INFLUENCE OF STEPPED ANNEALING ON THE PROPERTIES OF CONDUCTOR WIRE AFTER INGOTLESS ROLLING-EXTRUSION AND DRAWING OF ALUMINUM ALLOYS CONTAINING Zr, Ce, La AND Fe

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Received 05 March 2023
Accepted 24 April 2023

ABSTRACT

The paper presents the results of a study of the effect of stepped annealing on the microstructure, mechanical properties and electrical conductivity of longish rods Ø5 mm and wire Ø 0.7 - 1 mm made of aluminum alloys containing 0.13 % - 0.21 % zirconium, 0.40 % - 0.60 % cerium, 0.2 % - 0.3 % lanthanum and 0.19 % - 0.68 % iron, produced by ingotless rolling-extrusion (IRE) and drawing. The possibility of achieving a level of strength of a conductor wire of 172 - 179 MPa, a specific electrical resistance of 0.0291 - 0.0300 Ohm·mm^2/m, and elongation to failure of 1.5 % - 3 % with relatively small additions of zirconium, cerium, and lanthanum in the alloy due to an increase in the content of iron, the choice of deformation and heat treatment parameters. The structure of the wire grain structure was studied after IRE, drawing and stepped annealing 350ºC, 40 h - 440ºC, 20 h at different stages of drawing. The research was supported by a grant from the Russian Science Foundation No 22-79-00108, https://rscf.ru/project/22-79-00108/.

Keywords: aluminium, zirconium, cerium, iron, ingotless rolling-extrusion, stepped annealing, mechanical properties, electrical resistivity.

INTRODUCTION

Of particular importance for the development of modern enterprises for the deep processing of aluminum alloys is the introduction of technologies for the manufacture of conductor products with minimal expenditure of energy resources, expensive materials for the preparation of the alloy and operational losses. Therefore, processing methods that involve the combination of crystallization and deformation processes in one line attract great attention from leading scientific teams [1, 2].

An effective solution in this area is the technology of combined casting, rolling and extrusion ExtruForm, designed by the designers of RUSAL and scientists of the Department of Metal Forming of the Siberian Federal University, which makes it possible to reduce the production cycle by six times compared to the traditional method of producing wire rod [3, 4].

The ExtruForm line is intended for the production of wire rod for electrical purposes, and the design involves the production of a continuously cast billet in a rotary mold, followed by combined rolling and extrusion, which reduces operating costs by 25 % [5].

An urgent task is to exclude from the technological scheme the operation of casting an ingot in a mold, in particular, the authors of the patent [6] proposed a method of ingotless rolling-extrusion (IRE), which involves the manufacture of a continuous rod billet from a melt.

The IRE method provides an improved set of properties of rods made of aluminum alloys due to severe plastic deformation, since the melt after crystallization immediately experiences a large relative reduction of 40 % - 50 % during sectional rolling, then all-round uneven compression at the moment of pressing out at the die mirror, shear stress before extrusion from - due to the asymmetry of plastic deformation and an instantaneous decrease in the cross-section by a factor of 5 - 20 during metal extrusion [4, 6].

Achieving an improved set of properties of the conductor wire is associated with an active search for new compositions of low-alloy aluminum alloys that provide a high level of heat resistance, strength, ductility and electrical conductivity, therefore, research is focused on the creation of multicomponent alloys with a low content of rare earth and transition materials, such as hafnium, cerium, lanthanum, zirconium, scandium, yttrium, potassium, erbium, ytterbium, etc. This work is devoted to assessing the effect of different IRE conditions, drawing and stepped annealing on the complex of properties of a conductor wire made of aluminum alloys with different combinations of zirconium, cerium, lanthanum and iron.

Zirconium contributes to an increase in the thermal stability of the alloy with the possibility of reducing the electrical conductivity due to the formation of Al_3Zr nanoparticles in the structure after annealing. A good effect of strengthening and precipitation hardening of Al is achieved with a content of 0.20 % - 0.30 % Zr at temperatures of 400°C - 450°C, and the decomposition of a supersaturated Al solid solution with a content of 0.20 % Zr begins after 24 hours of exposure due to the abundant precipitation of coherent nanosized Al_3Zr (L12) phases at temperatures of 375°C - 425°C [7 - 10].

The addition of 0.5 % cerium to the Al alloy has a positive effect on improving the electrical conductivity due to the formation of binary, ternary, and quaternary compounds of aluminum with Si, Fe, a decrease in the static distortion of the solid solution lattice, and an increase in the mean free path of electrons. It has been proven that a change in the structure of the energy bands of electrons under the action of cerium can increase the effective number of electrons involved in conduction [11, 12].

An increase in the iron content in the Al-0.30%Zr-1%Fe alloy provides a significant increase in strength. Iron is slightly soluble in aluminum, forms excess intermetallic phases with it, and binds silicon dissolved in aluminum, which has a positive effect on electrical conductivity [13].

The aim of the work was to study the effect of stepwise annealing 350ºC, 40 h - 440ºC, 20 h on the grain structure, mechanical properties and electrical conductivity of wire Ø 0.7 - 1 mm from aluminum alloys containing 0.13 % - 0.21 % zirconium, 0.4 % - 0.6 % cerium, 0.2 % - 0.3 % lanthanum, and 0.19 % - 0.68 % iron after ingotless rolling-extrusion (IRE) and drawing.

EXPERIMENTAL

The chemical composition of experimental alloys with a general scheme for the manufacture of prototype wire samples are shown in Table 1 and Fig. 1.

alloy 1 – Al - 0.2 % Zr - 0.19 % Fe
alloy 2 – Al - 0.21 % Zr - 0.53 % Fe
alloy 3 – Al - 0.13 % Zr - 0.21 % Fe - 0.4 % Ce - 0.2 % La
alloy 4 – Al - 0.13 % Zr - 0.68 % Fe - 0.6 % Ce - 0.3 % La

The IRE parameters were chosen based on the data in [14]. Rods Ø 5 mm were produced by the IRE method with an increased elongation factor $\mu = 14.3$ on a CRE-200 laboratory unit. The melt from the mixer furnace *1* through the metal guide device *2* was fed into a closed box caliber formed by a roll with a protrusion *3* and a roll with a groove *4* (section A-A), in which it was compressed, rolled and extruded into the hole of the die *5*.

The diameter of the rolls along the protrusion D_1 = 214 mm; along the bottom of the stream caliber D_2 = 167 mm; gauge width b = 15 mm; minimum pass height on the common axis of the rolls h_1 = 7 mm; die mirror height h_2 = 20 mm. The temperature of pouring the melt into the roll caliber T was 800ºC - 850ºC depending on the content of alloying components; roll heating temperature 100ºC; roll rotation frequency ω = 4 rpm; degree of rolling deformation ε_1 = 50 %; drawing ratio μ and strain rate ξ at the moment of extrusion 14.3 and 0.74 s^{-1}.

Next, the rods were drawn on a chain drawing machine at a speed v = 0.17 m/s at room temperature 25ºC according to different technological schemes 1, 2, and 3 (Fig. 1) using stepped annealing 350ºC, 40

Table 1. Chemical composition of experimental alloys.

№	Al	Ce	La	Zr	Fe	Si	Cu	Mn	Mg	*T*, ºC
1	balance	–	–	0.20	0.19	0.08	0.002	0.001	0.001	800
2	balance	–	–	0.21	0.53	0.08	0.003	0.001	0.001	820
3	balance	0.40	0.20	0.13	0.21	0.08	0.003	0.010	0.001	850
4	balance	0.60	0.30	0.13	0.68	0.05	0.004	0.002	0.002	850
T – pouring temperature into roll caliber										

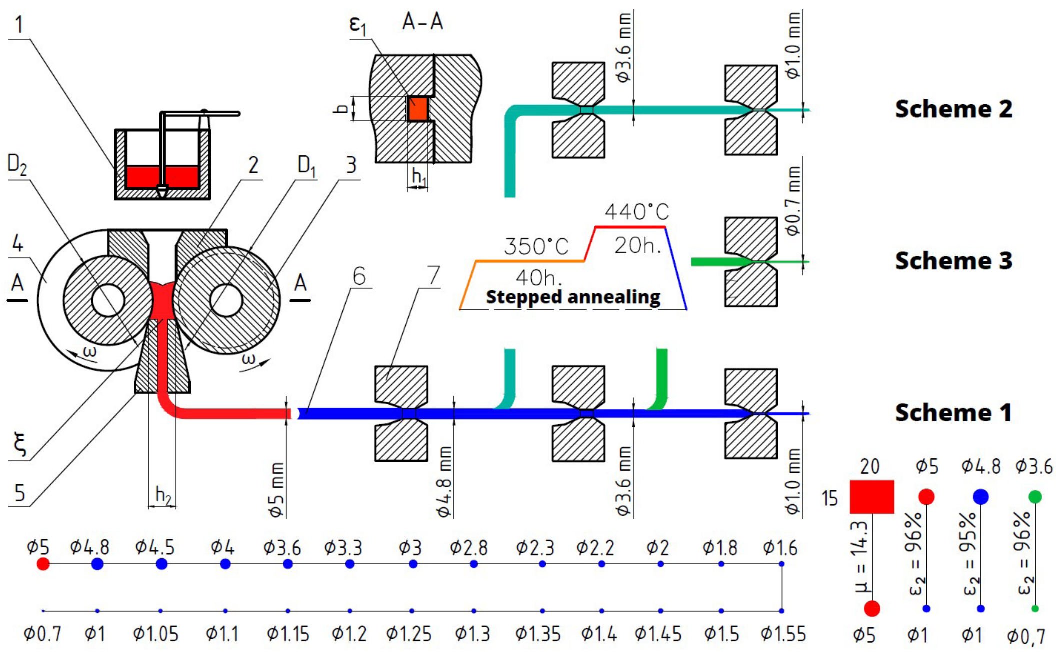

Fig. 1. Sample obtaining scheme by ingotless rolling-extrusion (IRE) and drawing methods.

h - 440ºC, 20 h, the mode of which was justified earlier in [14, 15]. The total relative drawing reduction ε_2 was 95 % - 96 % to ensure comparable cold deformation conditions after intermediate annealing.

Ultimate tensile strength R_m and elongation to failure *A* were determined by the tensile method on an LFM 20 machine from Walter Bai AG, electrical resistivity ρ was measured with a VITOK milliohmmeter on samples 1 m long. The character of grain distribution after deformation and stepwise annealing was revealed by oxidation of the surface of microsections in Barker's reagent with the composition H_2O - 98.2 cm^3 and HBF_4 - 1.8 cm^3 on a Struers LectroPol - 5 electrolytic polishing and etching unit. The structure of the grain was recorded on an optical microscope Carl Zeiss Observer 7 Mat.

RESULTS AND DISCUSSION

The results are presented in Tables 2-4 and in Fig. 2. IRE of rods Ø 5 mm with an elongation coefficient μ = 14.3 and reduction ε_2 = 96 % after drawing a wire Ø 1 mm (Table 2) from alloy 1 and alloy 3 provides R_m = 190 - 217 MPa, ρ = 0.0307 - 0.0321 Ohm·mm^2/m, and *A* = 2.3 % - 2.5 %. An increase in the iron content in alloy 2 and alloy 4 provides R_m = 200 - 224 MPa and ρ = 0.0314 - 0.0336 Ohm·mm^2/m.

Stepped annealing of wire Ø 4.8 mm from alloy 1 with further reduction ε_2 = 96 % to Ø 1 mm (Table 3) reduces R_m = 156 MPa and ρ = 0.0294 Ohm·mm^2/m, however, the addition of 0.53 % Fe in alloy 2 makes it possible to increase R_m = 176 MPa at ρ = 0.0291

Table 2. Properties without step annealing (scheme 1, Fig. 1).

Product	Rod Ø5 mm after IRE				Wire Ø4.8 mm			
Alloy	1	2	3	4	1	2	3	4
R_m, MPa	123	138	146	144	129	157	153	156
A, %	32	29	29	31	15	11	11	12
ρ, Ohm·mm²/m	0.033	0.0332	0.0305	0.031	0.0332	0.0338	0.0314	0.0319
Product	Wire Ø3.6 mm				Wire Ø1 mm			
R_m, MPa	162	185	189	188	217	224	190	200
A, %	4	8	3	5	2.3	2.7	2.5	2.3
ρ, Ohm·mm²/m	0.0329	0.0337	0.0315	0.0319	0.0321	0.0336	0.0307	0.0314

Table 3. Properties after stepped annealing at Ø 4.8 mm (scheme 2, Fig. 1).

Product	Rod Ø5 mm after IRE				Annealed wire Ø 4.8 mm			
Alloy	1	2	3	4	1	2	3	4
R_m, MPa	123	138	146	144	81	117	88	90
A, %	32	29	29	31	18	26	32	35
ρ, Ohm·mm²/m	0.033	0.0332	0.0305	0.031	0.0305	0.0301	0.0304	0.0303
Product	Wire Ø 3.6 mm				Wire Ø 1 mm			
R_m, MPa	141	166	–	–	156	176	164	174
A, %	4	8	–	–	2	2	2	3
ρ, Ohm·mm²/m	0.03	0.0296	–	–	0.0294	0.0291	0.0294	0.0294

Table 4. Properties after stepped annealing at Ø 3.6 mm (scheme 3, Fig. 1.).

Product	Rod Ø5 mm after IRE				Wire Ø 3.6 mm			
Alloy	1	2	3	4	1	2	3	4
R_m, MPa	123	138	146	144	162	185	189	188
A, %	32	29	29	31	4	8	3	5
ρ, Ohm·mm²/m	0.033	0.0332	0.0305	0.031	0.0329	0.0337	0.0315	0.0319
Product	Annealed wire Ø 3.6 mm				Wire Ø0.7 mm			
R_m, MPa	77	91	90	93	161	172	168	179
A, %	19	20	38	38	2	2	1.5	2
ρ, Ohm·mm²/m	0.03	0.0302	0.0302	0.0306	0.03	0.03	0.03	0.03

Ohm·mm²/m. A good combination of wire properties with a lower zirconium content of 0.13 % can be achieved by introducing 0.4 - 0.6 % Ce, 0.2 - 0.3 % La and 0.68 % Fe, in particular, alloys 3 and 4, after IRE of a Ø 5 mm rod, stepped annealing of a Ø 4.8 mm wire and drawing up to Ø 1 mm (ε_2 = 95 %) provide R_m = 164 - 174 MPa and ρ = 0.0294 Ohm·mm²/m. Ligature with iron, cerium and lanthanum is several times cheaper than zirconium.

The use of stepwise annealing of wire Ø3.6 mm and drawing up to Ø0.70 mm (ε_2 = 96 %) of alloys 3, 4 makes it possible to obtain a higher R_m = 168 - 179 MPa, however, the value of ρ also increases to 0.0300 Ohm·mm²/m (Table 4). The highest strength among the presented compositions has a wire made of alloy 2 and alloy 4 with a high iron content, R_m = 172-179 MPa; A = 1.5 - 2 %; ρ = 0.291 - 0.0300 Ohm·mm²/m, made according to scheme 3 (Table 4).

The structure of the rods of alloys 1, 2 (Fig. 2(a, b) consists of a solid solution of Al and iron-containing phases Al-Fe-Si, zirconium is dissolved in a solid solution and its content is 0.2 %. An increase in the

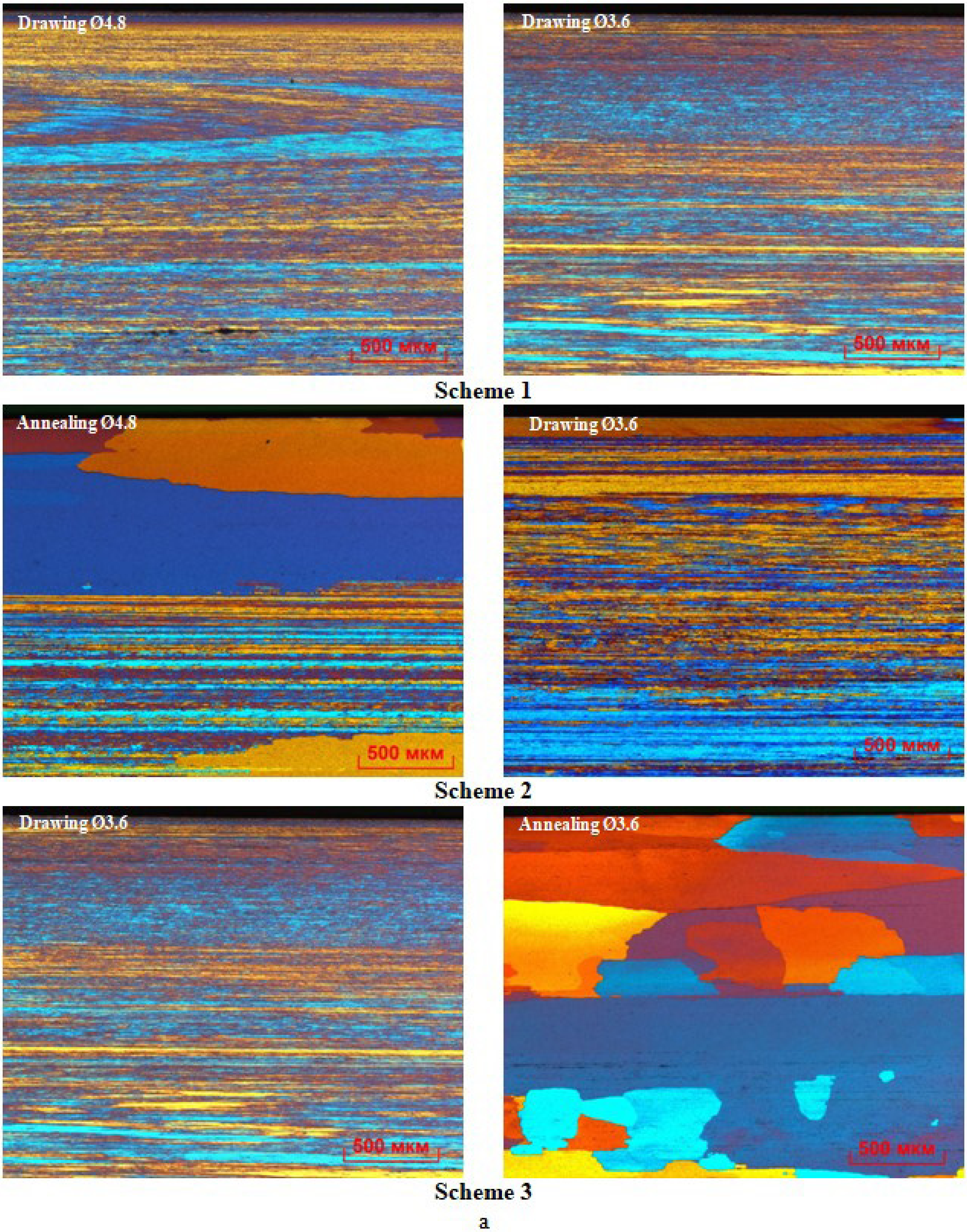

Fig. 2. Wire grain macrostructure after drawing and stepped annealing 350ºC, 40 h - 440ºC, 20 h: a - alloy 1.

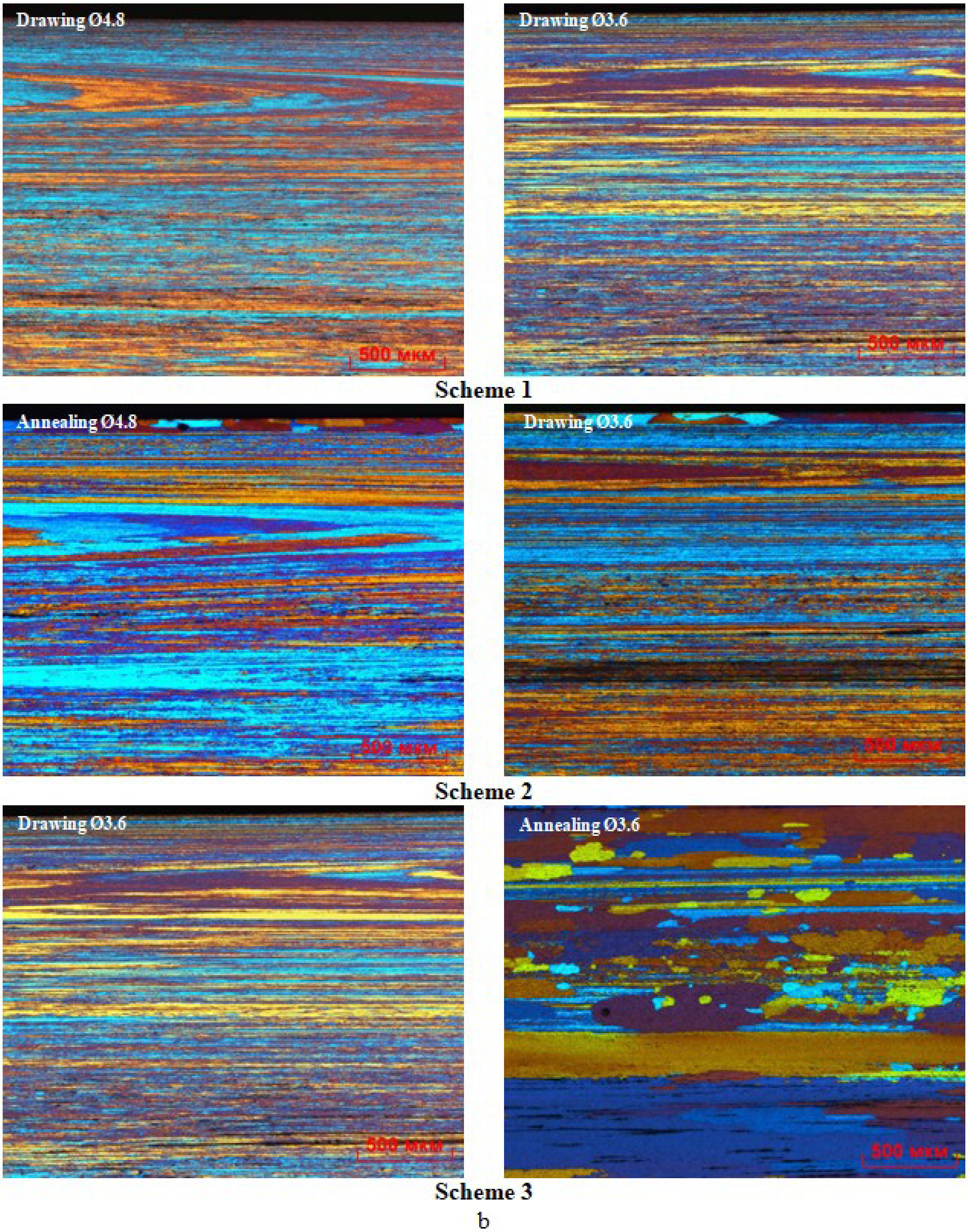

Fig. 2. Wire grain macrostructure after drawing and stepped annealing 350ºC, 40 h - 440ºC, 20 h: b - alloy 2.

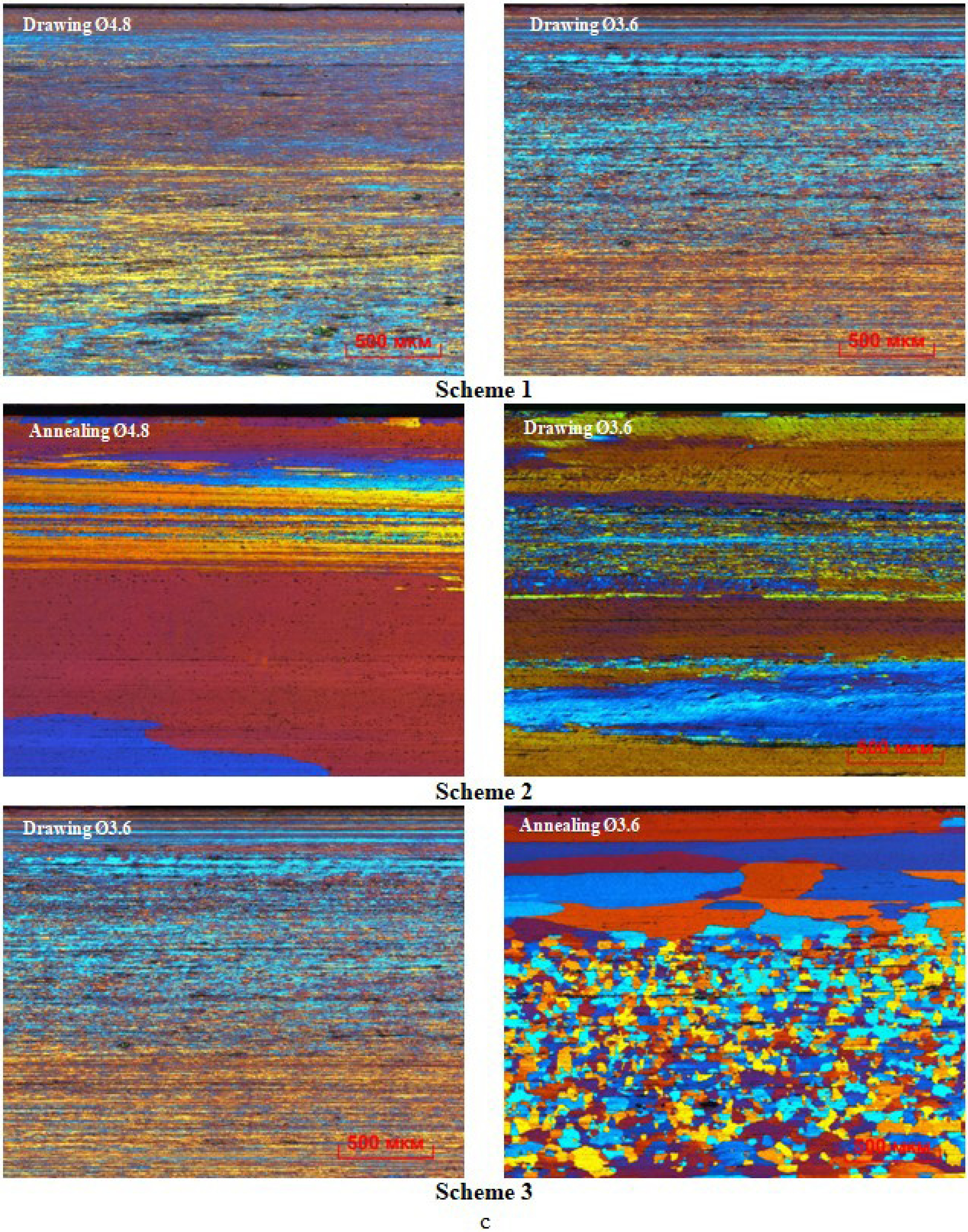

Fig. 2. Wire grain macrostructure after drawing and stepped annealing 350ºC, 40 h - 440ºC, 20 h: c - alloy 3.

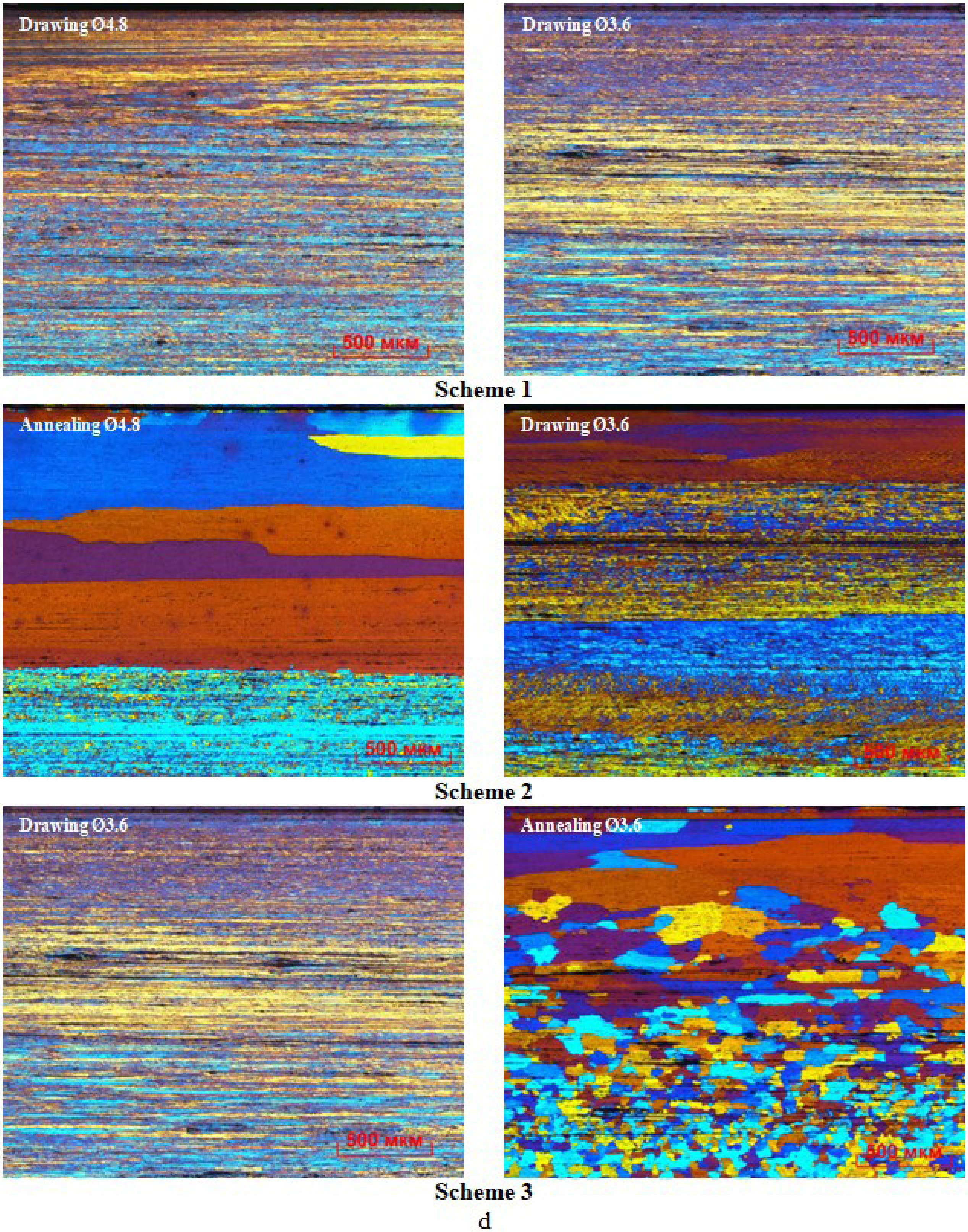

Fig. 2. Wire grain macrostructure after drawing and stepped annealing 350ºC, 40 h - 440ºC, 20 h: d - alloy 4.

iron content from 0.2 % to 0.53 % leads to an increase in the volume fraction of iron-containing phases. The structure of the rods from alloys 3, 4 (Fig. 2(c,d) includes eutectic phases of the Al_4(Ce, La) and Al-Fe-Si types, and in alloy 4 their volume fraction is higher due to the higher concentration of Ce, La and Fe. The structure of the wire grain after IRE and drawing has a fibrous structure (Fig. 2(d)).

Stepped annealing of wire Ø 4.8 and 3.6 mm from alloys 1, 3 (Fig. 2(a,c) leads to recrystallization of the structure and loss of strength, however, an increase in the iron content in alloy 2 and alloy 4 significantly slows down this process (Fig. 2(b,d)).

CONCLUSIONS

The results of the study showed that the use of stepped annealing at 350ºC, 40 h - 440ºC, 20 h at different stages of drawing rods after IRE leads to a significant decrease in electrical resistivity and makes it possible to achieve a good combination of mechanical properties. Wire Ø 0.7 - 1 mm from Al - 0.21 % Zr - 0.53 % Fe and Al - 0.13 % Zr - 0.68 % Fe - 0.6 % Ce - 0.3 % La alloys, manufactured according to technological schemes 2 and 3, has R_m = 172 - 179 MPa, A = 1.5 - 3 % and ρ = 0.0291 - 0.0300 Ohm·mm^2/m.

Acknowledgements

The research was supported by a grant from the Russian Science Foundation No 22-79-00108, https://rscf.ru/project/22-79-00108/.

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