STUDY OF THE EFFECT OF TWO-STAGE ANNEALING ON THE MICROSTRUCTURE AND PROPERTIES OF DEFORMED SEMI-FINISHED PRODUCTS FROM THE ALLOY Al-Zr SYSTEM, OBTAINED WITH THE APPLICATION OF TWIN ROLL CASTING-EXTRUDING METHOD

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Abstract. This article presents the results of the study of the joint influence of the conditions of twin roll casting-extruding of longish rod semi-finished products from aluminum alloy with a content of 0.3% zirconium, 0.2% iron and two-stage annealing on their structure and properties. The patterns of changes in the mechanical properties and electrical resistivity of rods depending on different conditions of deformation-heat treatment are shown. Processing modes are established to increase the level of strength, plasticity and achieve satisfactory electrical conductivity. The influence of two-stage annealing on the structure and properties of an experimental batch of rods made with different conditions of twin roll extruding has been investigated. The results of micro-X-ray spectral analysis and the granular structure of the samples before and after heat treatment are presented. The modes of twin roll casting-extruding and two-stage annealing are established, ensuring the manufacture of rods from the alloy under study with a good combination of mechanical properties and electrical conductivity.

Introduction

A promising method for the production of electrical wiring products is the twin roll casting-extruding method proposed by scientists from the Department of Metal Forming of the Institute of Non-Ferrous Metals and Materials Science of the Siberian Federal University. The method provides a significant increase in the strength and plastic properties of deformed semi-finished products due to an additional type of alternating strain and reduction of energy costs for their manufacture due to the occurrence of active friction forces during the interaction of the rolls with the metal being processed, contributing to the deformation process. In addition to the latter, the economic effect is associated with a reduction in technological conversions as compared to other methods of producing long-deformed semi-finished products from aluminum alloys [1-4].

Achievement of the required combination of properties of aluminum conductor products in production is ensured by the use of alloys doped with transition and rare earth metals. A significant increase in the strength of the conductors, maintained when heated to 230 ºC and satisfactory electrical conductivity, makes it possible to achieve an additive of zirconium in an amount of 0.1-0.3 %. Zirconium in the process of rapid melt crystallization completely enters the solid aluminum solution, and the subsequent annealing of the obtained semi-finished products leads to the separation of dispersed Al_3Zr phases along the grain and subgrain boundaries, which impede their movement during heating and ensure the stability of the wire structure. In addition, the decomposition of the solid solution after annealing leads to a decrease in the electrical resistance of semi-finished products, and the use of two-stage annealing according to the results of [4] provides a more significant reduction of this indicator. Increasing the iron content to 0.4% in the alloy has a more significant effect on the growth of strength properties and to a lesser extent affects the decrease in electrical conductivity [1-11].

The purpose of this work is to study the joint effect of different conditions of twin roll casting-extruding and two-stage annealing on the structure and properties of deformed semi-finished products from an alloy with a content of 0.3% zirconium and 0.2% iron.

Methods of carrying out researches

An experimental batch of rods from the alloy under study was obtained by twin roll casting-extruding method in accordance with the scheme in Fig. 1. The melt from the furnace 1 was poured into the receiving device 2, which ensures uniform metal supply to the indoor varietal caliber formed by a roll with a protrusion 3 and a roll with an annular groove 4, in which the metal crystallized, followed by rolling and extruding the bar through a calibrating hole in the installed at the exit of the rolls of the die 5. After cooling, the rods were subjected to two-stage annealing in a heat treatment furnace 6. The total chemical composition of the alloy is presented in Table 1.

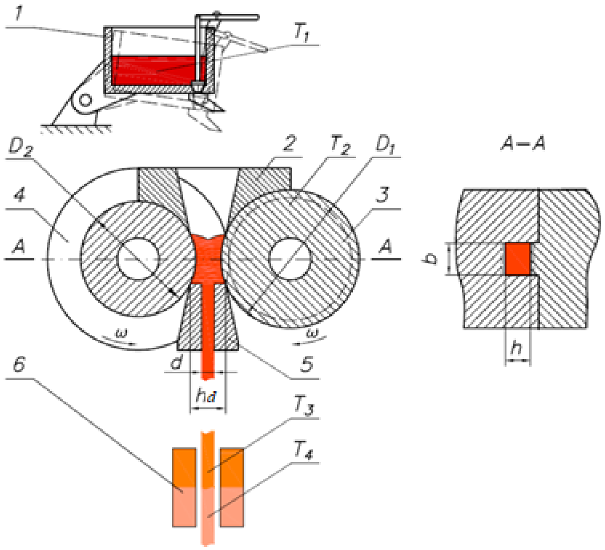


Fig. 1. The general scheme of twin roll casting-extruding with heat treatment:
1 – metal casting furnace; 2 – melt receiving device; 3 – roll with a protrusion; 4 – roll with an annular groove; 5 – die; 6 – heat treatment furnace

Table 1 – The chemical composition of the alloy Al-Zr

Al	Zr	Si	Fe	Cu	Mn	Mg	Cr	Ni	Zn	Ti	V	Ga
99.35	0.30	0.10	0.20	0.002	0.002	0.002	0.001	0.006	0.005	0.005	0.012	0.012

The geometrical parameters of the deforming unit were: roll diameter along the protrusion D_1 = 214 mm; on the bottom of the stream caliber D_2 = 167 mm; caliber width b = 15 mm; the minimum height of the gauge on the common axis of the rolls h = 7 mm; die height h_d = 20 mm; diameter of the extruded rods d = 5, 7 and 9 mm. The technological parameters were as follows: the temperature of the melt pouring into the caliber of the rolls T_1 = 800 ºC; roll temperature T_2 = 100 ºC; rotational speed ω = 4 rpm. The deformation parameters were calculated, thus, the degree of deformation during rolling was ε = 50%, drawing ratio during extruding - in the range μ = 4.4-14.3 and strain rate $\xi = 0.74\ s^{-1}$.

Extruded rods with different drawing ratios were subjected to two-stage annealing according to the mode chosen, based on the results of the research presented in work [4], namely: the temperature of the first stage was T_4 = 350 ºC, holding time 40 hours; second stage temperature T_5 = 440 ºC, holding time 20 hours. Next, we investigated the structure and properties of the rods before and after heat treatment.

Ultimate tensile strength R_m and elongation to failure A determined by stretching the machine LFM 20 Walter Bai AG, microhardness was measured by the Vickers method with a load of 2 N on the device AFFRI DM 8, electrical resistivity ρ of rods was measured on samples with an estimated length of 1 m on device «VITOK».

X-ray spectroscopic analysis of the composition of the phases was carried out on an EVO 50 scanning electron microscope using an INSA ENERGY dispersive spectrometer. The grain shape after twin roll casting-extruding was studied on an Axio Observer Al.m (Carl Zeiss) light microscope after etching thin sections in a one-percent HF solution at a magnification of ×1000. Samples for the study of the granular structure of semi-finished products were prepared by oxidizing the surface of thin sections in an aqueous solution of hydrofluoric and boric acid using an ATM KRISTALL 620 electrolytic polishing and etching unit. The grain structure was measured in a polarized light using an Axio microscope.

Results and its discussion

The results of the study of the properties of prototypes are summarized in Table 2. The values of the ultimate tensile strength R_m of the rods in the deformed state were 131-141 MPa, and two-stage annealing leads to a decrease in strength to 121-128 MPa. The minimum level of elongation to failure A = 24% was fixed on the rods after the most intensive mode of twin roll casting-extruding (μ = 14.3), which indicates a higher level of ductility compared to rod of the corresponding chemical composition obtained by the casting-rolling technology [4].

The microhardness of the rods after crystallization of the melt and rolling-extruding was 34-37 HV and decreased after two-stage annealing to 29-34 HV. Electrical resistivity ρ was in the range 0.0328-0.0330 Ohm·mm^2/m for rods in the deformed state, which is caused by the dissolution of zirconium in the solid aluminum solution during the rapid crystallization of the melt, and the subsequent annealing of the rods allows to reduce ρ to 0.0290-0.0297 Ohm·mm^2/m.

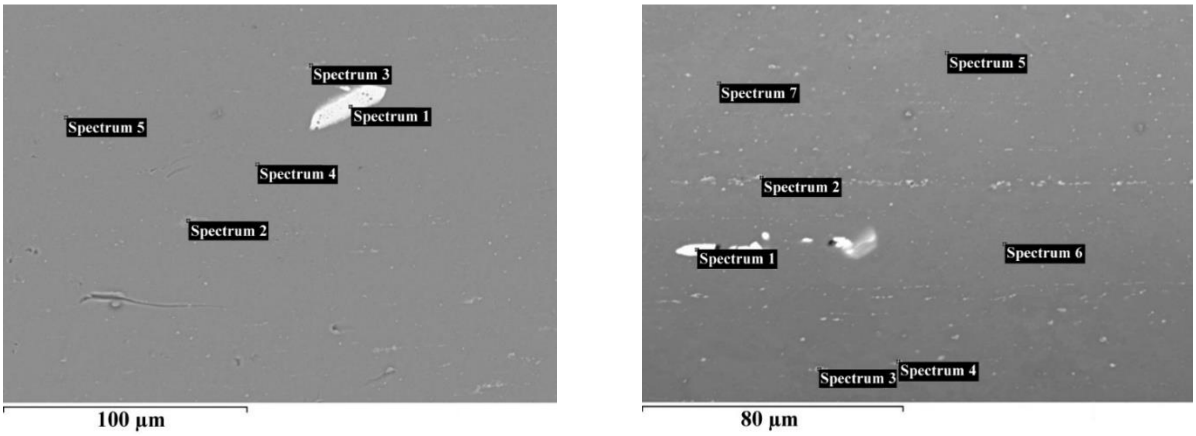
Table 2 – Properties of rods from the alloy under investigation after twin roll casting-extruding and two-stage annealing

d, mm	μ	Condition	R_p, MPa	R_m, MPa	A, %	ρ, Ohm·mm^2/m
5	14.3	Deformed	106	141	24	0.0330
		Annealed	96	128	25	0.0297
7	7.3	Deformed	102	136	25	0.0329
		Annealed	94	125	30	0.0293
9	4.4	Deformed	90	131	31	0.0328
		Annealed	90	121	35	0.0290

An increase in the drawing ratio of μ from 4.4 to 14.3 leads to an increase in the ultimate tensile strength of rods to 141 MPa, a decrease in the elongation to failure A to 24% and an increase in the values of ρ to 0.0330 Ohm·mm^2/m. The minimum value of electrical resistivity of 0.0290 Ohm·mm^2/m is achieved after two-step annealing of rods obtained with an extrusion ratio of 4.4, while annealing of rods made with a higher strain intensity (μ = 14.3) leads to a decrease in ρ to 0.0297 Ohm·mm^2/m and provides a level of ultimate tensile strength R_m = 128 MPa.

Analysis of the results of microhardness studies confirms the tendency of hardening of the rods with an increase in the drawing ratio. Increasing μ from 4.4 to 14.3 leads to an increase in microhardness values from 34 to 37 HV, and annealing reduces the hardness of the samples to 25 and 34 HV, respectively.

The microstructure of the semi-finished products after twin roll casting-extruding is an α-solid solution based on aluminum, excess AlFeSi (spectrum 2) phases oriented in the direction of deformation, as well as lamellar Al_3Zr (Fig. 2, a, spectrum 1) primary crystals with a size of 70 μm and zirconium content up to 50 masses %. The formation of primary Al_3Zr intermetallic compounds is not desirable, however, single clusters found in the structure do not have a significant effect on the mechanical properties of the rods studied, which is confirmed by the data in Table 2. To ensure more complete dissolution of zirconium in the solid aluminum solution, it is recommended to heat the melt to a temperature above than 800 ºC.

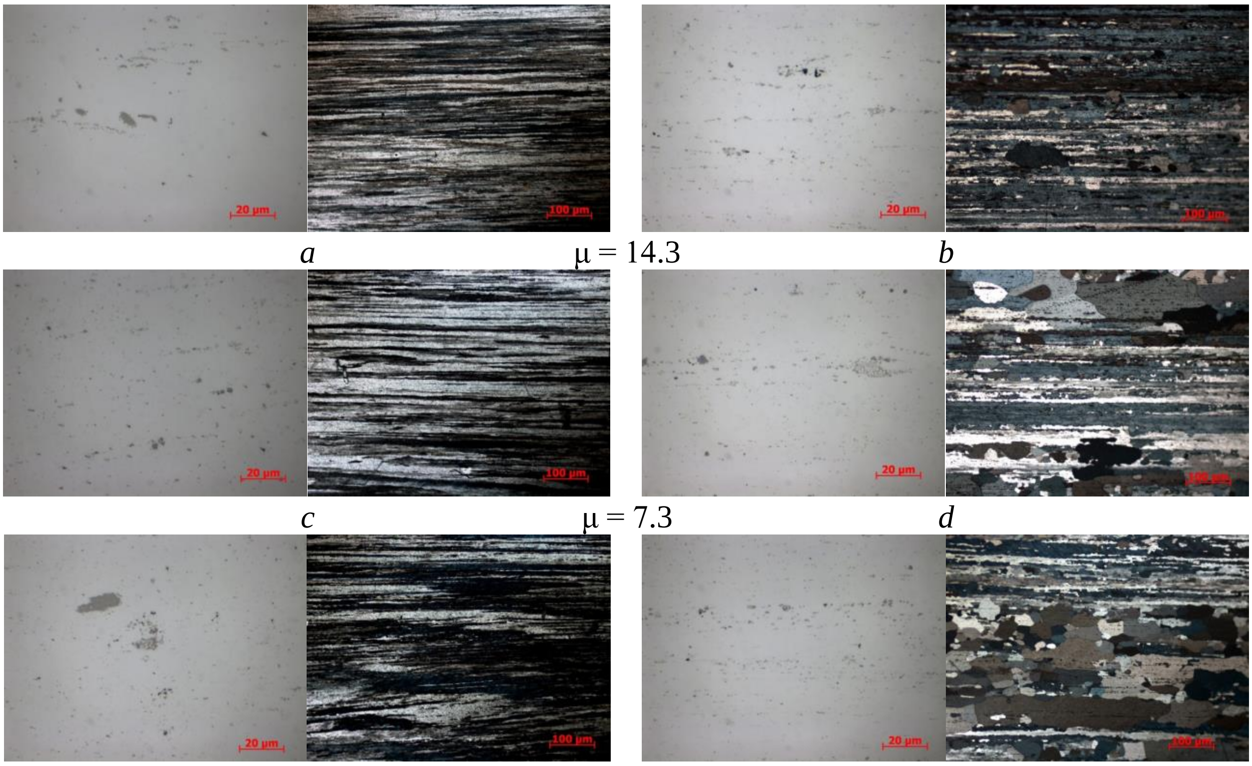


a *b*

Fig. 2. The microstructure of the investigated alloy rods with an electron microscope EVO 50: *a* – after twin roll casting-extruding; *b* – after two-stage annealing

Two-stage annealing ensures the decomposition of the solid solution supersaturated with zirconium with the release of dispersed Al_3Zr particles (Fig. 2, b, spectrum 3, 6 and 7), leads to a less significant decrease in the ultimate tensile strength, increase in ductility and a significant reduction alloying elements in solid solution.

Image analysis of the microstructure of rods after twin roll casting-extruding, presented in Fig. 3, showed that an increase in the drawing ratio does not lead to a significant change in the shape and size of the iron-containing and intermetallic Al_3Zr phases.



a μ = 14.3 *b*

c μ = 7.3 *d*

e μ = 4.4 *f*

Fig. 3. The microstructure of the rods of the investigated alloy (×1000):
a, *c*, *e* – rods with a diameter of 5, 7 and 9 mm, respectively, after twin roll casting-extruding;
b, *d*, *f* – rods with a diameter of 5, 7 and 9 mm, respectively, after two-stage annealing;
μ – drawing ratio during extruding

The structure of the rods in a longitudinal section before annealing is represented by grains elongated along the strain axis (Fig. 3, a, c, e). Two-stage annealing leads to the formation of a partially recrystallized structure of rods extruded with drawing ratios 4.4 and 7.3 (Fig. 3, e, f), but their strength remains at the level of 120-125 MPa. An increase in this indicator to 14.3 leads to an increase in the ultimate tensile strength after annealing to 128 MPa and ensures the stability of the rod structure (Fig. 3, b), which is probably due to the most complete decomposition of the supersaturated solid solution in the second stage of annealing due to the release of thermally stable coherent the aluminum lattice of the Al_3Zr phase particles on the finished centers formed during the first stage of annealing.

Conclusion

The study of the joint influence of the conditions of twin roll casting-extruding and two-stage annealing on the structure and properties of aluminum alloy rods with a content of 0.3% zirconium and 0.2% iron allowed us to draw the following conclusions. First, an increase in the drawing ratio from 4.4 to 14.3 provides an increase in the strength properties from 130 to 140 MPa and does not significantly affect the increase in the values of electrical resistivity, which grows from 0.0328 to 0.0330 Ohm·mm^2/m. Two-stage annealing allows to achieve a significant reduction in electrical resistivity to a minimum value of 0.0290 Ohm·mm^2/m after annealing the rods extruded with drawing ratio of 4.4, while the ultimate tensile strength is 120 MPa. Secondly, the use of two-stage annealing ensures the decomposition of the solid solution supersaturated with zirconium with the release of fine Al_3Zr particles, which impede recrystallization processes, and the rods made with a higher deformation intensity have the most stability (μ = 14.3). Thirdly, all the rods have a high level of plasticity, in the deformed state, the values of elongation to failure are in the range of 24-30%, in the annealed state – 25-35%. Fourth, a good combination of mechanical properties and electrical conductivity of rods is achieved after twin roll casting-extruding with drawing ratio of 7.3 and subsequent two-stage annealing. The value of the ultimate tensile strength after such a treatment mode is 125 MPa, elongation to failure of 30%, a microhardness of 29 HV and a specific electrical resistivity of 0.0293 Ohm·mm^2/m.

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Nomenclature

T	temperature, [ºC]
μ	drawing ratio, [-]
ε	degree of deformation, [%]
ξ	strain rate, [s^{-1}]
ω	roll rotational speed, [rpm]
R_m	ultimate tensile strength, [MPa]
R_p	yield strength, [MPa]
A	elongation to failure, [%]
ρ	electrical resistivity, [Ohm·mm^2/m]

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