THERMAL STABILITY OF ELECTRICAL CONDUCTIVITY AND MECHANICAL PROPERTIES OF THIN WIRES FROM ALUMINUM ALLOYS Al–0.25%Zr–(Si, Er, Hf, Nb)

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Abstract. The thermal stability of thin wires made of aluminum alloys Al–0.25%Zr, additionally alloyed with Si, Er, Hf, Nb, was studied. Cast blanks were obtained by induction casting in vacuum; wire with a diameter of 0.3 mm was obtained by drawing with preliminary deformation treatment of the blanks. The effect of the annealing temperature on the mechanical properties and specific electric resistivity (SER) of aluminum wires has been studied. The microstructure of wires in the recrystallized state is investigated. It is shown that as the annealing temperature increases, there is a monotonous decrease in tensile strength, micro-hardness, and SER. It is established that the ductility of the wire does not monotonously (with a maximum) depend on the annealing temperature. Optimal annealing modes have been determined, providing the best combination of tensile strength, microhardness and SER of aluminum wire.

Keywords: *aluminum, wire, strength, electric resistivity*

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INTRODUCTION

High-strength aluminum alloys with increased specific electrical conductivity are considered as a replacement for copper alloys [1–3], widely used for the manufacture of wires in aviation and automobile manufacturing. Thin aluminum wire up to 0.5 mm in diameter with high strength and electrical conductivity can also be used in the electric power industry. The traditional approach to the development of conductive aluminum alloys is to alloy them with elements that have little effect on specific electrical conductivity, but increase strength.

Currently, the most common eutectic aluminum alloys contain a high total concentration of rare earth elements (REE) such as La, Ce, Sm, etc. [4–9]. The second example is the alloys of the Al system.–Mg–Si [10–19], which have good plasticity, which allows them to be used to produce thin wires, but their strength is often insufficient. Deformation treatment of aluminum alloys can increase their strength, but has a negative effect on their plasticity and thermal stability. It is important to note that modern aluminum conductive alloys are subject to increased requirements for long-term thermal stability (see, for example, GOST R IEC 62004–2014 – not less than 130 MPa). Modern conductive eutectic aluminum alloys and Al alloys–Mg–Si has good thermal stability at short annealing times, but during long-term testing its performance often degrades sharply.

Conductor alloys of the Al –Zr system are being actively developed [20, 21]. In these alloys, at elevated temperature (above 350°C) and long-term exposure (50 – 100 h), strengthening particles of Al_3Zr with the $L1_2$ structure are formed. It should be noted that modern conductor alloys require ultra-long-term structural stability at temperatures of 180 – 220°C, close to the recrystallization temperature of pure aluminum. In Al –Zr system alloys, particles precipitate at higher temperatures or significantly longer annealing times [22, 23]. This necessitates additional annealing of aluminum alloy billets before drawing or rolling, which negatively affects the technological plasticity of the billet.

The second problem of Al –Zr – alloys is discontinuous decomposition of the solid solution, which leads to the precipitation of large needle-shaped (spindle-shaped) particles [24 – 26]. The theory of discontinuous decomposition of solid solution is currently well developed, and we will not dwell on this issue here. It is only important to note that the precipitation of such particles leads to a decrease in the technological plasticity of the aluminum alloy and to an increase in the number of wire breaks during cold drawing. To solve this problem, alloys of the Al –Zr system are alloyed with elements (Er, Hf, Y, etc.) that reduce the temperature of the onset of decomposition of the Zr solid solution in Al [27 – 31]. A promising approach is the combination of complex alloying with multi-stage heat treatment, which also helps to reduce the intensity of discontinuous decomposition of the solid solution [24].

The aim of the work – is the production and investigation of thermal stability of thin wires made from fine-grained Al – 0.25%Zr alloys, additionally alloyed with Si, Er, Hf, Nb. The results of studies on the thermal stability of microstructure, mechanical properties, and specific electrical resistivity (SER) of billets made from these fine-grained aluminum alloys were previously described in [32].

MATERIALS AND METHODS

The objects of the study were wires made of microalloyed aluminum alloys with a diameter of 0.3 mm. The chemical composition of the aluminum alloys is described in Table 1.

Table 1. Chemical composition of the studied alloys

Alloy	Chemical composition, wt. % (at. %)					
	Al	Zr	Si	Er	Hf	Nb
1	Base	0.25 (0.074)	0.10 (0.097)	0.25 (0.041)	0.20 (0.030)	—
2			0.10 (0.096)	0.25 (0.040)	—	—
3			0.10 (0.096)	—	—	—
4			—	—	0.15 (0.023)	—
5			—	—	0.25 (0.038)	—
6			—	—	—	0.15 (0.044)

Coarse-grained billets of alloys with dimensions of 20×20×160 mm were obtained by induction casting from high-purity aluminum A99(997) using an INDUTHERM VTC-200 casting machine. The fabrication modes of the billets are shown in Table 2. For the preparation of alloys, master alloys Al – 3%Zr, Al – 3%Hf, Al – 3%Si, Al – 3%Er, Al – 2%Nb were used, obtained by induction casting followed by rolling into foil with a thickness of 0.2 mm. Then the billets were subjected to equal-channel angular pressing (ECAP) and rotary swaging (RS). ECAP was carried out on a Ficep HF400L hydraulic press, in a square cross-section tooling.

Cylindrical billets with a diameter of 6 mm and a length of 1.3 – 1.5 m were manufactured using a HMP P5-4-21H rotary swaging machine. After RS, the billets were not annealed. The fabrication of thin wire with a diameter of 0.3 mm was carried out by drawing at room temperature, using a RODENT CGDE-1200 15.420 drawing mill. Carbide dies were used for drawing. The modes of deformation processing of billets before drawing are shown in Table 2.

Microhardness Investigations *Hv* were conducted in the center of the wire cross-section using a Qness A60+ hardness tester with a load of 20 g. The average measurement error of *Hv* was 3.5% of the measured value. For the measurement, wire samples were mounted in bakelite using a Buehler Simplimet 1000 press and subjected to mechanical polishing using a Buehler AutoMet 250 machine; at the final stage, polishing was performed with a colloidal suspension of SiO_2 (50 nm). The temperature and time of mounting were 160°C and 15 min, respectively.

Table 2. Wire Manufacturing Modes

Manufacturing Modes \ Alloy	1	2	3	4	5	6
Stage 1: Induction Casting						
Mold, mm	22×22×160, copper					
Ceramic crucible, cm^3	150					
Argon purging before melting, cycles	3					
Argon purging during heating, cycles	3					
Melt stirring	Induction					
Heating power, kW	4.5					
Melt holding temperature, °C	800					
Time until components melt, s	505	455	475	485	500	492
Holding time before pouring, min	20					
Pouring temperature, °C	780					
Cooling time, s	50 – 250					
including vibration time, s	50					
Stage 2: ECAP						
Temperature, °C	250					
Number of cycles	4					
Speed, mm/s	0.1					
Channel intersection angle, °	90					
Stage 3: Rotary Swaging						
Temperature, °C	20°C					
Deformation scheme, mm	∅ 20 → 6					
Total accumulated strain, %	70					
Stage 4: Drawing						
Temperature, °C	20°C					
Deformation scheme, mm	∅ 6 → 0.3					
Total accumulated strain, %	95					

Table 3. Properties of wires in the initial state

No.	1	2	3	4	5	6
Wire properties						
Hv, MPa	565±15	545±20	520±20	495±10	515±15	395±10
σ_u, MPa	268±16	260±10	261±19	186±18	224±22	170±15
δ, %	0.3±0.2	1.0±0.6	0.6±0.4	1.5±0.7	2.2±0.5	1.9±1.5
ρ, μΩ·cm	3.53±0.05	3.35±0.05	3.35±0.05	3.48±0.05	3.44±0.05	3.31±0.05
Characteristics of workpieces after ECAP + RD						
Hv, MPa	500 ± 15	510 ± 20	465 ± 15	420 ± 15	430 ± 15	400 ± 10
ρ, μΩ·cm	3.47 ± 0.03	3.45 ± 0.04	3.23 ± 0.04	3.15 ± 0.02	3.16 ± 0.02	3.43 ± 0.04

The microstructure investigation was carried out using a Jeol JSM-6490 scanning electron microscope (SEM) with Oxford Instruments INCA 350 energy dispersive microanalysis and a Jeol JEM-2100F transmission electron microscope (TEM) with a Jeol JED-2300 energy dispersive X-ray spectrometer.

For tensile tests, a Lloyd Instruments LR5K Plus testing machine was used. The 0.6 m long samples were tested at room temperature with a strain rate of 10 mm/min (0.001 s^{-1}). During the tests, a "stress σ - strain ε" diagram was recorded, from which the ultimate tensile strength σ_u and elongation to failure ™ were determined. Fractographic examination of sample fractures was performed using a Jeol JSM-6490 SEM.

Heat treatment of the samples was carried out in an EKPS-10 air furnace. The temperature control accuracy was 5°C.

To measure the electrical resistivity of the wire, a digital L-C-R meter E7-8 was used. For each sample with a length of 0.6 m, the cross-sectional area was examined at 10 points with an accuracy of 10 μm for measuring the sample diameter. The error in measuring the electrical resistivity was 0.05 μOhm · cm.

Further, the wires made from alloys No. 1 – 6 (see Table 1), for brevity, will be designated as wires No. 1 – 6.

EXPERIMENTAL RESULTS

Wires in the initial state have a highly deformed structure; the average size of fragments is ~0.2 – 0.5 μm. The chemical composition of the wires does not significantly affect the microstructure parameters of the alloys in the initial (non-annealed) state.

Table 3 shows the research results of the wire properties in the initial state.

The minimum microhardness (395 MPa) in the initial state is observed for the wire made from the Al – 0.25Zr – 0.15Nb alloy (alloy No. 6). The microhardness values for wires No. 1 – 5 range from 495 MPa (alloy No. 4) to 565 MPa (alloy No. 1). This exceeds the microhardness values for these alloys in the as-cast state by 150 (alloy No. 6) and 250 MPa (alloys No. 1 – 5) [32], but is close to the hardness of the initial fine-grained billets (Table 3).

Tensile tests showed that in the initial state, wires No. 1, No. 2, and No. 3 have the highest ultimate tensile strength values. The stress-strain curves σ (ε) have a typical appearance for heavily strengthened metals; the stage of uniform plastic flow is small (Fig. 1).

Fig. 1. Stress-strain diagrams of wire samples No. 1 (a) and No. 5 (b).

The relative elongation to fracture δ is ~1% (Table 3). Despite the low ductility, fractographic analysis showed that wires from all alloys in the initial state fracture in a ductile manner; the fracture surfaces represent a collection of dimples of various sizes (Fig. 2).

Fig. 2. Fractographic analysis of wire fractures in the initial state. The numbers in the figures correspond to the alloy numbers in Table 1. SEM.

The lowest electrical resistivity in the initial state is observed in wires No. 4 (3.11 μΩ·cm) and No. 5 (3.12 μΩ·cm). The highest electrical resistivity is observed for wire No. 1 (3.53 μΩ·cm), which contains the maximum concentration of alloying elements.

The results of the study on thermal stability of the wires are presented in Fig. 3. All alloys show a monotonically decreasing character of microhardness dependence on the temperature of 30-minute annealing. From Fig. 3a, it can be seen that the softening of wires No. 1 – 5 begins when heated to a temperature of 200°C. Analysis of the dependencies $H_V(T)$ shows that the addition of niobium to the Al – 0.25%Zr alloy (alloy No. 6) negatively affects its hardness and strength. The main reason is that in the presence of Nb, large $Al_3(Zr,Nb)$ particles with $D0_{23}$ structure are formed [33]. This leads to a decrease in the volume fraction of Al_3Zr particles with $L1_2$ structure, which make the greatest contribution to the strength and thermal stability of the fine-grained alloy. Wires No. 1 and No. 2 have the maximum hardness after 30-minute annealing at 500°C (Fig. 3a). It should be noted that the hardness of the annealed wires is higher than the hardness of the workpieces annealed at the same temperatures (see [32]).

Fig. 3. Dependencies of H_V (a), σ_t (b), ρ (c) on the temperature of 30-minute annealing of aluminum wires.

For all alloys, a decrease in electrical resistivity is observed with increasing annealing temperature by ~0.2 μΩ·cm (Fig. 3c). The intensive decrease in electrical resistivity, caused by the decomposition of the solid solution in aluminum alloys, begins after heating to 200 – 250°C. The minimum electrical resistivity after annealing at 500°C is characteristic of alloy No. 2, but it should be noted that its value significantly exceeds the electrical resistivity of pure aluminum (~2.7 μΩ · cm). The obtained result indicates that complete decomposition of the solid solution did not occur in the studied wires.

Fig. 3b shows the results of tensile tests. The nature of the change in the curves $\sigma(\varepsilon)$ after annealing is shown in Fig. 1; on the curves $\sigma(\varepsilon)$ for annealed alloys, a clearly defined stage of uniform plastic flow is observed. As can be seen from Fig. 1 and Fig. 3, annealing leads to a decrease in tensile strength and a non-monotonic change in elongation to fracture. For all wire samples, except those made of alloy No. 5, there is an increase in elongation to fracture after heating to 400 – 450°C and a decrease in ductility when the annealing temperature is increased to 500°C. After annealing at 500°C, the value of δ does not exceed 7% for all alloys except No. 5. For wire No. 5, the value of δ is approximately 32%. The results of electron microscopy studies show that at these temperatures, there is growth of $Al_3(Zr,X)$ particles precipitated as a result of annealing (Fig. 4), and abnormal grain growth is also observed (Fig. 5a). It should be noted that in alloy No. 5 after annealing, a homogeneous fine-grained microstructure with an average grain size of about 5 μm is preserved (Fig. 5b), which probably determines its increased ductility after annealing at a temperature of 500°C (Fig. 3c). The particles are predominantly precipitated in the grain volume (Fig. 4).

Fig. 4. Al3Zr particles in wires made of alloy No. 2 (a) and No. 5 (b) after annealing at 300°C. TEM.

Fig. 5. SEM images of the microstructure of wire No. 2 (a) and No. 5 (b) after annealing at a temperature of 500°C (30 min). SEM.

Fig. 6. Fractographic analysis of fractures of wire samples No. 1 and No. 5 after heat treatment (30 min) at various temperatures. SEM.

Fractographic analysis (Fig. 6) of the fracture area shows that annealing leads to a change in the nature of fractures; no dimples were found on the fracture surface.

DISCUSSION OF RESULTS

The microhardness of blanks No. 1 – 6 was 500±15 MPa (No. 1), 510±20 MPa (alloy No. 2), 465±15 MPa (No. 3), 420±15 MPa (No. 4), 430±15 MPa (No. 5) and 400±10 MPa (No. 6) [32]. Thus, the microhardness of the wires in the initial state is higher than the microhardness of the blanks. The electrical resistivity of the wires is close to the electrical resistivity of the blanks.

The ultimate tensile strength dependence on microhardness of the investigated aluminum wires is presented in Fig. 7. The figure shows that between σ_B and H_v there is a reliable correlation, but the nature of the dependence $\sigma_B(H_v)$ differs from the usual linear function.

Fig. 7. Dependence of ultimate tensile strength on microhardness of the investigated aluminum wires.

Fig. 8 summarizes the results of the electrical resistivity and ultimate tensile strength studies of the wires. Dashed lines indicate the level of characteristics that should be provided in new conductive alloys serving as replacements for the industrial alloy 01417 ($\rho \leq 3.0$–3.1 μΩ·cm, $\sigma_B \geq$ 160 – 200 MPa) (see [34]).

Fig. 8. Diagram "Electrical resistivity (ρ) – ultimate tensile strength (σ_b)" for wires (filled markers) and billets (empty markers).

Analysis of the data presented in Fig. 8 shows that the wires have higher values of electrical resistivity ($\rho > 3.1$ μΩ·cm), but after heat treatment (200°C, 30 min), all wires have the required ultimate tensile strength values. It should also be noted that according to the requirements of GOST R IEC 62004-2014, a 1-hour annealing at 400°C simulates long-term (more than 350,000 hours) operation of the wire at 150°C. The obtained result means that the developed wires have the necessary level of thermal strength stability.

In our opinion, the main reason for the increased electrical resistivity values of the annealed wires is the incompleteness of the solid solution decomposition process. This is an extremely unexpected result, since, as can be seen from work [32], the electrical resistivity of fine-grained billets from alloys No. 1 – 6 after annealing at 500°C is in the range of 2.9 – 3.1 μΩ · cm, which meets the specified requirements. Since the electrical resistivity of billets, measured by the eddy current method, is close to the electrical resistivity of wires (Table 3), the observed differences for annealed billets and annealed wires are evidently related to differences in the nature of the second phase particles precipitation.

Fig. 9. Diagram "microhardness – electrical resistivity" for wires (empty markers) and billets (filled markers) from alloys No. 1, 2 and 3.

Thus, annealed wires possess simultaneously increased (relative to fine-grained blanks) hardness and electrical resistivity. This is most clearly seen from the "microhardness - electrical resistivity" diagram presented in Fig. 9, which compares data for wires and blanks.

It is usually assumed that additional deformation has a weak effect on the process of Al_3X particles precipitation [34], but may influence the mechanism of their precipitation [36].

Analysis of the dependencies of electrical resistivity on annealing time using the Mel – Johnson – Avrami – Kolmogorov equation showed that the main mechanism of solid solution decomposition in the studied fine-grained alloys No. 1 – 6 is the precipitation of particles on the cores of lattice dislocations [32].

Wires No. 1 – 6 after annealing at 500°C have a finer-grained microstructure than blanks annealed at the same temperatures (see [32]). This leads to the fact that the hardness of annealed wires exceeds the hardness of annealed blanks.

The obtained result means that, in accordance with the Zener equation, smaller particles precipitate during wire annealing than during blank annealing. In our opinion, this is due to the preferential precipitation of particles in the grain volume during wire annealing (Fig. 4), while during blank heating, particles precipitate along the cores of lattice dislocations ([32]). Since the diffusion coefficient in the crystal lattice is much smaller than the diffusion coefficient along dislocation cores, the intensity of precipitation and growth of particles during wire annealing will be less than during annealing of fine-grained blanks.

CONCLUSION

The features of changes in mechanical properties and electrical resistivity of wires made from aluminum alloys Al – 0.25%Zr, microalloyed with Si, Er, Hf, Nb, were investigated.

It is shown that after annealing of wire from alloy No. 5 (Al – 0.25Zr – 0.25Hf) at 500°C, a homogeneous fine-grained structure and an increase in plasticity to 7% are observed.

It has been established that as a result of wire annealing, there is a decrease in the intensity of electrical resistivity changes compared to the changes in electrical resistivity in fine-grained billets from which these wires were manufactured by drawing. Alloy No. 2 (Al – 0.25Zr – 0.10Si – 0.25Er) after heat treatment at 300°C (30 min) has the optimal combination of strength and electrical resistivity.

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CONFLICT OF INTERESTS

The authors declare that they have no conflict of interest.

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