Study of the thermal stability of structure and mechanical properties of submicrocrystalline aluminum alloys Al-2.5Mg-Sc-Zr

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Abstract. The paper provides the results of experimental studies of the microstructure, mechanical properties, and corrosion resistance of cast and submicrocrystalline (SMC) aluminum alloys Al-2.5Mg-Sc-Zr with different ratios of scandium and zirconium (Sc + Zr = 0.32 wt.%). SMC structure in alloys was formed by methods of severe plastic deformation: equal-channel angular pressing and rotary swaging. In our paper, we demonstrate that the alloys have high thermal stability – the recrystallization point in SMC alloys is 250-275 °C. We prove that the replacing scandium with zirconium leads to an increase in the thermal stability of the solid solution of scandium in cast and SMC aluminum alloys Al-Mg-Sc-Zr.

1. Introduction

Today, the submicrocrystalline (SMC) structure of Al-Mg aluminum alloys is stabilized by controlled separation of Al₁Sc nanoparticles, which provide an additional increase in strength at room temperature, creep resistance, fatigue life, corrosion resistance, etc. [1-8].

The disadvantage of Al₁Sc particles is that their level of thermal stability is not high enough, which is indicated by the rapid growth of previously released particles. This leads to an increase in the size of the stable grain in accordance with the Zener equation, as well as to a decrease in aluminum alloys in accordance with the Orovan equation. This problem is particularly relevant for SMC materials in which the intensity of Al₁Sc particles release and growth can significantly exceed similar characteristics of conventional coarse-grained materials [9-13].

This problem can be solved by partially replacing scandium with rare earth elements and transition metals (M) with (1) lower diffusion coefficient in aluminum (compared to scandium), (2) high solubility in the Al₁Sc phase, and (3) capable of forming particles with an Al₁M structure with a lower growth and coalescence rate (compared to Al₁Sc). This contributes to the formation of the structure "core (Al₁Sc) – shell (Al₁M)» in the resulting particles and a significant decrease in their growth rate.

Based on the preliminary analysis of the literature [14-20], we selected zirconium as an object of our study (M = Zr), since it provides a significant reduction in the growth rate of Al₁Sc particles. We set the total concentration of scandium and zirconium at Sc + Zr = 0.32 wt.%, since this value, based on the
literature data, at the same time provides a minimum volume fraction of primary intermetallide particles and the maximum increase in strength (hardness) during the aging process.

The aim of the work is to study the thermal stability of the structure and mechanical properties of new SMC aluminum alloys Al-Mg-Sc-Zr.

2. Materials and methods

The objects of our study were the following materials: Al-2.5%Mg-0.22%Sc-0.10%Zr, Al-2.5%Mg-0.20%Sc-0.12%Zr, Al-2.5%Mg-0.18%Sc-0.14%Zr, Al-2.5%Mg-0.16%Sc-0.16%Zr, Al-2.5%Mg-0.14%Sc-0.18%Zr, Al-2.5%Mg-0.12%Sc-0.20%Zr, Al-2.5%Mg-0.10%Sc-0.2%Zr. Experimental ingots 22×22×140 mm were obtained by induction casting in INDUTHERM VTC-200 vacuum casting machine. After casting, the alloys were not quenched and homogenized. Equal Channel Angular Pressing (ECAP) of aluminum alloys was carried out using a Ficep HF400L hydraulic press (400 ton) at a temperature of 225 °C. Rotary Swaging (RS) was carried out using a R5-4-21 HMP machine (Germany) at room temperature by deforming a ∅ 20 mm bar into a ∅ 6 mm bar.

The value of specific electrical resistivity (SER) was measured using the eddy current method using a SIGMATEST 2.069 instrument. Mechanical tensile tests were carried out using a Tinius Olsen H25KS tensile testing machine. Creep tests were carried out in accordance with GOST 3248-81. Fatigue tests were carried out according to the cantilever bending scheme on smooth cylindrical samples. The loading frequency was 50 Hz, the cycle asymmetry coefficient was $R_\sigma = -1$. Microhardness measurements were performed using an HVST1000 hardness tester. Tests for intergranular corrosion (IGC) were carried out in an aqueous solution of 3% NaCl+HCl according to GOST 9.021-74. The tests were carried out by the electrochemical method using a potentiostat-galvanostat R20 (Russia). Electrochemical studies were carried out by registering Tafel curves and potential-time dependencies. Fractographic analysis of fractures and microstructure studies were carried out using a Jeol JSM-6490 SEM with an Oxford Instruments INCA 350 EDS microanalyzer. Macrostructure studies were performed using a Leica IM DRM metallographic microscope.

Samples were annealed in an SNOL-1 625/11-43 air oven with a controlled heating system.

3. Experimental results

3.1. Structural studies

In the initial state, cast alloys typically have a homogeneous coarse-grained structure. The average grain size decreases from 1000-1200 µm to 30-40 µm when alloys are doped with scandium and zirconium. The structure of alloys with a total content of Sc+Zr>0.3 wt.% contains single large light particles with an average size of ~ 0.35-1.3 µm. In alloys with a scandium content of less than 0.3 wt.%, primary particles are not found. The results of EDS microanalysis show the presence of scandium in the particles. The structure of the alloys also contains particles with an average size of ~0.5-5 µm in which iron together with Sc and Zr is present.

The study of the kinetics of the decomposition of a solid solution was carried out by studying the electrical resistivity and measuring microhardness. Figure 1 shows the resistivity and microhardness against the temperature during a 30-minute annealing of Al-2.5Mg-Sc-Zr cast alloys (Sc+Zr = 0.32 wt.%), as well as the in SER variation ($\Delta\rho$) against the time of isothermal annealing for the investigated cast alloys. An analysis of the results shows that partial substitution of scandium with zirconium (while maintaining the total amount Sc+Zr = 0.32 wt.%) leads to a monotonic increase in the thermal stability of the solid solution of scandium and zirconium in aluminum (the temperature of the onset of the decrease in resistivity ($T_1$) corresponding to the solid solution decomposition point was chosen as a characteristic of thermal stability): the onset temperature of the solid solution decomposition in the alloy Al-2.5Mg-0.22Sc-0.12Zr is 200 °C, and in the cast alloy Al-2.5Mg-0.10Sc-0.22Zr, the temperature $T_1$ rises to 275 °C. It is also important to note that with partial substitution of scandium with zirconium, an increase in temperature $T_1$ is accompanied by a simultaneous decrease in the rate of release of Al$_3$Sc particles - a decrease in the resistivity ($\Delta\rho$) during annealing. In the cast alloy Al-2.5Mg-0.22Sc-0.12Zr...
the scale of change in SER after annealing at 325°C (30 min) is 0.36 μΩ-cm, and in cast alloy Al-2.5Mg-0.10Sc-0.22Zr, the value ∆ρ is 0.20 μΩ-cm. The results of EDS microanalysis show that the majority of released particles simultaneously contain both scandium and zirconium. This indicates that the released particles are Al₃(ScₓZr₁₋ₓ) intermetallic compounds with the following structure “core (Al₃Sc) - shell (Al₃Zr)”. Thus, it can be argued that the substitution of zirconium for scandium in the composition of the released intermetallic compounds leads to an increase in the thermal stability of the structure of Al-Mg-Sc cast aluminum alloys.

Microstructure studies by SEM that the RS leads to the formation of a mixed grain-subgrain structure with an average fragment size of about 0.5 μm, ECAP produces a SMC structure with an average grain size of about 1 μm. We should note that the analysis of the results of X-ray phase analysis showed a significant difference in the nature of the formed fields of internal stresses after various schemes of severe plastic deformation - in particular, RS creates tensile fields of internal stresses in aluminum rods, the magnitude of which is very large and reaches 200-250 MPa. The ECAP forms compressive fields of internal stresses, the value of which does not exceed 100 MPa.

We also found that the dependence of the SER on the temperature of annealing for all the SMC aluminum alloys went through two stages - the stage of a slight decrease in the SER when heated to a temperature T₁ and the stage of a rapid decrease in the SER to a value corresponding to the annealed state (figure 2). Using the XRD method, we showed that a decrease in the SER at the first annealing stage, which is not observed in cast alloys, is associated with the recovery processes leading to a decrease in the density of defects introduced during deformation (a decrease in the degree of internal stresses).

Figure 1. Dependences of microhardness (a) and electrical resistivity (b) on the temperature during a 30-minute annealing of Al-2.5Mg-Sc-Zr cast alloys with various scandium and zirconium content

Figure 2. Dependence of the electrical resistivity and microhardness on the temperature of a 30-minute annealing of SMC Al-Mg-Sc-Zr alloys obtained by the ECAP
We should note that in the SMC alloys, the onset temperature $T_1$ of the solid solution decomposition within 25 °C corresponds to the value of $T_1$ in the cast alloys – in the alloy Al-2.5Mg-0.22Sc-0.12Zr after ECAP and RS the onset temperature of the solid solution decomposition is 225 °C, and in the SMC alloy Al-2.5Mg-0.10Sc-0.22Zr, the onset temperature of the solid solution decomposition is 275 °C. It is interesting to note that, in alloys with a high zirconium content (0.20, 0.22% Zr), the dependences of microhardness on the temperature of 30-minute annealing do not have a maximum limit corresponding to the process of separation of particles of the second phase, while in alloys with a reduced Zr content, the microhardness growth by the second stage of annealing do not exceed 100 MPa.

3.2. Tensile test
The results of tensile tests of samples of SMC-ECAP alloys at room temperature show that an increase in the zirconium content leads to a monotonic increase in the tensile strength ($\sigma_b$) from 170 MPa to 245 MPa and a slight decrease in ductility (elongation to fracture) from 25-27% to 20%. We should note that the ductility of SMC alloys at room temperature is quite high, although it turns out to be less than the ductility of coarse-grained alloys at room temperature (35-40%). Fractographic analysis shows that fractures of SMC samples after tensile tests are viscous. Fractures of samples of annealed SMC alloys after tensile tests at room temperature have the same characteristic features (crack growth zone and rupture zone) as fractures of unannealed SMC samples. Ductile failure occurs in the form of a shear; annealing at temperature of 400 °C leads to an emergence of shear pits in the rupture zone. We should also note that the recrystallized alloys contain deep pits in the crack growth zone.

Figure 3. Fractographic analysis of fracture samples of the SMC alloy Al-2.5Mg-0.14Sc-0.18Zr after testing at elevated temperatures (300 °C - top, 500 °C - bottom)

Analysis of test results at higher temperatures shows that an increase in test temperature leads to a monotonic decrease of the flow stress and a very significant increase in the ductility of the material – at the deformation temperature of 500 °C and a strain rate of $10^{-2}$ s$^{-1}$ the flow stress does not exceed 20 MPa and elongation in Al-2.5Mg-0.22Sc-0.10Zr alloy exceeds 600%. The fractographic analysis of fractures of the samples after tests for superplasticity shows that the crack growth zone occupies almost the entire fracture area. Failure in the region of crack growth is viscous in nature – there are numerous pits formed as a result of the fusion of micropores. The fractures have a pronounced grain structure, which indicates that testing for superplasticity at elevated temperatures leads to strain-induced grain growth. It is important to note that the fractographic analysis did not reveal any large primary particles.
of intermetallic compounds in the fracture zone. The value of the coefficient of speed sensitivity \( m \), determined from the slope of the dependence of the flow stress on the strain rate in the logarithmic coordinates \( \lg(\sigma_y) - \lg(\dot{\varepsilon}) \), varies from 0.22 to 0.32 (at temperatures of 400 and 500 °C).

In conclusion of this section, we would like to note that the nature of the formed fields of internal stresses has a significant effect on the ductility of SMC aluminum alloys. As shown above, the use of RS leads to the formation of tensile fields of internal stresses, the value of which reaches 200-250 MPa (in contrast to ECAP, which forms compressive fields of internal stresses). The tensile strength of SMC samples obtained by RS varies from 270 to 300 MPa, and ductility - from 6.5% to 11.5%. We also noted a slight simultaneous increase in strength and ductility associated with an increase in the Zr content in the alloys - in the SMC alloy Al-2.5Mg-0.10Sc-0.22Zr, the flow strength is 290 MPa, the tensile strength is 300-305 MPa, the elongation is 11.3-11.5%. Increasing the temperature of the 30-min annealing of SMC alloy with 0.10Sc and 0.22Zr leads to a decrease in strength and increase in ductility – annealing at 500 °C decreases the magnitude of the flow stress to 130 MPa, tensile strength to 210 MPa, and increases the elongation to fracture to 27 %.

![Figure 4](image-url). Tensile stress-strain curves during testing of cast (a) and SMC (b) alloy Al-2.5Mg-0.14Sc-0.18Zr at room temperature after various modes of annealing. SMC structure was formed by RS

3.3. Creep tests

The creep dependences (see Figure 5) have the common three-stage character, and the stationary flow stage in the case of SMC materials is clearly expressed. As the stress and test temperature increase, a monotonic increase in creep rate is observed. An analysis of the dependences of the creep rate on stress (at a fixed temperature) using the power-law creep equation shows that at low temperatures (below the particle release temperature), the coefficient value is \( n = 5-7 \); and tests at higher temperatures that are accompanied by the release of particles are characterized by the value of \( n > 10 \), which does not correspond to the known theoretical creep models [21]. An analysis of the results showed that it is possible to explain the high values of the coefficient \( n \) using a threshold creep mechanism - taking into account the threshold stress in the equation of power creep allows obtaining values of the coefficient \( n \) close to 4-5, considering the fact that the abovementioned stress values depend on the size and volume fraction of second phase particles, as well as the test temperature.

We established that the activation energy of creep increases along with increasing in zirconium concentration from 101 kJ/mol to 123 kJ/mol. The calculated values of the activation energy are noticeably higher than the activation energy of diffusion along the grain boundaries in pure aluminum (84 kJ/mol), which is due to the influence of magnesium - as shown in [22], alloying aluminum with magnesium leads to a significant increase in the activation energy of grain boundary diffusion.

The elongation to fracture of SMC samples is 1.3–2 times (depending on the test temperature) higher than the elongation of coarse-grained samples under similar conditions. The fractographic analysis of fractures shows that fractures are viscous in nature; at the same time testing at elevated temperatures and low stresses results in strain-induced grain growth.
3.4. Fatigue tests

The results of fractographic analysis of fractures of coarse-grained samples show that all alloys contain areas of the fracture origin, a zone of stable crack growth, a rupture zone, and also a zone of structurally dependent growth of a fatigue crack. The incipient fatigue crack is transcristalline in nature and originates on the surface of the sample, while its growth remains viscous. This is also evidenced by the presence of fatigue grooves and fatigue lines in the zone of stable growth of the fatigue crack. Fatigue lines and pits are present in the rupture zone, indicating the viscous nature of the fracture of cast samples. The presence of large primary particles in the fractures was not detected.

Preliminary studies have shown that the formation of a SMC structure does not lead to a change in the nature of fatigue fracture - four types of regions are also present in fractures of SMC samples, and the fracture pattern itself is viscous (see Figure 6).

At the same time, the durability of the SMC material is higher: for example, the endurance limit based on $10^5$ loading cycles for a coarse-grained Al-2.5Mg-0.14Sc-0.18Zr alloy is 60 MPa, and the same value for SMC material after RS can reach 145 MPa. This difference can be observed when comparing the results of fractographic studies of samples at the same levels of durability, but for different conditions of the material: for SMC material at low stress amplitudes, the size of the zone of stable crack growth significantly decreases due to an increase in the size of the zone of structurally dependent crack growth (within this zone, the crack growth rate decreases significantly) (see Figure 6). It is also necessary to emphasize that the indicator in the Baskquin’s ratio for the SMC alloy ($q = 0.137$) is more than 2 times lower than the indicator for the cast state ($q = 0.311$), which indicates a smoother change in durability from the stress level in the SMC state. Similar results were obtained when studying alloys with a different content of scandium and zirconium.

Corrosion-fatigue tests were carried out in a 3% aqueous solution of NaCl. Analysis of the results of corrosion-fatigue tests shows that the durability of the SMC material in air is higher than in the medium: for example, the endurance limit based on $6 \times 10^5$ loading cycles for the SMC alloy Al-2.5Mg-0.10Sc-0.20Zr in air is 120 MPa, however, during testing in the medium it does not exceed 85 MPa (the...
difference is 27% compared to tests in air). We should also note that the indicator in the Basquin’s ratio when tested in air is lower than the indicator for testing in a medium by more than 2 times.

The fractographic analysis shows that the fracture of SMC samples after corrosion-fatigue tests contains a fracture focus area, a zone of stable crack growth and a rupture zone (see Figure 7). The surface of samples contains the traces of corrosion damage, resulting from a prolonged exposure of the sample to corrosion aggressive medium. In the studied case, the incipient fatigue crack is transcrystalline in nature and originates on the surface of the sample, while its growth remains viscous. The presence of traces of corrosion damage to the surface of the sample in the crack nucleation zone indicates the influence of the corrosive medium on the nucleation of fatigue cracks. We should note that the volume of corrosion traces increases with increasing of the stress level, which is rather surprising, since it is traditionally assumed that an increase in the stress amplitude, leading to faster fracture and a shorter exposure of the sample to the medium, should reduce the influence of the corrosion medium on the intensity of the corrosion-mechanical failure. A probable reason for this is a more intense destruction of the protective oxide film on the surface of aluminum alloys associated with an increase in the amplitude of the applied voltage.

![Figure 7. SMC alloy Al-2.5Mg-0.12Sc-0.20Zr after RS. General view of fatigue fractures during testing in air (a. \( \sigma_a = 100 \) MPa, b. \( \sigma_a = 120 \) MPa, c. \( \sigma_a = 190 \) MPa).](image)

### 3.5. Electrochemical corrosion tests

Studies have shown that active etching of the grain boundaries of coarse-grained alloys occurs at an electrolyte concentration of less than 0.9-1 vol.% HCl, while active dissolution of grain boundaries in SMC alloys begins at an HCl concentration of more than 1 vol.%. In this regard, for further research, we selected the composition of 3% NaCl 1%+HCl with a pH = 0.1-0.2. The accelerated dissolution of grain boundaries (dendrite boundaries) in Al-Mg-Sc-Zr cast alloys at lower HCl concentrations (compared to SMC alloys of a similar composition) is obviously associated with a higher local concentration of magnesium at grain boundaries (dendrite boundaries) in cast alloys.

The studies showed that the cathodic and anodic sections of the dependences "current density - potential" for the SMC alloys obtained by RS and ECAP are not symmetrical, which does not allow using the Tafel method to assess the corrosion density. The nature of changes in the corrosion resistance of SMC alloys was assessed on the basis of the analysis of changes in the value of the corrosion potential. We found that the formation of the SMC structure leads to a decrease in the corrosion potential by 0.1–0.15 mV, which corresponds to the scale of the influence of crystal lattice defects on the standard potential of metallic materials. It is interesting to note that an increase in the proportion of zirconium in Al-Mg-Sc-Zr alloys (at a constant amount of Sc+Zr = 0.32 wt.%) leads to a shift of the corrosion potential by 30–40 mV to a more negative region: the corrosion potential of the SMC alloy Al-2.5Mg-0.22Sc-0.10Zr after RS is -731 mV, and the corrosion potential of the SMC alloy Al-2.5Mg-0.10Sc-0.22Zr after RS is -752 mV. The annealing decreases the value of the potential; in fact, in alloys with a higher proportion of Zr this effect is less noticeable – a 30-minute recrystallization annealing of the SMC alloy Al-2.5Mg-0.22Sc-0.10Zr leads to a decrease in the corrosion potential from -731 mV to -792 mV, and a similar annealing of the SMC alloy Al-2.5Mg-0.10Sc-0.22Zr leads to a decrease in the corrosion potential from -752 mV to -780 mV.
Figure 8. Tafel dependences “current density - potential” for SMC alloy Al-2.5Mg-0.1Sc-0.22Zr (a), Al-2.5Mg-0.12Sc-0.20Zr (b), Al-2.5Mg-0.14Sc-0.18Zr (c), and Al-2.5Mg-0.18Sc-0.14Zr (g)

4. Conclusions
1. We studied the features of solid solution decomposition in cast and SMC aluminum alloys. Our research shows that partial substitution of scandium with zirconium (while maintaining their total concentration Sc+Zr = 0.32 wt.%) leads to a monotonic increase in the thermal stability of the Sc and Zr solid solution in aluminum - an increase in the temperature at which the resistivity decreases, corresponding to the temperature at which the solid solution decays, and to a simultaneous decrease in the intensity of the electrical resistivity decrease during the annealing process.

2. Mechanical tests showed that the formation of a SMC structure leads to a significant increase in strength and a decrease in the ductility of aluminum alloys. We determined that the scale of reduction in the ductility of aluminum alloys is due to the nature of the fields of internal stresses that form during severe plastic deformation – the formation of tensile fields of internal stresses during RS leads to a decrease in ductility to 6.5-10% and an increase in tensile strength to 270-300 MPa. The formation of compressive internal stresses in aluminum alloys by the ECAP allows preserving the ductility at a fairly high level (about 20%) with a similar strength of aluminum alloys. The fractures of the SMC samples after tensile tests are viscous.

3. We showed that an increase in test temperature leads to a monotonic decrease of the flow stress and a very significant increase in the ductility of the material – at the deformation temperature of 500 °C and a strain rate of 10^{-2}s^{-1} the flow stress does not exceed 20 MPa and elongation in Al-2.5Mg-0.22Sc-0.10Zr alloy exceeds 600%. A fractographic analysis of fractured samples after tests for superplasticity shows that fractures remain viscous in nature. The fractures have a pronounced grain structure, which indicates the presence of strain-induced grain growth in the process of testing for superplasticity at elevated temperatures.

4. Fatigue tests according to the cantilever bending scheme showed that the formation of the SMC structure leads to an increase in fatigue strength and durability of aluminum alloys by more than 2 times. The results of a fractographic analysis of fractures of coarse-grained samples show that the incipient fatigue crack is transcryalline in nature and originates on the surface of the sample, while its growth is viscous.

5. Creep tests showed that the stationary flow stage for SMC alloys is clearly expressed. The elongation to fracture of SMC samples is 1.3–2 times (depending on the test temperature), which is higher than the
elongation of coarse-grained samples under similar conditions. The exponent in the power creep equation at low temperatures (below the temperature at which particles are released) is n = 5–7. The activation energy of creep increases with increasing Zr concentration from 101 to 123 kJ/mol.

6. The formation of the SMC structure leads to a noticeable increase in the corrosion-mechanical strength of aluminum alloys, while the analysis of the results of electrochemical studies indicates that the aging process of SMC alloys is accompanied by a decrease in corrosion resistance, which is probably due to the release of intermetallic particles at grain boundaries and, as consequence, the formation of additional microvoltaic pairs, which provoke intergranular corrosion. The fractures of the SMC samples after corrosion-mechanical tests are viscous.

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