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Roasting effect on the electrical resistivity of the Al-0,5%Zr alloys

The effect of regimes of deformation and heat treatment on electrical resistivity and vickers hardness of 2 aluminum alloys containing 0.2% Zr and 0.4% Zr (mass%) was studied. Flat ingots (10x40x200 mm and 40x120x200 мм) were produced by casting into a graphite moulds. ingots were hot and cold rolled to obtain sheets (thickness 1.3 mm and 5 mm for cold rolled and hot rolled accordingly). Ingots and sheets were annealed according to multistage modes in the temperature range 200–650 °C with a step of 50 °C and 3 h holding at each stage. Polished samples cut from the central part of the ingots (as-cast and annealed) were studied. the structure was examined in optical (om, axiovert 200 mmat) and electron scanning electron (tescan vega 3) microscopes. Thin foils for transmission electron microscopy (tem) were prepared by electrolitic thinning in a perchloric acid–alcohol solution and studied at 160 kv.

Key words: aluminum alloys, system Al–Zr, phase Al_3Zr, electrical resistivity, deformation and heat treatment, hardening and softening.

Introduction

In the recent years the interest for the aluminum alloys, strong enough, maintaining high electrical conductivity even after the heating up to 250–300 °C grows. Traditional wire line made of technical aluminum of the A5E mark does not satisfy the requirements given, because it loses the strength even after the short-term heating in such temperature range [1, 2].

The solution of the problem is quiet successful approach on the producing of wire line made of low-alloy aluminum with addition of the zirconium [3–6].

The primary pattern of the aluminum wire line designed to produce the lines (core conductors) according to the rules is rod, obtained by the continuous casting and rolling technique on the properzzi and southwire types of equipment [7]. In the wire line made of Al-Zr alloy all required specifications mainly the electrical resistivity and strength (after heating up to 300^0C) are determined by the rod metallurgical structure.

Aluminum wire thermostability depends on the zirconium in the alloy as well as the melting, casting and heat treatment processing methods. Positive effect of the zirconium on thermostability is condition by the nanoparticles of the l12 (Al_3Zr) phase, formed in the rod when roasting [8–10]. This is exactly what identified the objectives set out in this work, and is the most important:

a) to obtain aluminum alloy hot-rolled sheets, with 0,5% Zr, in case of realization of the continuous casting and rolling on the industrial installations;

b) to study the roasting temperature effect (up to 650 °C and including) on the specific electrical resistivity and strength of these sheets;

в) to rationalize a profitable ratio between the roasting temperature and zirconium concentration.

Experimental technique

In the capacity of the main study objects sheets of aluminum alloy with 0,5% Zr were taken. Experimental alloys were prepared of the primary alumium of the a7e (GOST 11069–2001) mark in the graphite-clay crucible of electrical resistance furnace. Zirconium was introduced into the alloy under temperature of 850–900 °C (i.e. higher than a liquidus point) in the form of Al–15% Zr (GOST 53777–2010) ligature [11]. 40x120x200 mm ingots were obtained by casting into the graphite casting-form. Sheets made of such ingots (figure 1) were obtained as follows: to heat ingots casted to 450 °C, then roll the sheets with 87,5% degree of squeezing and up to 5 mm thickness.

Figure 1. Appearance of the experimental ingots and sheets made of them

Chemical alloys composition study was performed on the ARL 4460 spectrometer, results are shown in the table 1.

Table 1

Chemical composition of experimental alloys

Alloy		Concentration, % wt.			
№	Reference	Si	Fe	Zr	Al
0	00Zr	0,0073	0,140	-	base
1	02Zr	0,072	0,139	0,180	base
2	03Zr	0,074	0,131	0,283	base
3	04Zr	0,080	0,140	0,380	base
4	05Zr	0,075	0,133	0,476	base

A sheet roasting was performed under the temperature of 300–650 °C (table 2), with accuracy ranging of ±5 °C, stepwise in a muffle electric furnace («snol»).

Table 2

Ingots roasting conditions of the Al–Zr–Si system alloys

reference	Roasting method
t 300	t 300 °C, hour
t 350	t 300 +350 °C, 3 hours
t 400	t 350 +400 °C, 3 hours
t 450	t 400 +450 °C, 3 hours
t 500	t 450 +500 °C, 3 hours
t 550	t 500 +550 °C, 3 hours
t 600	t 550 +600 °C, 3 hours
t 650	t 600 +650 °C, 3 hours

Specific electrical resistivity (γ) and Vickers hardness (HV) were measured for each condition. The γ — value was determined by the eddy-current method on the VE-26NP device, then specific electric resistance (ρ) was calculated. Vickers hardness was determined on the Wilson Wolpert 930 N hardness tester with the following parameters: pressure-50 H, hold time — 15 c.

Ingots and sheets metallurgical structure was studied on the light (SM) and electronic scanning microscope (SEM) respectively: Axio Observer MAT and TESCAN VEGA 3. For polished section preparations the electrolytic polishing and mechanical polishing were used under 12V voltage, electrolytic conductor, which has 6 parts of C_2H_5OH, 1 part of $HClO_4$ and 1 part of glycerin.

Fine-structural investigation (first of all to indicate the Al_3Zr precipitation) was carried out on the JEM2100 electronic microscope (TEM) with high resolution and 200kW acceleration voltage. As the objects of study foils obtained by the thinning-down of sheets were used.

For digital analysis of the phase structure (components concentration calculation and the mass fraction of phase — in the aluminum solid solution (Al) the Thermo-Calc (TTAL5 database) software was used.

Experiment results and their analysis

Metallographic ingots examination showed the difference absence in the metallurgical structure between the experimental alloys, because the zirconium upon crystallization was entirely introduced into the aluminum solid solution composition (hereinafter (Al), Fe and Si concentration are approximately the same (see table 1).

Metallurgical structure observed is similar with the electrotechnical aluminium metallurgical structure of the A7E mark: phases inclusions in the form of skeletal fragments or the boundaries of the dendritic cells (Al) [12]. During the rolling process equiaxial grain structure was changed to the fibrous, phases inclusions with the presence of ferrum were extended too.

The roasting has no appreciable effect on the structure detectioned in the light microscope, therefore structural and phase conversion upon roasting were evaluated by the results of special electrical resistivity changes and strength, by the reports results as well. As per condition diagram the Al–Zr experimental alloys (disregarding Fe and Si impurities) at all temperatures fall into the two-phase field $(Al)+Al_3Zr$ (figure 2). Exception is the 02Zr and 03Zr alloys, which at the temperature 650 ^{o}C should be one-phase. Calculations were made upon stable and metastable equilibrium, in accordance with this $D0_{23}$ and $L1_2$ phases [13]. Zr solubility in the solid solution of (Al) increases under temperature over 400–450 ^{o}C. Solubility is significantly higher by the metastable version as well.

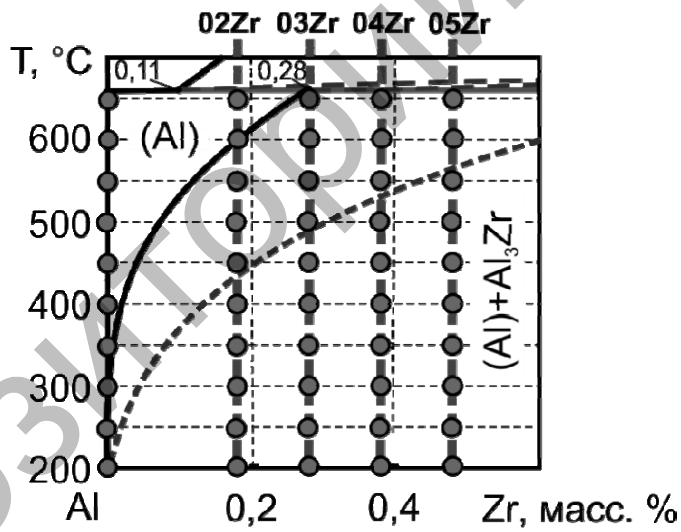

Figure 2. Condition diagram Al–Zr fragment with marked conditions of experimental alloys under different roasting conditions (table 2): dotted graph — the solvus of the $L1_2$ metastable phase

Special electrical resistivity dependence of the experimental alloys on the temperature of the last step of roasting is quite difficult (figure 3). When comparing with unalloyed aluminum, of which resistivity value is comparable to the experimental precision, in the alloys with addition of zirconium a great changes are revealed. Especially they are great upon maximum concentration of zirconium in the alloy (C_{Zr}), 05Zr alloys has the difference in special electrical resistivity approximately of 5,4 Ohm·m·10^{-9} (or ~ 15%).

As can be seen from the dependences, shown in the figure 3a, when multistep roasting is applied a minimum value of electrical resistivity is observed under 450 ^{o}C, it is explained by the maximum reduce of the zirconium concentration ($C_{Zr\text{-}(Al)}$) in the aluminum solid solution. This contradicts with the data, calculated by the metastable version on which under such a temperature the $C_{Zr\text{-}(Al)}$ value is very significant. There are two explanations of this situation. First, upon low temperature (below 400 ^{0}C) zirconium diffusion in the (Al) is quite low, therefore total decomposition process requires more time [14, 15]. By data [10] 500-

hours of roasting fewer than 300 °C is not enough. Second, upon 450 °C, there is possibility to rely on the equilibrium solubilityof Zr in the (Al), which is quiet lower than the metastable version (for $D0_{23}$ phase).

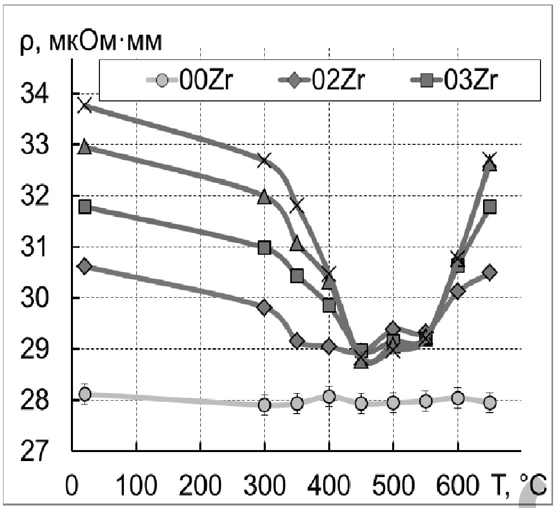

Figure 3. Temperature effect of the last step of roasting on the specific electrical resistivity of the experimental alloys sheet

Upon temperature increase there is effect of C_{Zr} is observed. In the 02Zr and 03Zr alloys there is softening upon temperature of 350 °C and 400 °C is observed respectively. In more hydra metals of 04Zr and 05Zr the hardness is high enough even at 450 °C. At 500 °C and above all alloys are softening.

Closing

Temperature effect of roasting up to 650 °C on hardness and specific electrical resistivity is studied. (SER) of the aluminum alloys hot-rolled sheets with 0,5 % wt of Zr in case of realization on the industrial installations of continuous casting and rolling.

Using the calculations and experimental techniques we defined that SER (specific electrical resistivity of concentrate) mostly depends on the concentration of zirconium in the aluminum solid solution, it is minimum upon 3-hours of roasting at 450 °C. From the other hand, is conditioned with the quanitity of nanoparticles of the $L1_2$ (Al_3Zr) phase, determining the thermo stability, maintaining of the mechanical hardening.

It is shown that a good alignment of SER values, strength and thermo stability is possible to achieve, if the temperature of thermal heating will be 400–450 °C, and concentration of zirconium is not below 0,3%.

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Д.У.Смагулов, Н.А.Белов, А.М.Достаева

Al-0,5%Zr қорытпаларының электр кедергісіне күйдірудің әсері

Соңғы жылдары тіпті 250–300 ^{o}C дейін қыздырудан кейін де сақталатын жоғары электрөткізгіштік пен жеткілікті беріктіктен тұратын алюминий қорытпаларына деген қызығушылық артуда. А2Е маркалы техникалық алюминийден жасалған дәстүрлі сымдар бұл талаптарды қанағаттандырмайды, себебі олар осындай температуралар кезінде қысқа мерзімді қыздырудан соң да беріксізденеді. Мақалада құйма және беттер түрінде орындалған алюминий қорытпаларының меншікті электр кедергісіне күйдірудің әсері зерттелген. Салқын илемделген беттерде ыдырау баяуырақ жүретіні анықталған. 450 ^{o}C кезінде күйдірілген беттерде электркедергісі мен каттылықтың жақсы үйлесімді мәніне қол жеткізуге болатыны көрсетілген.

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Влияние обжига на удельное электрическое сопротивление Al-0,5%Zr сплавов

Изучено влияние режимов деформационно-термической обработки на удельное электросопротивление (УЭС) и твердость двух алюминиевых сплавов, содержащих 0,2 и 0,4% Zr и выполненных в виде слитков и листов (холоднокатаных и горячекатаных). Установлено, что медленнее всего распад последнего происходит в слитках, а наиболее быстро — в холоднокатаных листах. С использованием функции желательности показано, что наилучшего сочетания значений УЭС, твердости и стойкости к разупрочнению можно добиться в холоднокатаных листах сплава с 0,4% Zr, отожженного при 450 ^{o}C. Такой комплекс свойств обусловлен, главным образом, формированием достаточного количества наночастиц фазы L12 (Al_3Zr), которые определяют сохранение деформационного упрочнения.