Studying Thermal Stability of Cast and Microcrystalline Alloys Al-(2.5, 4)%Mg-Sc-Zr

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Abstract. The article presents the results of experimental studies of the structure and mechanical properties of cast and microcrystalline (MC) aluminum alloys Al-(2.5, 4)%Mg-Sc-Zr with a total content of Sc+Zr = 0.32%. The Sc content was varied from 0.10% to 0.22% at an interval of 0.02%, with the Zr concentration in the alloy being changed proportionally. In their initial state, cast alloys are characterized by a homogeneous coarse-grained macrostructure. The structure of the alloy structure presents single submicron particles Al3(Sc,Zr) that are formed during crystallization of the alloys. It has been demonstrated that the recrystallization onset temperature in MC alloys depends on the scandium-to-zirconium ratio.

1. Introduction
Currently, the stability of the microcrystalline (MC) structure of Al-Mg aluminum alloys is ensured by the controlled breakdown of an oversaturated solid solution of Sc in Al with the formation of nanoparticles Al3Sc [1-6]. The downside of Al3Sc particles is their low thermal stability (high growth rate). This leads to an increase in the grain size during annealing of MC alloys where the intensity of the release and growth of Al3Sc particles can significantly exceed similar characteristics in coarse-grained materials. This problem can be solved by partial replacing of Sc with rare earth elements (REEs) and transition metals (TM) with the diffusion rate in aluminum lower than that of scandium and high solubility in the Al3Sc phase [2, 7-11]. This will allow the breakdown of a solid solution to form particles with an Al3M (M – REEs, TM) structure, as well as particles with a "core Al3Sc - shell Al3M" structure with low growth (coalescence) rate.

The purpose of this study is to research thermal stability and mechanical properties of Al-Mg-Sc-Zr MC aluminum alloys where expensive scandium is partially replaced by less costly zirconium.

2. Methods and materials
Al-2.5%Mg-Sc-Zr and Al-4%Mg-Sc-Zr alloys are the objects of the study. The total content of Sc+Zr = 0.32%. The Sc content was varied from 0.10% to 0.22% at an interval of 0.02%, with the Zr concentration in the alloy being changed proportionally. Bars of 20×20×160 mm were produced by
induction casting. The alloys were not tempered or homogenized after casting. The MC structure was formed by the Equal Channel Angular Pressing (ECAP) method at a temperature of 225 °C.

Microhardness measurements were taken with an HVS-1000 microhardness tester (50 g load, 10 s loading time). Specific electrical resistivity (SER) was measured using the eddy current method with a SIGMATEST 2.069 instrument. Microstructure was researched using a JSM-6490 scanning electron microscope with an Oxford Instruments INCA 350 energy-dispersive microanalyzer. Macrostructure was researched using a Leica IM DRM metallographic microscope. Samples were annealed using a SNOL air furnace with a controllable heating system.

3. Experimental results

In their initial state, Al-2.5%Mg-Sc-Zr cast alloys have a coarse-grained homogeneous structure in the central part of the bar and a columnar crystal structure at the edges of the bar. 4%Mg cast alloys have a coarse-grained homogeneous structure with a grain size of ~30-50 μm on the scandium-to-zirconium ratio in the alloy. The structure of alloys with a total content Sc + Zr> 0.30% demonstrates primary particles with an average size of ~0.35-1.3 μm containing scandium. No primary particles were detected in alloys with an Sc content < 0.30%. The research results suggest that SER experimental values are quite close to their theoretical values, calculated using the additivity rule \[12\] (see Table 1). This signals that most of scandium and zirconium is in a solid solution.

| Sc and Zr content | 0.22Sc+0.10Zr | 0.20Sc+0.12Zr | 0.18Sc+0.16Zr | 0.14Sc+0.16Zr | 0.12Sc+0.16Zr | 0.10Sc+0.20Zr | 0.14Sc+0.18Zr | 0.16Sc+0.22Zr |
|-------------------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|
| Al-2.5%Mg-Sc-Zr alloys |
| Experimental     | 4.50           | 4.59           | 4.37           | 4.62           | 4.55           | 4.54           | 4.50           | 4.50           |
| Calculated       | 4.62           | 4.62           | 4.61           | 4.61           | 4.60           | 4.60           | 4.59           |                |
| Al-4%Mg-Sc-Zr alloys |
| Experimental     | 5.43           | 5.24           | 5.37           | 5.39           | 5.34           | 5.37           | 5.25           | 5.40           |
| Calculated       | 5.43           | 5.43           | 5.42           | 5.42           | 5.41           | 5.41           | 5.40           | 5.40           |

Figure 1 shows SER and microhardness dependences on the temperature of a 30-minute annealing of Al-2.5%Mg-Sc-Zr cast alloys. The analysis of the results demonstrates that substituting scandium with zirconium leads to an increase in thermal stability of the solid solution of scandium and zirconium in aluminum. The onset temperature of solid solution breakdown in alloys is 275 °C. At the same time, the rate of Al₃Sc particle precipitation is decreasing. The EDS microanalysis results demonstrate that most of the released particles contain both scandium and zirconium.
Figure 1. Dependences of microhardness (a) and SER (b) on the temperature of a 30-minute annealing of Al-2.5%Mg-Sc-Zr cast alloys with different contents of scandium and zirconium.

Figure 2 shows SER and microhardness dependences on the temperature of a 30-minute annealing of 4%Mg cast alloys. The analysis of the results demonstrates that substituting scandium with zirconium leads to an increase in thermal stability of the solid solution of scandium and zirconium in aluminum. The onset temperature of solid solution breakdown in alloys is ~200 °C. The EDS microanalysis results suggest that most of the formed particles contain both scandium and zirconium.

Analysis of Hv(T) dependences suggests that the greatest hardening effect during annealing of cast alloys is observed for the alloys with an increased content of scandium (0.18-0.22%Sc). It should be noted that annealing at 475-500 °C causes SER to increase in 2.5Mg and 4Mg cast alloys. This is due to the dissolution of previously released Al3Sc particles. This result indirectly indicates that during the breakdown of the solid solution, Al3Sc and Al3Zr particles were released separately.

The result was then confirmed while researching SER dependences on the duration of cast alloys isothermal annealing. Mechanisms of solid solution decomposition during annealing of aluminum alloys were identified from the analysis of researching specific electro resistance using the Johnson-Mehl-Avrami-Kolmogorov (JMAK) model [13]. It was demonstrated that breakdown of solid solution in cast alloys is two-staged, which is caused by a "separate" release of Al3Sc particles at lower temperatures at the cores of lattice dislocations, and then, at higher temperatures, a release of Al3Zr particles at the boundaries of dendritic crystals (grain boundaries).

Studies of MC aluminum alloys demonstrate that there is a post-ECAP increase in microhardness of aluminum alloys by ~200 MPa while SER remains practically the same.

Studies of the microstructure of annealed microcrystalline Al-2.5%Mg-Sc-Zr alloys demonstrate that the recrystallization onset temperature is 375-400 °C. It was found that SER dependences on the annealing temperature for all MC alloys are two-staged – a slight reduction of SER during annealing down to 275 °C and a rapid reduction of SER at higher temperatures occur (Figure 3). It should be noted that the onset temperature of solid solution breakdown in Al-2.5%Mg-Sc-Zr cast and MC alloys is 250-275 °C and is quite weakly dependent on the scandium-to-zirconium ratio (see Figure 3b). It should also be noted that, despite high microhardness values of post-ECAP MC alloys, the microhardness of Al-2.5%Mg-Sc-Zr MC alloys after annealing at 500 °C is lower than that of similarly-composed cast alloys after equivalent annealing. This result suggests that the second-phase particles are predominantly released at the MC alloy grain boundaries during annealing. This leads to faster growth of the released...
particles and, as per the Orowan equation, to a decrease in their contribution to the aluminum alloy hardness.

**Figure 3.** Dependence of microhardness (a) and SER (b) on the temperature of a 30-minute annealing of Al-2.5%Mg-Sc-Zr MC alloys with different contents of scandium and zirconium.

### 4. Conclusions

The specifics of solid solution breakdown in Al-2.5%Mg-Sc-Zr and Al-4%Mg-Sc-Zr cast and microcrystalline aluminum alloys have been studied. Studies have shown that partial substitution of Sc with Zr leads to a monotonic increase in thermal stability of the Sc and Zr solid solution in aluminum.

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