dissolution of Al₃Sc particles at higher heating temperatures. This increases the concentration of scandium in the solid solution and reduces specific the electrical resistivity as a result. It should also be noted that the solid solution decomposition in scandium-containing alloys begins at a lower temperature (275 °C) than in alloys with a higher zirconium content. In our opinion, this result shows that Al₃Sc and Al₅(Zr,REE,TM) precipitates are formed independently in the alloys under consideration.

Figure 3. Dependence between the electrical conductivity of the bars made of new conducting alloys and the temperature of two-hour-long annealing

The workpieces were then scalped to remove oxides and subjected to multistage cold rolling in grooves. No intermediate annealing was applied in order to prevent copper diffusion into the surface of the aluminum wire. Rolling was performed on copper-coated workpieces. As a result, samples of composite aluminum wires with a diameter of 0.26 mm and a copper coating approx. 10 μm thick were produced (figure 4). Rolling to a diameter under 0.5 mm caused isolated wire breaks due to single micropores and submicron Al₅(Zr,REE,TM) phases in the central part of the bar.

Figure 4. Prototypes of small-sized composite aluminum wires obtained from the new conducting alloys
Mechanical tensile tests of the wires at room temperature demonstrate that they are quite strong – after drawing, the tensile strength of the wires made of the new alloys exceeds 500 MPa. The hardness of the wires made of Al-0.3Zr-0.1Sc and Al-0.2Zr-0.1Sc-0.1Hf alloys runs to 480-491 MPa. There is virtually no steady plastic flow in stress-deformation diagrams, which indicates low plasticity of the work-hardened metal. After long-term heat resistance tests at 200 °C (200 h), the wire strength reduces significantly, demonstrating, however, very high plastic performance, which implies that copper diffusion from the buffer layer causes only insignificant embrittlement (figure 5a). The hardness of the aluminum wire drops slightly during annealing – for example, during annealing at 200 °C (200 h), the hardness of the wire made of pure A99 aluminum is reduced from 226 MPa to 190 MPa while for the hardness of the wire made of Al-0.3Zr-0.1Sc alloy goes from 480 MPa down to 436 MPa. As figure 5b shows, post-annealing hardness distribution in the cross-section of the wire remains uneven, with hardness near the surface layer 50-70 MPa higher than in the central part.

Figure 5. Tensile stress-deformation diagrams of the small wire samples (a), microhardness distribution (b) and microstructure (c) over a cross section of small wire prototypes

The tensile strength of the wire made of Al-0.3Zr-0.1Sc alloy is reduced to 160-170 MPa after annealing at 200 °C (200 h), whereas the percentage elongation to fracture increases by up to ~10%.

The fracture analysis has shown that the annealed samples after tensile tests demonstrate ductile failure. No detachment of the copper buffer layer from the aluminum wire surface was observed. The results of energy-dispersive spectroscopy showed that the depth of copper diffusion into the surface of the aluminum alloy never exceeded 10 μm.

The microstructural analysis demonstrated that prolonged annealing at 200 °C leads to secondary recrystallization, which in turn causes an increase in the average grain size in the aluminum wire (see figure 5c). The average grain size in the wire made of Al-0.3Zr-0.1Sc alloy increases from 0.2-0.3 μm in the initial state up to ~1-2 μm after annealing at 200 °C, 200 hours (see figure 5c). After similar annealing, the average grain size in the wire made of pure A99 aluminum exceeds 200 μm. This result suggests that during prolonged annealing, partial decomposition of the solid solution and precipitation of Al3Sc and Al3Zr particles of nanoscale and submicron sizes R occur [3]. In accordance with the Zener equation (dR = α·R/fv, where dR is the stable grain size, fv is the volume ratio of precipitated particles, α is the numerical factor), this stabilizes the fine-grained structure of the aluminum alloy.

In our opinion, the formation of a homogeneous recrystallized fine-grained microstructure in the wire contributes to an increase in the plastic flow stability of the aluminum wire during tensile tests, even if accompanied by a noticeable decrease in strength (see figure 5a). In our opinion, this marked decrease in strength can be due to the simultaneous onset of recrystallization processes, which leads to a lower tensile strength in accordance with the Hall–Petch equation: σb = σ0 + K·d−1/2, where σ0 is the yield stress, K = 0.68 MPa·m1/2 [3] is the grain boundary strengthening coefficient (Hall–Petch coefficient). Inserting the experimental values of grain sizes (d1 = 0.2-0.3 μm, d2 = 1-2 μm) into the Hall–Petch equation, it can be demonstrated that at σ0 = const, the recrystallization process should lead
to a decrease in the tensile strength from ~ 500 MPa to 190-270 MPa. The estimates are quite close to the experimentally measured value $\sigma_b = 160-170$ MPa (see figure 5a). In our opinion, the lower experimental values of tensile strength (160-170 MPa) compared to the calculated value (190-270 MPa) can be due to partial coagulation of Al$_3$(Sc,Zr) particles during prolonged annealing. In accordance with the Orowan equation [6], this leads to a decreased contribution of the second phase particles to the aluminum alloy strength.

4. Conclusions
The thermal stability of the structure and mechanical properties of small composite copper-coated wires made of microalloyed fine-grained aluminum alloys have been studied. It has been shown that the alloys have high thermal stability at long-term isothermal exposure within the temperature range of 180-200 °C. It has been confirmed that annealing is accompanied by recrystallization processes that impair the mechanical properties of the wire. The rate of copper diffusion into the surface layers of the aluminum wire is negligible.

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References
[1] Yang C, Masquellier N, Gandiolle C and Sauvage X 2020 Multifunctional properties of composition graded Al wires Scripta Materialia 189 21-4.
[2] Medvedev A, Arutyunyan A, Lomakin I, Bondarenko A, Kazykhanyan V, Enikeev N, Raab G and Murashkin M 2018 Fatigue properties of ultra-fine grained Al-Mg-Si wires with enhanced mechanical strength and electrical conductivity Materials 8 1034.
[3] Nokhrin A, Shadrina I, Chuvil’deev V and Kopylov V 2019 Study of structure and mechanical properties of fine-grained aluminum alloys Al-0.6wt.%Mg-Zr-Sc with ratio Zr:Sc = 1.5 obtained by cold drawing Materials 12 316.
[4] Moisy F, Gueydan A, Sauvage X, Keller C, Guillet A, Nguen N, Martinez M and Hug E 2018 Elaboration of architectured copper clad aluminum composites by a multi-step drawing Process Materials Science Forum 941 1914-9.
[5] Matveyev Y A, GavriloVA V P and Baranov V V 2006 Light conducting materials for aircraft wires Cables and Wires 5 22-3 (in Russian).
[6] Martin J W 1980 Micromechanisms in Particle-Hardened Alloys (Cambridge, UK: Cambridge University Press) 167 p.