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STRENGTH AND PLASTICITY

Influence of Heat Treatment on the Structure and Properties of an Al–7% REM Conductive Aluminum Alloy Casted in an Electromagnetic Crystallizer

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Abstract—A comparative analysis of a wire of the Al–7% REM thermostable conductive alloy produced by casting into an electromagnetic crystallizer (EMC) and using the granular technology (RS/PM) method has been carried out. The effect of the annealing temperature (up to 600°C) on the structure of Al–7% REM rods produced using the EMC technology has been studied. Mechanical properties and specific electrical resistivity of the thermostable aluminum wire have been analyzed. It has been shown that the physicomechanical properties of the wire made of the rod cast into an EMC are comparable to those of RS/PM-produced wire.

Keywords: conductive alloy, electromagnetic crystallizer, Al–REM alloys, heat treatment, electrical conductivity

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INTRODUCTION

There has been increased interest in recent times in thermostable materials based on aluminum alloys with high electrical conductivity and sufficient strength, which is retained unchanged after heating up to 250–300°C [1–3]. The use of wires made of commercial aluminum and ABE-type alloys is impossible, because even short-term heating above 200–250°C leads to their strong softening [4, 5]. In the 1970–1990s, Dobatkin V. I. and et al. (All-Russian Institute of Light Alloys) [6, 7] suggested alloying aluminum with cerium and other rare-earth metals (REMs), including in particular, the mischmetal (Mm), to create thermostable alloys. The addition of REMs makes it possible to significantly increase the high-temperature characteristics. As a result, a 01417 alloy (Al–7% REM, according to Russian specification TU 1-809-1038-96) was developed. This alloy was intended for preparation using so-called granular technology, i.e., by rapidly solidification with subsequent treatment using powder metallurgy (RS/PM) methods. The high-temperature aluminum alloy 01417 is intended to produce a wire capable of long-term operation at a temperature up to 250°C, which is used to make aircraft on-board wires instead of copper wires, thereby reducing the weight of the product from 100 to 300 kilograms. In this case, the gain in terms of the increase in specific electrical conductivity is up to 30% [8].

Since the RS/MP method is rather complex, an alternative technology has been developed at the Research and Production Center of Magnetic Hydrodynamics. This method makes it possible to produce long-length ingots of a small cross section by casting in an electromagnetic crystallizer (EMC) [9]. Since the cooling rates in both approaches are approximately the same (about 10^{3}°CK/s), the properties of the wires were also assumed to be comparable. Thus, the use of the new technology implies a gain in manufacturability. To substantiate these expectations, a complex of investigations should be performed. In this regard, the aim of this work was as follows:

(1) to study the structure of the 01417 alloy produced by casting in an EMC, starting from the initial rod to the wire, made of this alloy in the process of a thermomechanical treatment;.

(2) to determine the effect of the annealing temperature on the mechanical properties of the wire;

(3) to compare the basic properties (strength/electrical conductivity/thermostability) of the wires of the 01417 alloy produced by two technologies: casting in an EMC (a novel EMC approach;) and the basic RS/PM (base) method.

EXPERIMENTAL

As the initial objects of the investigation, rods made of the 01417 alloy 9.5 and 4 mm in diameter, which were casted in an EMC at the RPC of Magnetic Hydrodynamics under industrial conditions, were used [10]. The chemical composition of the initial rods is given in Table 1.

We first studied the effect of the annealing temperature on the microstructure of the rods, since in the case of alloys of this type the morphology of eutectic inclusions determines both the technological plasticity and the final properties of the deformed semi-finished products [11]. We therefore initially studied the effect of the annealing temperature on the microstructure of rods. The preliminary heat treatment of the experimental samples was carried out in the range of 300–600°C with a step of 100°C using a SNOL 8.2/1100 muffle electric furnace. The 01417 alloy under study is characterized by a highly dispersed eutectic structure, which is almost not revealed by optic microscope. In this regard, the microstructure was studied using a TESCAN VEGA 3 scanning electron microscope.

Apart from structural studies, the hardness of the samples (which correlated with the strength) was measured after each step of annealing was measured using a Metkon DURALINE MH-6 tester under a load of 1 N for 10 seconds.

The annealed rods were then rolled into square wires (2 × 2 mm) using a VEM-3m laboratory-scale rolling mill (MISiS) and were then manually extruded using dies of various diameters into wires 1 and 0.5 mm in diameter. The produced wires were studied in the initial state and after annealing. The schematic of the thermomechanical treatment of the initial rods is presented in Fig. 1 and the evolution of the structure at various stages is given in Table 2.

The tensile tests of the wire were carried out using a Z250 Zwick/Roell universal machine according to the Russian Standard GOST 1497–84 at a loading rate of 10 mm/min. The specific electrical resistance of 1-mm wire was measured using a GOM-802 (GP + RS) digital milliometer in accordance with the Russian Standard GOST 7229–76. The value of the specific electrical resistance was used to calculate the specific electrical conductivity.

Table 1. Chemical composition of the 01417 alloy

Rod diameter	Content of elements, wt %						
	La	Ce	Pr	Fe	Si	Sm	Al
∅9.5 mm	2.3	4.4	0.1	0.2	0.1	0.0	Remainder for balance
∅4.0 mm	2.5	4.7	0.1	0.2	0.1	0.1	

RESULTS AND DISCUSSION

In the Al–Ce phase diagram (it can be applied for most REMs, including mischmetal), a eutectic structure takes place at a very low solubility of cerium in (Al) [6, 7, 12–15]. The most reliable parameters of the eutectic reaction $L \rightarrow$ (Al) + $Al_{11}Ce_3$ (which is also designated as Al_4Ce) are 9.8% and 645°C according to [12, 14, 15].

The maximum solubility of Ce in (Al) changes insignificantly as the crystallization rate (V_c) increases. However, the dispersion of the eutectic and the shift of the eutectic point toward a higher cerium content occur. According to [6], at $V_c = 10^3$ K/s, the eutectic concentration is about 15% Ce. Since lanthanum and other REMs included in the mischmetal form have phases that are isomorphic to those of aluminum-containing isomorphic phases (the general designation $Al_{11}RE_3$) [16], this value can also be accepted for the other alloys of the Al–REM system. It therefore follows that the 01417 alloy should contain approximately equal fractions of primary aluminum solid solution and of the eutectic.

At the first stage, we studied the initial structure of rods with a diameter of 9.5 mm (R1) and 4 mm (R2). Figures 2a and 2b show the microstructure of 9.5-mm (R1) and 4-mm (R2) rods casted in an EMC. The observed initial microstructure is similar to the microstructure of hypereutectic silumins of the AK7 -type

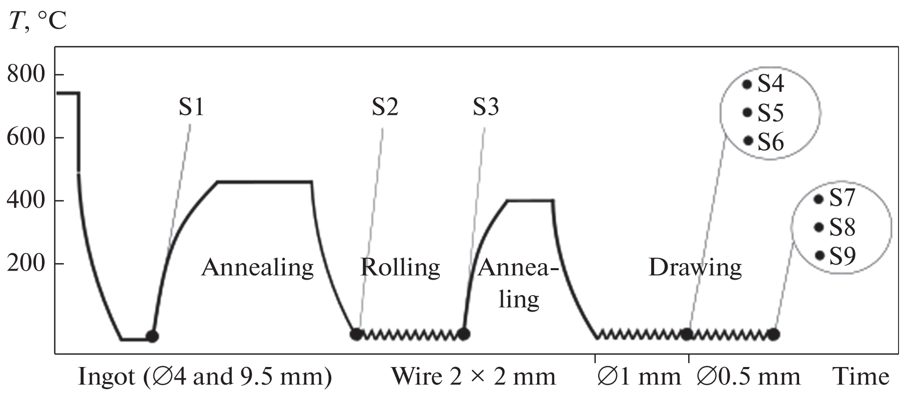

Fig. 1. Technological scheme of the steps of treatment for production of the wire from the initial rods of the 01417 aluminum alloy.

Table 2. Thermomechanical treatment of samples of the 01417 alloy samples (see Fig. 2)

Notation	Operations step	Planned structural changes
S1	Cast rod (9.5 and 4.0 mm in diameter)	Highly dispersed eutectic (Al) + Al_nMm_3
S2	Annealing of rods at 450°C for 3 hours	Submicron-scale fragmented eutectic particles
S3	Rolling of rods to 2 mm in diameter	Deformation strengthening
S4	Drawing of 2-mm wire to 1 mm in diameter	Deformation strengthening
S5	Annealing of 1-mm wire at 300°C for 3 hours	Non-recrystallized (subgrain) structure
S6	Annealing of 1-mm wire at 400°C for 3 hours	Recrystallized structure
S7	Drawing of 1-mm wire to 0.5 mm in diameter	Deformation strengthening
S8	Annealing of 0.5-mm wire at 300°C for 3 hours	Non-recrystallized (subgrain) structure
S9	Annealing of 0.5-mm wire at 400°C for 3 hours	Recrystallized structure

hypereutectic silumins [17]. However, the REM-containing eutectic is much more dispersed than the aluminium-silicon eutectic. The size of the eutectic branches of the aluminide phase $Al_{11}RE_3$ is less than 200 nm and is difficult to define even by SEM. Annealing at 300°C leads to no noticeable changes compared to the cast structure. At 400°C, a partial fragmentation of particles of the eutectic origin occurs (Figs. 2c, 2d). In the R2 rod, the shape changes in that the shape of the particles are more noticeable, which is likely due to the slightly finer cast structure caused by a higher cooling rate during crystallization. This is confirmed by the mean size of the dendritic cells size which was 1.99 (± 0.06) and 1.78 (± 0.06) μm for the R1 and R2 rods, respectively. Annealing at 500°C favors a spheroidization of the formed particles of the aluminide phase in addition to fragmentation. With an increase in the annealing temperature to 600°C, the particles become coarser, which is consistent with the known regularities [11, 17].

Apart from the analysis of the microstructures, the hardness of the rods was measured at each stage of the multistage annealing to evaluate their strength characteristics. The results are presented in Fig. 3. The hardness is seen to decrease as the annealing temperature increases. This is explained by the processes of fragmentation and spheroidization of the highly dispersed eutectics [18]. Note that the experimental samples of various diameter hardness of R1 and R2 at all stages of the heat treatment have comparable values of hardness. Since the formation of coarse particles of the $Al_{11}RE_3$ phase leads to softening (Fig. 3) and since the

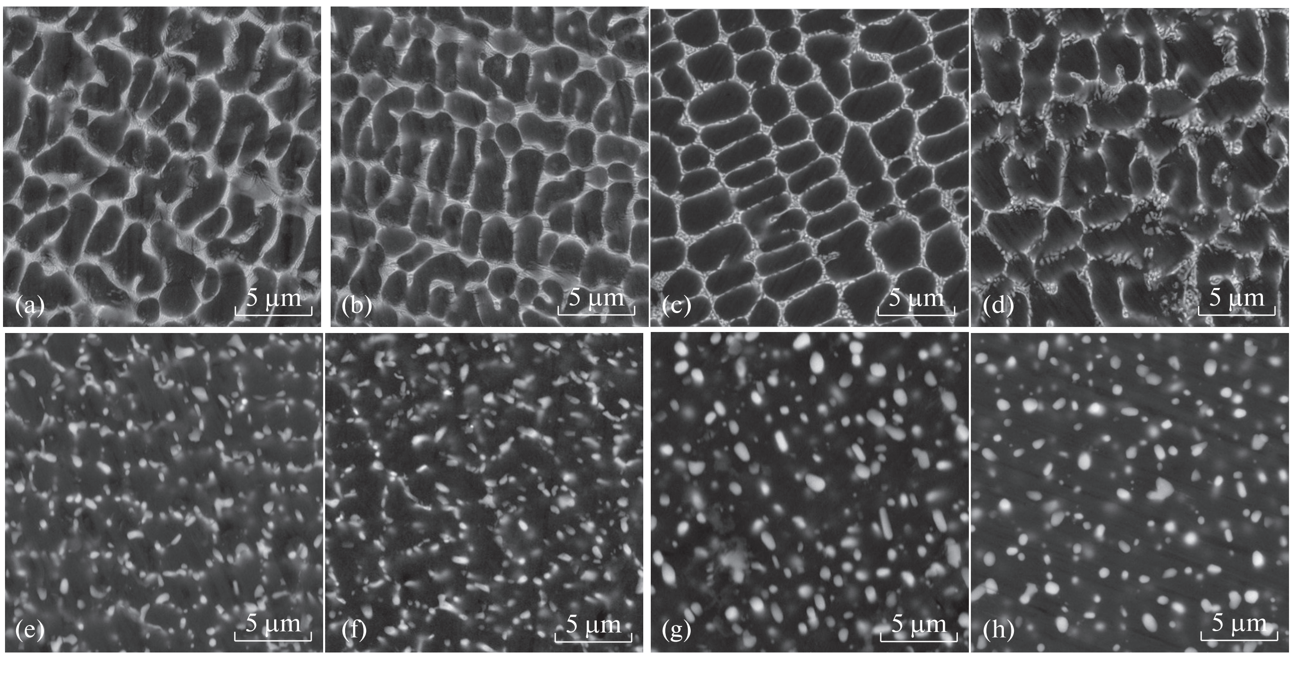

Fig. 2. Effect of the annealing temperature on the microstructure of rods of the 01417 alloy: rods (a, c, e, j) rods of 9.5 mm; and (b, d, f, h) 4 mm in diameter; (a, b) in the initial state; and after annealing at (c, d) 400, (e, f) 500, and (j. h) 600°C for 3 hours.

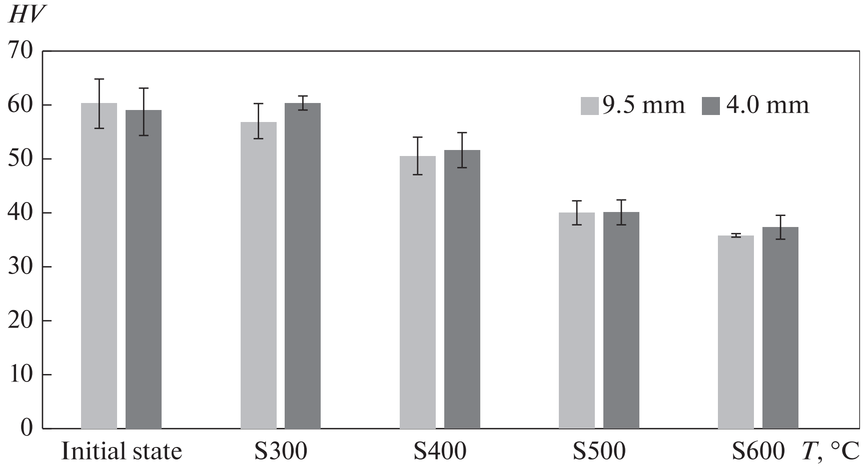

Fig. 3. Effect of annealing on the hardness as a function of annealing temperature of the rods of the 01417 alloy. Annealing time is 3 hours.

initial (unfragmented) structure of the rods does not ensure a necessary deformation plasticity [5–7, 18], choosing the optimal annealing temperature is of great importance. Based on the analysis of experimental and literature data [6, 18], the temperature of 450°C was chosen for the rods under study (Fig. 1).

Table 2 and Figure 1 demonstrate the steps of the technological treatment of cast rods R1 and R2 which consisted of the following steps: (i) annealing of the cast blank billet (S2);, (ii) cold rolling of the rod into a wire with a cross section of 2 × 2 mm (S3);, and (iii) intermediate annealing followed by drawing of the wire to a diameter of 1 mm (S4) and 0.5 mm (S7) in diameter. After the drawing , the wire was subjected to mechanical tensile tests wire in three states: the initial state (S4, S7) and after heating to 300°C (S5, S8) and 400°C (S6, S9) was carried out. The results of the mechanical tensile tests properties are given in Table 3. The diameter of the initial rod is seen to only slightly affect the mechanical properties of the wire. In particular, in the state S4, the ultimate tensile strength is about 230 MPa; the plasticity is about 1%. Annealing at 300°C slightly decreases the strength characteristics. This indicates the development of pre-crystallization processes [11]. After annealing at 400°C, the strength decreases; the plasticity increases to 8%. This can be explained by the occurrence of recrystallization. Such processes for similar systems of alloying were considered in [17].

When measuring the specific electric conductivities of the R1 and R2 rods, a noticeable difference between them was revealed. The specific electric conductivity for the 9.5-mm R1 wire in state S4 was 52.3% IACS; that of the and 4.0-mm R2 wire in state S7 was 52.3% IACS and 56.1% IACS, respectively. This fact can apparently be explained by the difference between the dimensional structural parameters, such as the size of dendritic cells size and distance between the eutectic plates.

When a rolled wire 9.5 mm in diameter for electrical application is produced under industrial conditions using setups of continuous casting and rolling (Continuous Properzi), it is the R1 wire obtained from a cast blank billet with a similar diameter that is of most interest. Figure 4 shows the microstructures of the R1 wire with a diameter of 1 mm in the initial state (S4) and after annealing at 400°C (S6) at various magnifications. The analysis of the microstructures has shown that in the initial state (Fig. 4a), the direction of drawing is observed.

Table 3. Mechanical properties of wire made of the 01417 alloy

Notation	Sample	Mechanical properties		
		σ_u, MPa	$\sigma_{0.2}$, MPa	δ, %
S4	∅1 (from 4)	233	207	0.8
	∅1 (from 9.5)	226	196	1.0
S5	∅1 (from 4)	203	201	0.2
	∅1 (from 9.5)	201	188	0.4
S6	∅1 (from 4)	170	164	2.8
	∅1 (from 9.5)	164	155	8.0
S7	∅0.5 (from 4)	221	193	1.2
	∅0.5 (from 9.5)	221	187	1.2
S8	∅0.5 (from 4)	200	186	0.4
	∅0.5 (from 9.5)	200	179	0.5
S9	∅0.5 (from 4)	167	152	9.1
	∅0.5 (from 9.5)	165	146	9.6

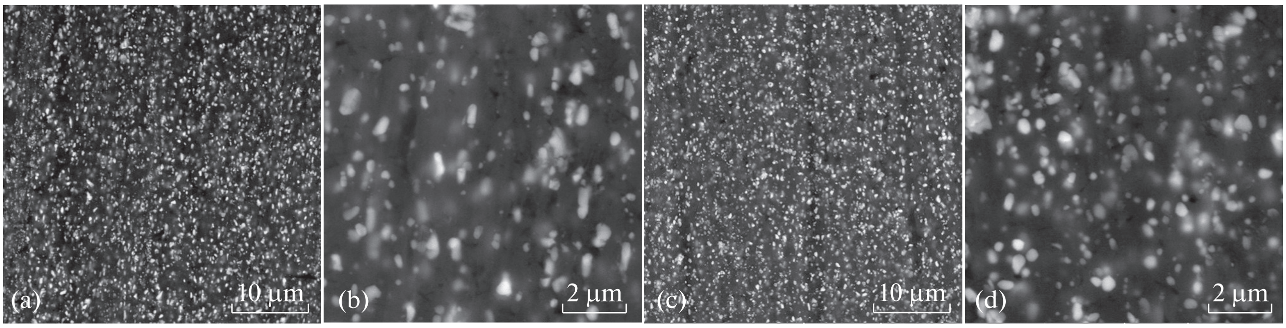

Fig. 4. Microstructure (SEM) of a 1-mm wire produced from a 9.5-mm rod of the 01417 alloy in (a, b) S4 and (c, d) S6 states (see Table 2).

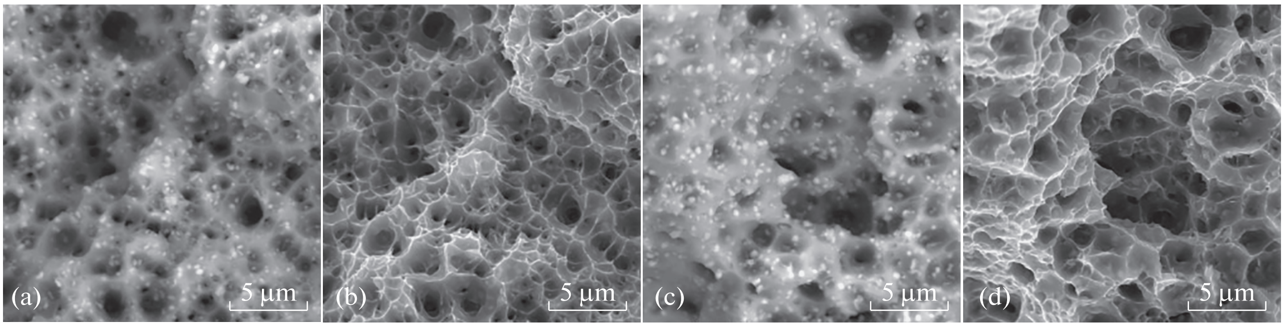

Fig. 5. SEM images of the fracture surface of a 1-mm wire produced from a 9.5-mm rod of the 01417 alloy after tensile tests: (a, b) S4 state and (c, d) S6 state (Table 2);. (a, c) in backscattered electrons and (b, d) in secondary electrons.

Annealing at 400°C leads to spheroidization and some coarsening owing to the coalescence of eutectic particles [17, 19] with an average size of about 0.5 μm (Fig. 4b). Note that the microstructure of the wire in all states contains submicron-scale REM-containing particles. This confirms the correct choice of the annealing temperature of initial rods, since a combination of sufficient plasticity and dispersed structure is achieved [6, 20, 21].

The structure of fracture peculiarities of the wire was analyzed by SEM (in backscattered-electron and secondary-electron modes). In all cases, there is a dimple-type fracture (Fig. 5). The dimples are nucleated at the interfaces between the matrix and the inclusions. The dimples observed in the R1 wire in the state S6 (Figs. 5c, 5d) are deeper than those in the state S4 (Figs. 5a, 5b), which is confirmed by the experimental values of plasticity for these two states (Table 3).

Table 4 shows the properties of the wire of the 01470 alloy obtained by casting in an EMC and by the RS/PM method. The properties of the RS/PM-produced wire with a diameter of 2 mm were taken from [6]. It is seen that the values of the ultimate tensile strength of the wires and their electrical conductivities are comparable. The difference in plasticity is explained by the difference in the deformation method. The mechanical properties and the electrical conductivity of the wire produced from the cast rod R2 are not infe-

Table 4. Physicomechanical properties of the wires R1 and R2 under study and the wire produced by the RS/PM method [6]

Notation	Mechanical properties		Physical properties	
	σ_u, MPa	δ, %	ρ, μOm m	% IACS
R1	226	1.0	0.033	52
R2	233	0.8	0.031	56
01417 RS/PM	180–230	4.6–2.5	0.031–0.032	56–54

rior to the properties of the RS/PM-produced wire. The proposed method of using an EMC seems promising for producing wire made of alloys not only like the 01417 alloy, but also of other alloys of similar type, whose required structure is formed under conditions of rapid crystallization.

CONCLUSIONS

(1) The effect of the annealing temperature (up to 600°C) on the structure of the Al–7% REM alloy fabricated by casting in an electromagnetic crystallizer (EMC) has been studied. It has been shown that upon annealing up to 300°C, the REM-containing aluminides entering into the composition of the highly dispersed eutectic remain practically unchanged upon annealing at 300°C. After annealing at 400°C, traces of a fragmentation are noted. At higher temperatures, a spheroidization of eutectic inclusions followed by their coarsening (up to 1.5 μm) occurs. This is accompanied by a decrease in the hardness.

(2) It has been shown that a completely spheroidized structure formed upon annealing at 450°C makes it possible to produce a wire with a diameter up to 0.5 mm. In the course of deformation, a fibrous structure is formed consisting of aluminum grains containing uniformly distributed submicron-scale REM-containing aluminide particles.

(3) It has been found that the strength properties of the wire annealed at 300°C remain almost unchanged. As a result of annealing at 400°C, the strength decreases, while the plasticity increases. This is caused by recrystallization.

(4) The properties (strength/electrical conductivity/thermal stability) of the wire of the Al–7% REM alloy produced by the novel approach (casting in an EMC (novel approach)) and basic (RS/PM) method, as well as their microstructures, are similar, which agrees with the similarity of their microstructures.

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