



# Effect of composition and quenching cooling rate on the structure and morphology of Al-TM, Al-TM–REM alloys

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## Abstract

The changes in the structural state of the base Al-3Zr wt% alloy depending on the casting temperature, quenching cooling rate, and the target alloying were studied. It was shown that low cooling rate of  $10^1$  deg/sec of the base alloy causes formation of two preferential size (about 10 and 2  $\mu\text{m}$ ) of dispersed particles that uniformly distributed in the Al-0.12Zr wt% matrix. Increasing cooling rate to  $10^3$  °/s causes rise of Zr content in the matrix from 0.12 to 2.4 wt%. Thus, in this case, a precondition of formation of nano-sized Al<sub>3</sub>Zr hardening particles is appeared. At the highest cooling rate ( $10^6$  °/s), turbulent convection flows are formed, as well as a vortex structure with Al<sub>3</sub>Zr particles. The addition of 3–4% Mg to the base alloy increases the melt viscosity, suppresses thermal diffusion processes, levels the concentration on the free and contact side, and increases the degree of supersaturation of the solid solution. The use of Sc, Er micro-alloying completely eliminates the formation of large particles, and contributes to the refining of primary intermetallic compounds to  $d \sim 1 \mu\text{m}$  with a significant increase in their precipitation density.

**Keywords** Al-TM · Al-TM–REM alloys · Nanoparticles · Melt spinning · Aging · Anomalously supersaturated solid solution · Magneto-hydrodynamic (MHD) stirring · Dispersion hardening

## Abbreviations

TEM	Transmission electron microscopy
SEM	Scanning electron microscopy
DO <sub>23</sub>	Crystal lattice structure
TM	Transition metals
REM	Rare-earth metals
MHD	Magneto-hydrodynamic (stirring)

## Introduction

Modern structural aluminum alloys are dispersion-hardened multicomponent and multiphase alloys. The large volume fraction of dispersed reinforcing particles in the matrix allows to achieve a high level of strength and operational

characteristics. They determines the possibility of their use for critical structures. However, the possibilities of improving the properties of Al-based alloys due to alloying with components having high solubility in Al, taking into account the improvement of deformation and heat treatment, are largely exhausted. For this reason, the prospects for the creation of new Al-based alloys with improved properties (strength, heat and corrosion resistance, and operating and technological characteristics) are associated with the formation in the Al matrix of a large volume fraction of highly dispersed refractory trialuminides of TM (transition metals) and REM (rare-earth metals). Thus, the design of new-generation Al-based alloys requires new approach to significant increase the concentration of low-soluble transition metals (TM), rare-earth metals (REM) in aluminum, and development of dispersing methods of trialuminides. Conventional methods of quenching and subsequent aging in this case are not effective.

Suppression of parted diffusion processes at the crystallization front due to a rapid quenching of alloys from the liquid state ( $10^4$ – $10^6$  °/s) allows to significantly increase the content of low-soluble elements in the alloy, which creates preconditions for design a new generation of dispersion-hardening alloys with high functional and operational properties.

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Studies of rapid quenched Al alloys alloyed with insoluble transition and rare-earth metals have shown the fundamental possibility of obtaining supersaturated solid solutions of these elements in aluminum (Dobatkin et al. 1995; Napalkov and Makhov 2002). Control of the decomposition kinetics of such solid solutions makes an opportunity to form a structure that provides increased strength and thermal stability. AIRBUS has developed the “scalmalloy” technology, which allows obtaining an abnormally supersaturated solid solution with low-soluble refractory elements in Al–Mg by melt spinning and subsequent hot pressing (Scalmalloy 2017). Multicomponent AlMgScZrMn alloys were used which are characterized by high specific strength  $\sigma_B \sim 650$  MPA (at  $\gamma \sim 6.5$  g/cm<sup>3</sup>), good weldability, and high corrosion and fatigue properties. An essential disadvantage of this technology is the problems with degassing of shredded ribbons and phase transformations during high-temperature compaction of aged alloys.

The aim of the study is to design a new technological approach for the production of dispersion-hardening alloys with Al–TM ligatures using rapid quenching from the liquid state. It should change the structure of ingot and reduce grain size, as well as provide anti-recrystallization and

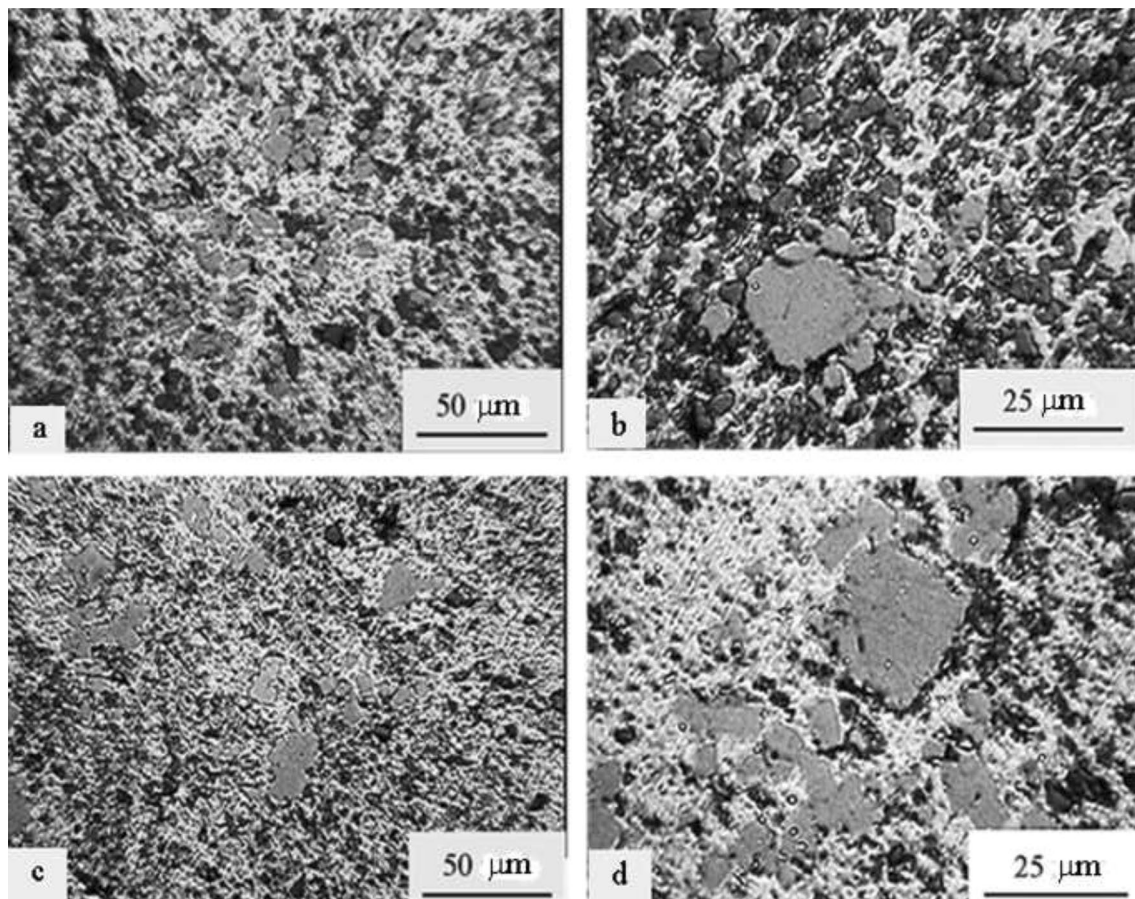
strengthening effects due to formation of nano-dispersed particles in the course of decomposition of abnormally supersaturated solid solutions during high-temperature annealing and thermo-mechanical treatments.

An Al–Zr ligature was selected as the base alloy for the study, which can be considered as a candidate to replace an expensive Al–Sc ligature. The decomposition of the Al–Zr alloy occurs with the formation of nano-dispersed particles of the metastable Al<sub>3</sub>Zr phase, completely isomorphic to the stable Al<sub>3</sub>Sc strengthening phase.

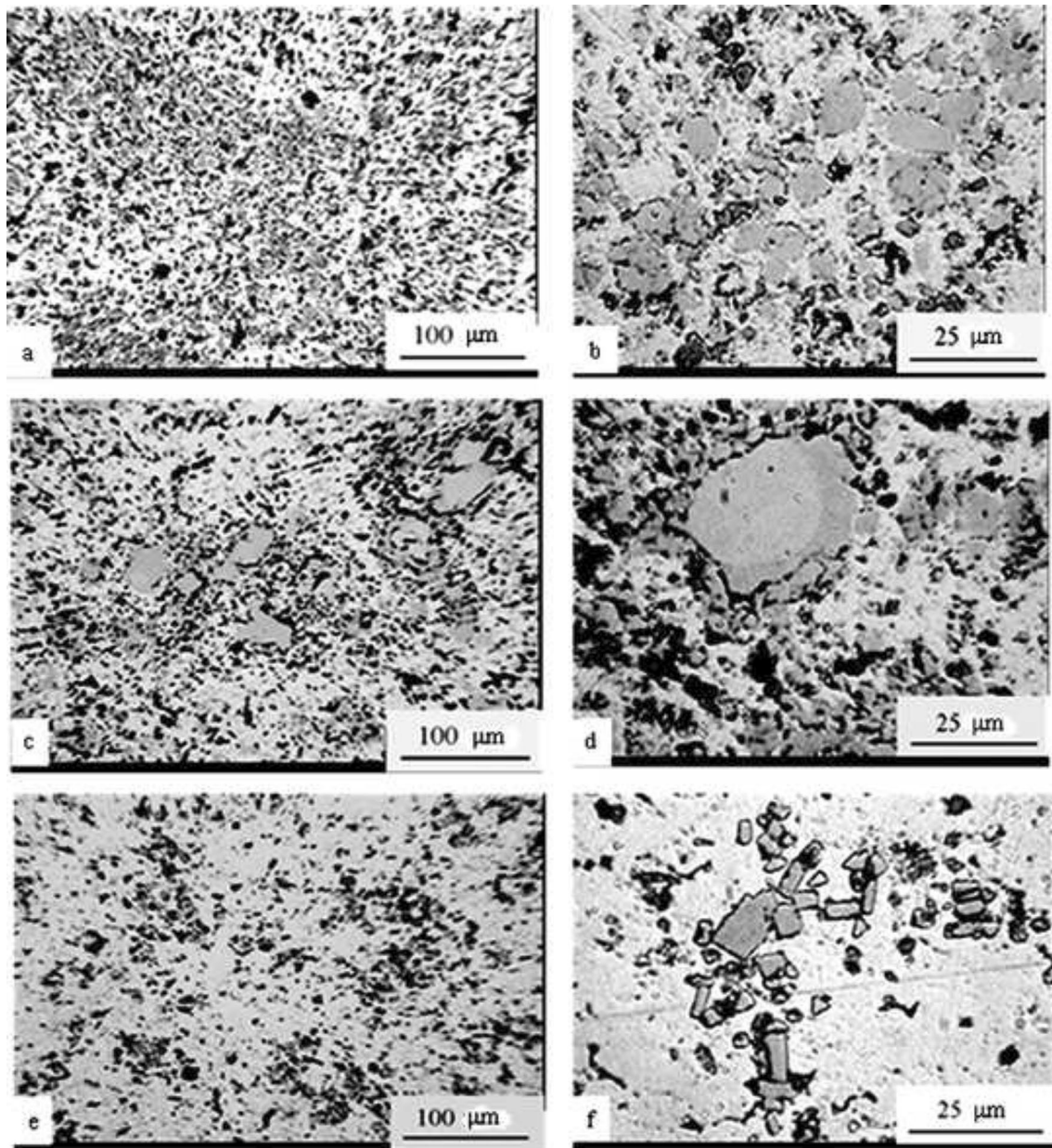
## Experimental

To study the structure of the samples, both structure sensitive properties (microhardness and electrical resistivity measurements) and direct structural methods (metallographic, X-ray, transmission (TEM), and scanning (SEM) electron microscopy) were used.

The microstructure was studied on an NEOPHOT-2 microscope in the range of optical magnifications from  $\times 50$  to  $\times 1000$ . Etching of the samples was performed with



**Fig. 1** The influence of the mixing time of the melt at 700 °C in the MHD installation on the size of the intermetallics in the Al<sub>3</sub>Zr alloy: **a, b**  $t = 1$  h; **c, d**  $t = 2$  h



**Fig. 2** Effect of additional alloying on the distribution of particles in the matrix of the Al3%Zr alloy: **a, b** the original alloy Al3%Zr; **c, d**—Al3%Zr2%Sc; **e, f** Al3%Zr5%Mg. Casting temperature 700 °C; cooling rate ~ 10 °C/s

Keller's reagent of following composition: HNO<sub>3</sub>—2.5 ml, HCl—1.5 ml, HF—1 ml, and H<sub>2</sub>O—95 ml.

Electron microscopic studies were performed on a JEM-2000FXII transmission electron microscope. The dark field technique was used to analyze the precipitation phases. Phases of type L1<sub>2</sub> were investigated in superstructural reflexes (100) and (110). To prepare the foil for TEM, a 20% solution of HNO<sub>3</sub> in methyl alcohol at a temperature of – 30–(– 40) °C was used.

The microhardness values were measured with a PMT-3 Vickers tester using 10 g loads, and the time of loading was

12 s. The average microhardness value was determined by  $n \geq 20$  measurements.

Measurements of electrical resistivity were performed in automatic mode on samples of 50 mm long and 1–2 mm wide during their continuous heating in the range from 20 to 600 °C with specified heating rates or during long-term isothermal annealing. To determine the temperature interval of aging and the activation energy of aging processes, the reduced temperature coefficient of electrical resistance  $\alpha = 1/R_0 \times dR/dT$  was analyzed, where  $R_0$  is the initial resistance of the sample at room temperature.

**Fig. 3** X-ray analysis of the phase state of Al–Zr ligatures obtained by ingot technology

## Results and discussion

Comparative analysis of the structural state of ligature alloys depending on Zr concentration, casting temperature, and cooling rate was provided to investigate the factors determining the formation of abnormally supersaturated Al solid solutions alloyed with low-soluble refractory element Zr. The Zr content varied from 1 to 9 wt%. Liquid casting temperatures ranged from 750 to 1400 °C. Cooling rate varied from  $\sim 10^1$  °/s during die casting to chill mold to  $\sim 10^3$  °/s during quenching on a water-cooled copper base (rapid cooling) and up to  $\sim 10^6$  °/s (ultra-rapid cooling) during quenching on a rotating copper drum (melt spinning).

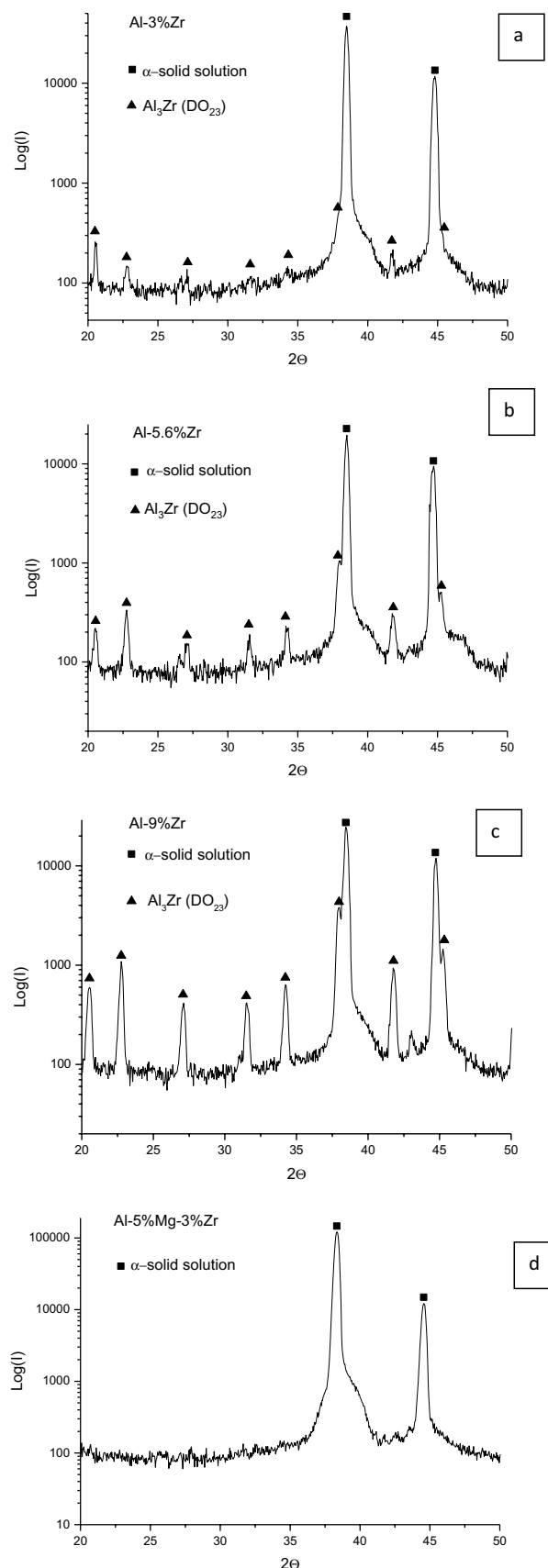
### Ingot technology (melt cooling rate $\sim 10^1$ °/s)

General characteristics of the Zr concentration effect, MHD time of melt treatment, and additional alloying of Mg, Sc on the structural state of Al–Zr ligature.

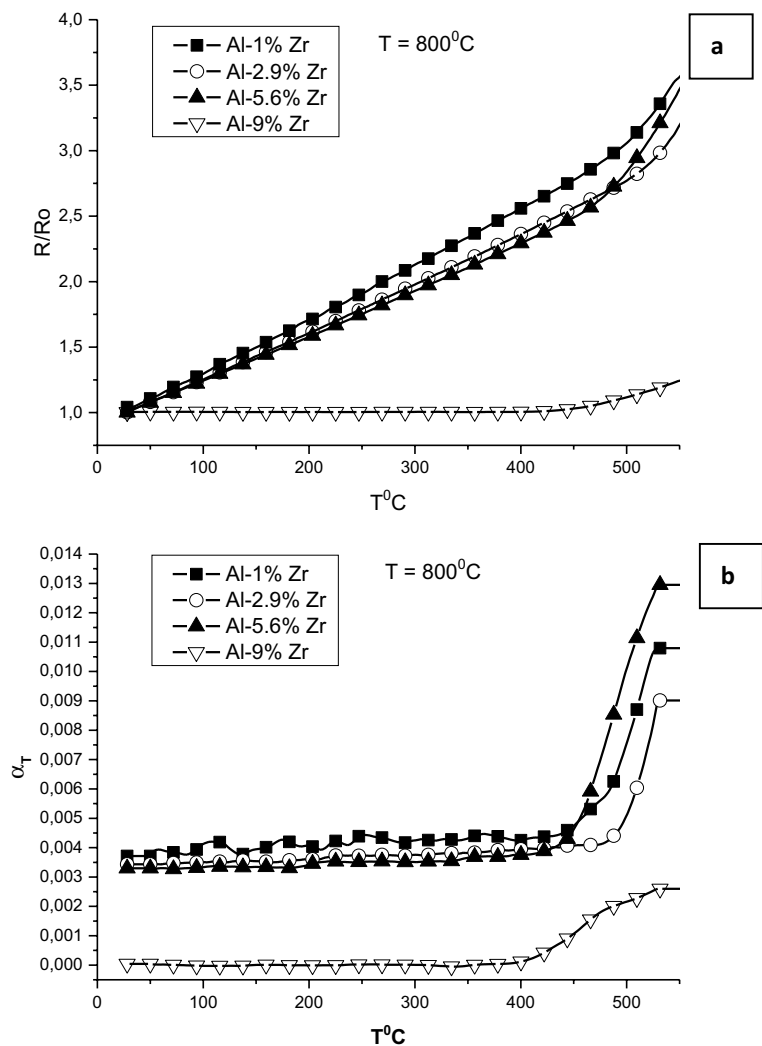
Al–Zr alloys for the present study were obtained by a direct alloying of iodide Zr and Al in a vacuum MHD unit FTIMS NAS of Ukraine, which provides intensive electromagnetic mixing of the melt. Zr was introduced into molten Al in the form of chips with a size of  $2 \times 10$  mm. The melt of Al3%Zr composition was hourly processing in the MHD unit and thereafter cooled with rate of  $10^1$  °/s. Two types of intermetallics were observed in the alloy, small ( $d \sim 2\text{--}3$   $\mu\text{m}$ ) particles uniformly distributed in the matrix and clusters of large ( $d \sim 10$   $\mu\text{m}$ ) particles. All small particles had an equiaxed round shape and differ in etching ability from large particles, which may be evidence of their different composition (Fig. 1 a, b). Increasing the melt processing time in the MHD equipment is accompanied by coalescence of particles, which leads to an increase in their size and coarsening of the structure (Fig. 1 c, d).

Al3%Zr ligature was further alloyed with Mg and Sc. Mg has a high solubility in Al and reduces the crystallization temperature, Sc helps to stabilize the metastable Al<sub>3</sub>Zr phase L1<sub>2</sub> (Fig. 2). The introduction of Sc to the ligature alloy Al3%Zr led to a roughening of the structure. Intermetallic clusters up to 30  $\mu\text{m}$  in size appear in the matrix (Fig. 2 a, b, c, d). In contrast to Sc, magnesium significantly reduces the size and density of dispersoids, and intermetallics are faceted (Fig. 2 d, e). It indicates a deceleration in both coagulation and coalescence of particles.

X-ray phase analysis of all double Al–Zr ligatures obtained by ingot technology, after hourly processing of the melt in the MHD installation and casting from a temperature of 700 °C–800 °C, reveals the presence of equilibrium intermetallic Al<sub>3</sub>Zr-type DO<sub>23</sub> in  $\alpha$ -solid solution (Fig. 3). An



**Fig. 4** Temperature dependence of electrical resistivity  $R/R_0$  and  $\alpha_T$  for studied alloys. Casting temperature 800 °C, cooling in the mold



increase in the Zr content leads to an increase in the volume fraction of the stable  $\text{Al}_3\text{Zr}$  phase.

In the ternary Al–Mg–Zr ligature, any intermetallics were not detected by X-ray, which indicates an anomalous supersaturation of Zr in  $\alpha$ -solid solution.

The concentration of Zr in the solid solution was determined from the curve's slope of the temperature dependence of electrical resistivity. It was taken into account that 1% Zr (by weight), which is in solid solution, increases the electrical resistance of high-purity aluminum ( $\rho_{\text{Al}} = 2.65 \mu\text{Ohm} \cdot \text{cm}$  at 20 °C) by  $1.74 \mu\text{Ohm} \cdot \text{cm}$  at 20 °C. Taking into account that:  $\rho_{\text{alloy}} = \rho_{\text{Al}} \frac{\alpha_{\text{Al}}}{\alpha_{\text{alloy}}}$  (Alyuminiy 1989), the Zr concentration in solid solution can be calculated by the expression  $X\% \approx 1.52 \left( \frac{\alpha_{\text{Al}}}{\alpha_{\text{alloy}}} - 1 \right)$ .

According to the calculations, in case of ingot technology, the equilibrium Zr content in solid solutions was 0.12, 0.26, and 0.32% for alloy Al1%Zr, Al2.9%Zr, and Al5.6%Zr, respectively. No phase transformations were not observed during continuous heating at a rate of 3 °/s in the

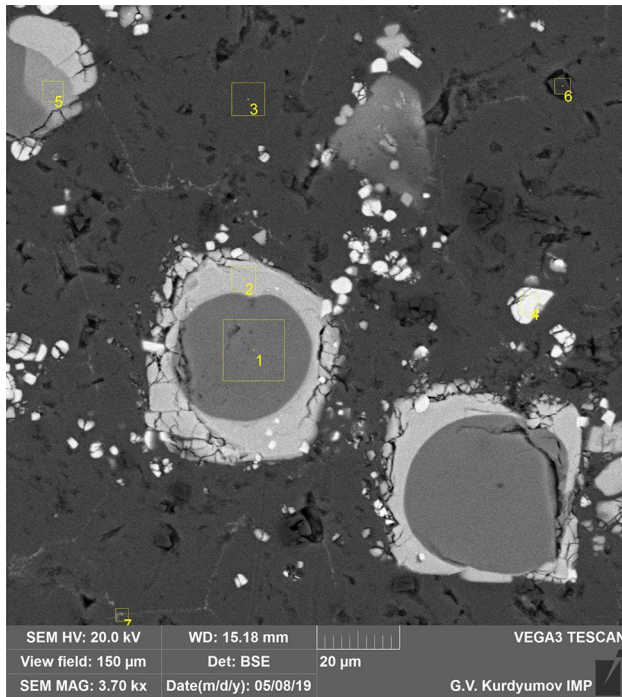
temperature range of 20–400 °C in these alloys. The volume fraction of intermetallics in the Al9%Zr alloy was very large to correctly estimate the Zr content in the matrix (Fig. 4).

### The effect of Sc content on the morphology of the $\text{Al}_3\text{Zr}$ phase

It is known (Diagrammy sostoyaniya 1996) that Sc and Zr have unlimited mutual solubility in the solid state. Data on interchangeability of these elements in the intermetallics  $\text{Al}_3\text{Sc}$  and  $\text{Al}_3\text{Zr}$  are ambiguous, so an attempt to check this issue using a scanning electron microscope in the study of Al3% Zr2%Sc, Al3% Zr0.05% Sc (wt%) alloys was made. MHD melt treatment was performed for 1 h, and the processing and casting temperature was 800°C.

The chemical compositions of the alloy in the points in Fig. 5 are given in Table 1.

As follows from the analysis of the given data, the matrix contains only 0.12% Zr and 0.36% Sc (mark 3). The alloy contains composite particles larger than 40  $\mu\text{m}$ . The core of



**Fig. 5** The structural state of the alloy Al3%Zr2%Sc. Quenching temperature 800 °C; quenching rate  $V=10^6/s$

the composite particles is the intermetallic phase  $Al_3Sc$ ; the shell is a ternary intermetallic compound  $Al_3(Sc,Zr)$  of variable composition (marks 2, 5). The size of  $Al_3Zr$  particles is an order smaller than composite one  $\sim 2\text{--}4\ \mu\text{m}$ , containing up to about 49% Zr and 2.2% Sc.

To prevent the formation of large composite particles, the Sc content in the alloy was reduced to 0.05%. The structure of intermetallics in the alloy Al3% Zr0.05% Sc is shown in Fig. 6.

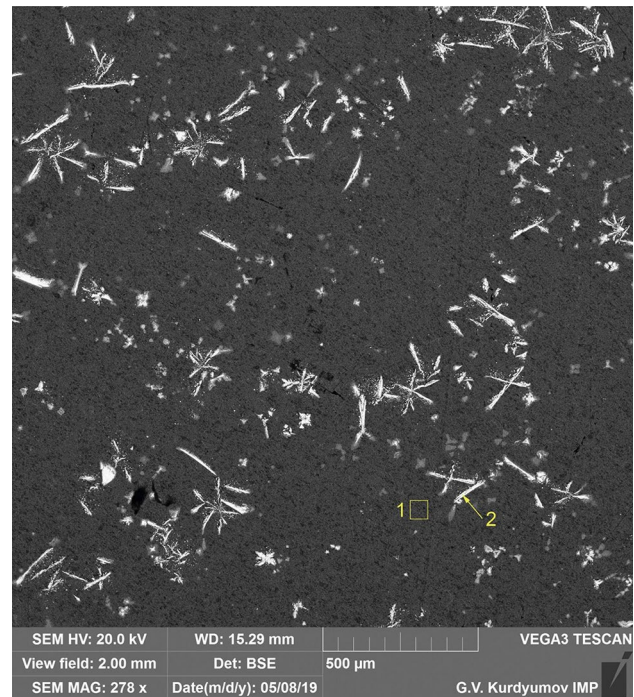
The decreasing Sc content in the alloy is accompanied by an increase in the concentration of Zr in the matrix to 0.12% (Table 2), with a complete change of  $Al_3Zr$  particle

morphology from compact to rod shaped with length of 50–150  $\mu\text{m}$ .

## Effect of melt overheating, rapid, and ultra-rapid cooling from the liquid state, on the structural state of ligatures

### Melt overheating

The effect of melt overheating to 900, 1000, and 1100 °C and cooling rates  $\nu=10^1, 10^3, 10^6\ %/s$  on the change of the Al3%Zr alloy structural state was studied.



**Fig. 6** The structural state of the alloy Al3%Zr0.05%Sc. Quenching temperature 800 °C; quenching rate  $V=10^6/s$

**Table 1** The chemical compositions of the alloy Al3%Zr2%Sc in the points in Fig. 5

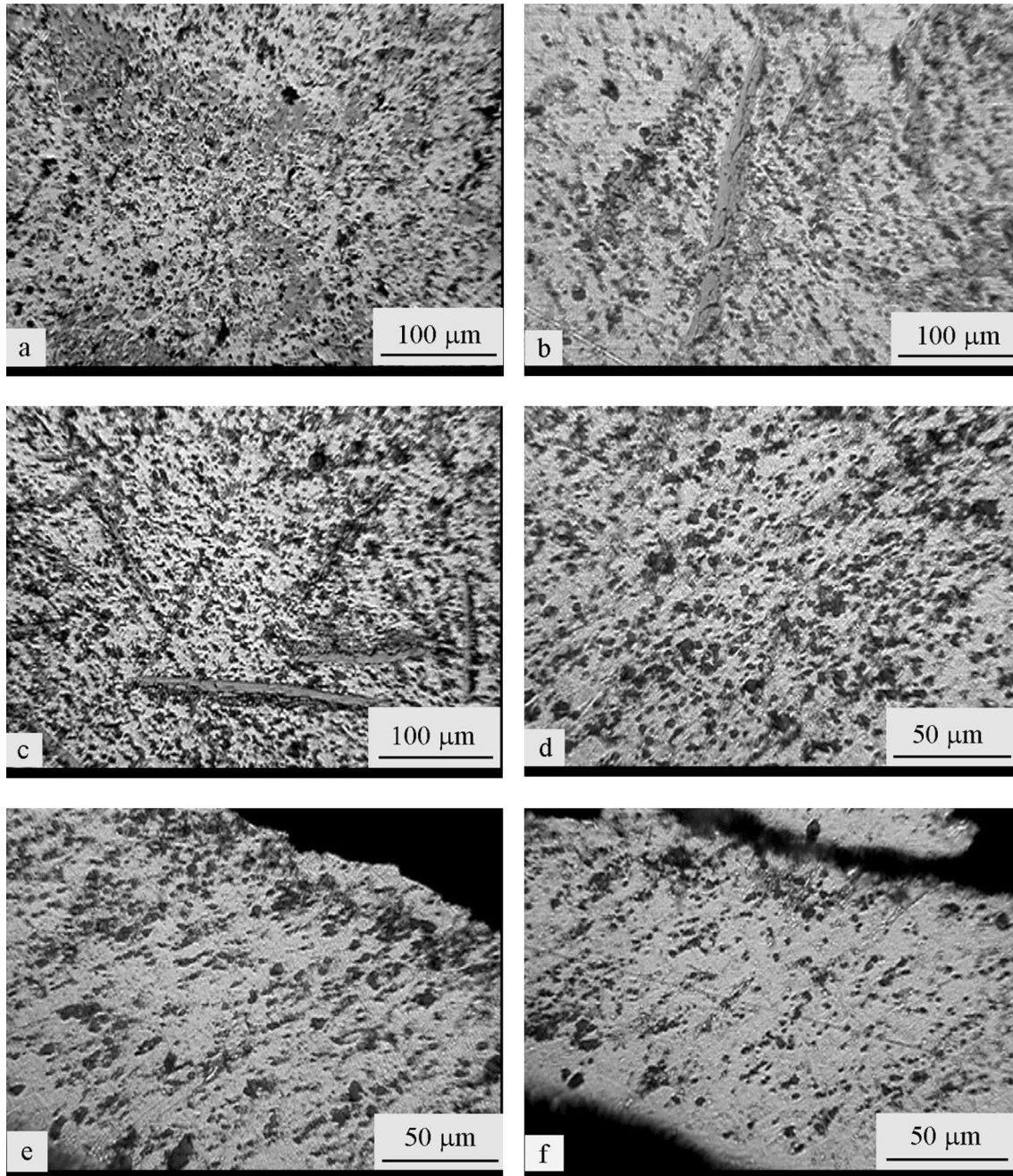
	Al		Sc		Zr	
	wt%	at.%	wt%	at.%	wt%	at.%
1	64.24	74.97	35.72	25.02	0.04	0.01
2	54.14	74.09	17.65	14.49	28.21	11.42
3	99.52	99.75	0.36	0.22	0.12	0.03
4	49.02	75.68	2.21	2.04	48.78	22.27
5	57.05	72.99	27.63	21.22	15.32	5.8

**Table 2** The chemical composition of the alloy Al3% Zr0.05% Sc in the points in Fig. 6

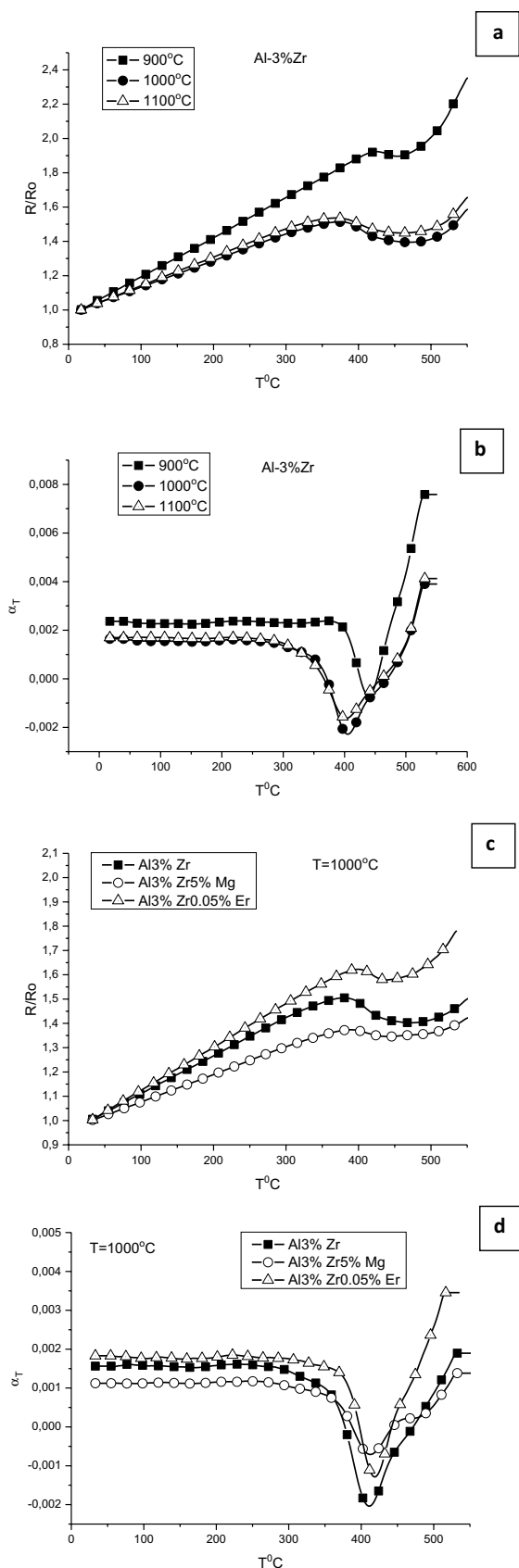
	Al		Sc		Zr	
	wt%	at. %	wt%	at. %	wt%	at. %
1	99.77	99.92	0.04	0.02	0.19	0.06
2	51.47	77.98	0.59	0.54	47.94	21.48

It was found that at low cooling rate in a crucible ( $v = 10^1$  °/s) without the use of stirring in the MHD unit when overheated to 900 °C, the inhomogeneous distribution of intermetallics remains in the matrix with size of large

particles of 4–6  $\mu\text{m}$ , and small of 1  $\mu\text{m}$  (Fig. 7a). When the melt overheats to 1000 °C–1100 °C, particle coalescence processes occur, which are accompanied by self-assembly of intermetallics into rod-like large inclusions



**Fig. 7** Influence of overheating temperature of Al3%Zr melt on equilibrium  $\text{Al}_3\text{Zr}$  particles sizes during cooling in the mold (a, b, c  $V_{\text{cool}} \sim 10^0/\text{s}$ ) and during cooling on a copper plate (d, e, f  $V_{\text{cool}} \sim 10^3/\text{s}$ ). Melt overheating temperatures: a, d 900 °C; b, e 1000 °C; c, f 1100 °C



**Fig. 8** Temperature dependences of resistivity and  $\alpha_T$  for initial (a, b) and doped (c, d) alloys

up to 150–200  $\mu\text{m}$  in length (Fig. 7b, c). When the melt is overheated up to 1000  $^\circ\text{C}$ , formed rods are fragmented with thickness up to  $\sim 7$ –10 microns (Fig. 7b). Increasing the melt temperature to 1100  $^\circ\text{C}$  leads to the compaction of the rods, whose thickness reduced by half, to 3–4 microns, while maintaining the length (Fig. 7c).

### Rapid melt cooling

When the crystallization rate is increased by two orders, up to  $10^3$   $^\circ/\text{s}$ , due to quenching on a water-cooled copper hearth, large particles completely disappear. There are uniformly distributed intermetallic particles in the matrix with size of  $\sim 1$   $\mu\text{m}$  (Fig. 7d). At increased temperature of the melt, 1000  $^\circ\text{C}$  (Fig. 7e) and 1100  $^\circ\text{C}$  (Fig. 7f), the distribution density of  $\text{Al}_3\text{Zr}$  particles in the matrix decreases, which is an indirect indicator of the increase of Zr solubility.

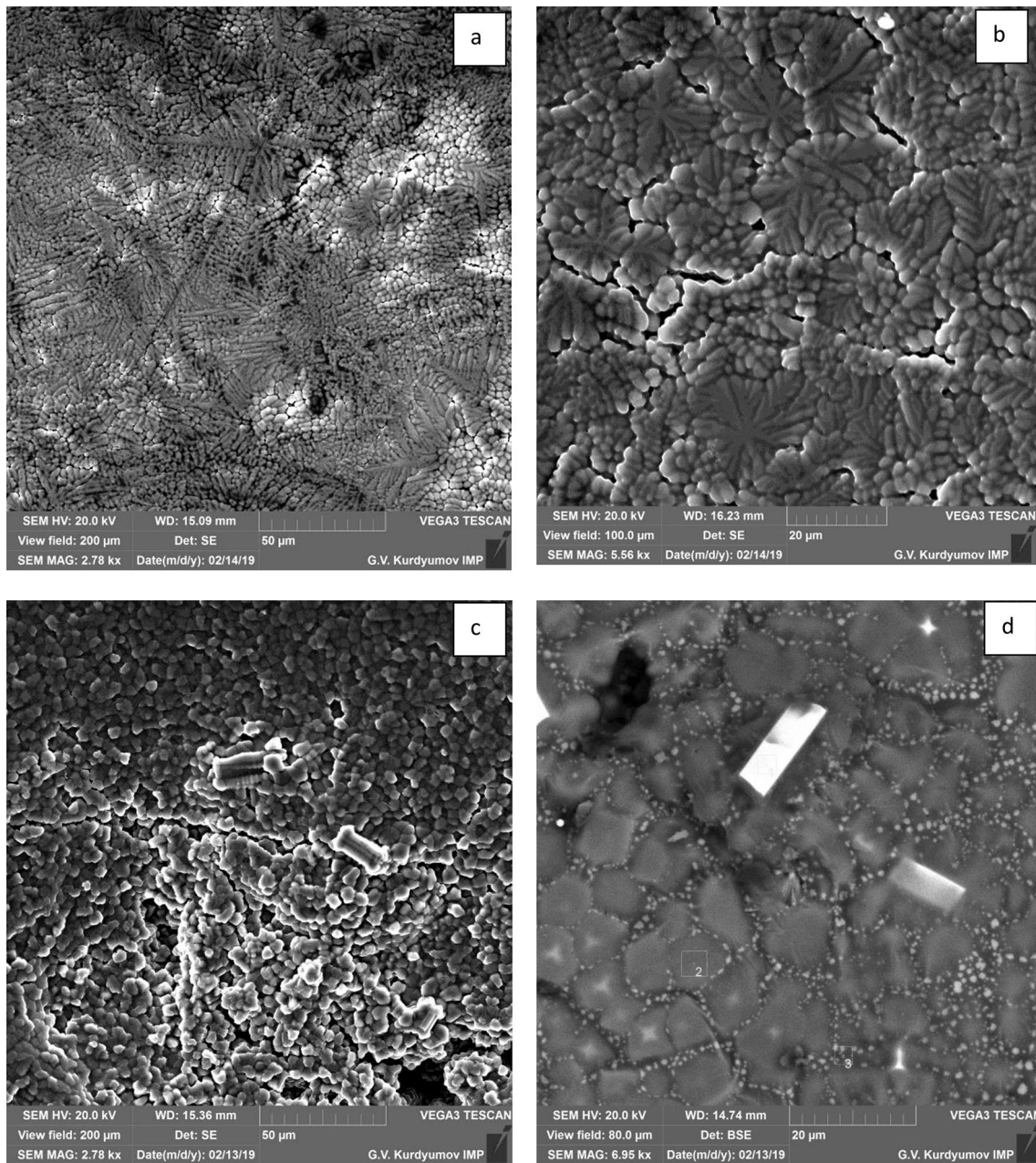
Analysis of the temperature dependence  $R/R_0 = f(T)$  (Fig. 8a) has shown that the content of Zr in the solid solution after quenching from the temperature of 1000  $^\circ\text{C}$  at a rate of  $10^3$   $^\circ/\text{s}$  was 2.4 wt%. Increasing the quenching temperature to 1100  $^\circ\text{C}$  does not increase the solubility. To take into account that the maximum solubility of Zr in solid Al on the equilibrium state diagram comes to 0.11%, the solubility increases more than 20 times and an anomalously supersaturated solid solution is obtained. Thus, in these conditions, it was possible to suppress the processes of separation diffusion at the crystallization front. It follows from the relation  $\alpha_T = f(T)$  (Fig. 8b) that the alloys are capable to decompose in the aging temperature range 350–500  $^\circ\text{C}$  with the maximum decomposition rate at 400  $^\circ\text{C}$  (Fig. 8). Adding a third element to the binary Al–Zr alloy can change the kinetics. For example, Mg has a high solubility in Al, so slows down the aging process, and Er, which is practically insoluble in Al, accelerates the aging process (Fig. 8c, d).

Doping with magnesium increases the viscosity of alloys, i.e., inhibits diffusion processes in the melt, as follows from Fig. 9 and Table 3. The introduction of zirconium into Al–Mg alloys changes the crystallization structure from dendritic to cellular and it is possible to observe anomalous supersaturation of Zr even at a cooling rate of  $\sim 10^3$   $^\circ/\text{s}$ . It is seen from Fig. 9 that the alloy is overlaid; there are particles of pure zirconium (light particles). Iron, which is present in the alloy as an impurity, segregates to the grain boundaries.

### Ultra-rapid melt cooling

The structure of the alloy changes cardinally at ultra-fast cooling rate of the melt ( $10^6$   $^\circ/\text{s}$ ) due to the use of melt spinning during quenching from temperatures of 1000  $^\circ\text{C}$ –1100  $^\circ\text{C}$ . The grain structure is fragmented to micron sizes (Fig. 10), the quality of liquation changes.





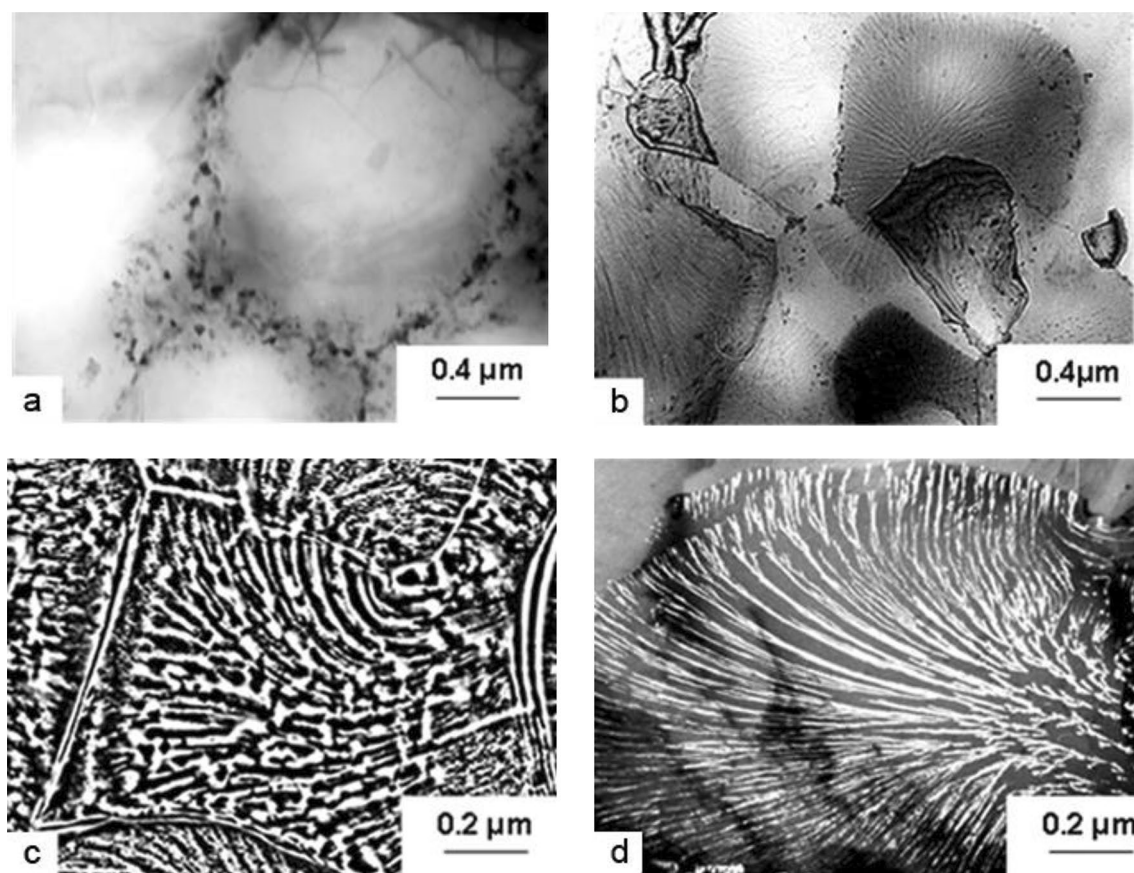
**Fig. 9** Alloy structures Al-6%Mg (a), Al-10%Mg (b), and Al-3%Zr-6%Mg (c, d). Iron segregation at grain boundaries (d)

**Table 3** The chemical compositions of the alloy Al-3%Zr-6%Mg in the points in Fig. 9

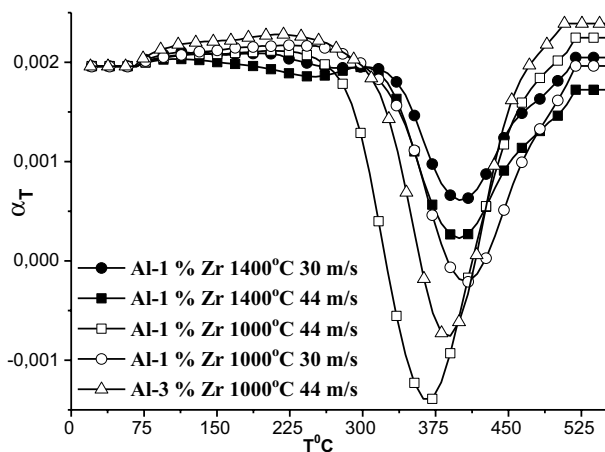
	Al		Zr		Mg		Fe		C		O	
	wt%	at. %	wt%	at. %	wt%	at. %	wt%	at. %	wt%	at. %	wt%	at. %
1	46.72	61.36	45.28	17.59	0.75	1.09	0.13	0.08	5.59	16.48	1.54	3.4
2	85.78	79.33	3.3	0.9	2.06	2.11	0.08	0.04	7.55	15.7	1.24	1.93
3	81.97	77.63	0.0	0.0	4.69	4.93	5.87	2.69	5.97	12.7	1.19	1.89

Usual peritectic type liquation with intermetallics in the center of grains for cast Al–Zr alloys changes on liquation of eutectic type with allocation of intermetallics on grain

borders. Size of intermetallics was ~20–30 nm. (Fig. 10a). When the melt temperature decreases to 1000 °C, the formation of a branched “eutectic-like” fan structure is observed



**Fig. 10** Effect of melt-spinning modes on the structure of fast-quenched Al<sub>1,5</sub>Zr alloy ribbons: **a**  $T=1400$  °C,  $V_L=44$  m/s, light field image; **b, c, d**  $T=1000$  °C,  $V_L=30$  m/s: **b** light field image, **c, d** dark field image in the superstructural reflex (111) of the metastable Al<sub>3</sub>Zr phase



**Fig. 11** Temperature dependence of electrical resistance temperature coefficient (heating rate— $3$  °/min)

(Fig. 10b–d) with a branch thickness of 3–5 nm. According to the measurements of electrical resistivity  $\alpha_T=f(T)$ ,

the experimental alloys are abnormally supersaturated and decompose in the range 350–500 °C (Fig. 11). The dissipation heat rate changed by changing the speed of rotation of the water-cooled copper drum. The structural state obtained during quenching from 1000 °C was more sensitive to changes of the cooling rate due to changes in the drum rotation (Fig. 11).

Increased temperature difference due to overheating of the melt and increasing heat dissipation in the moving stream cause convective currents and convective instability vortices. Convection creates a temperature gradient in the melt, which may be accompanied by thermodiffusion, as a result of which convection jets are decorated with zirconium (Fig. 10b–d). It is known that thermodiffusion is usually observed in gases and liquids, where the interaction between molecules is much weaker than the interatomic interaction. Clusters can act as such structural units in metal melts (Napalkov and Makhov 2002). The system loses stability in the area of clusters presence. Nonequilibrium phase transformation occurs with the formation of a dissipative structure (Berezina et al. 2002, 2012; Prigozhin and Kondepudi 2002) at  $T=1000$  °C. The convective flows are destroyed at lower

temperatures ( $T < 1000$  °C) due to the action of viscous friction forces, and there are no clusters at higher temperatures ( $T > 1400$  °C).

## Conclusions

Structural studies of Al–Zr alloys alloyed with different content of Zr (1–9%), heat treated in different conditions: quenching temperatures, the time of magneto-hydrodynamic (MHD) stirring, and crystallization rates proved:

1. Usual methods of obtaining Al–Zr ligatures with some modifications, such as MHD melt stirring and overheating to 1000 °C, do not provide the formation of supersaturated solid solution of Zr in Al.
2. Overheating to 1000 °C in combination with increasing the cooling rate of the melt due to the use of melt spinning makes possible obtaining nanostructured ligatures.
3. An anomalous supersaturation in Al–Mg–Zr alloys occurs, even when using ingot technology, due to the increase in the viscosity of the melt by Mg adding. The usual dendritic structure of crystallization changes to cellular.
4. The principal possibility of obtaining a new type of Al–Zr-based ligatures, which not only modifies the grain structure of the ingot, but also provides an increase in the volume fraction of reinforcing nanoparticles of refractory  $\text{Al}_3\text{Zr}$  phase using Al3%Zr alloy, casting temperature 1000 °C and crystallization speed  $\sim 10^3\text{--}10^6$  %/s was shown.

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## Compliance with ethical standards

**Conflict of interest** The authors declare that they have no competing interests.

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