An investigation of thermal stability of structure and mechanical properties of Al-0.5Mg-Sc submicrocrystalline aluminum alloys

I S Shadrina¹, A V Nokhrin¹, V N Chuvil’deev¹, V I Kopylov¹,², A A Bobrov¹

¹ Lobachevsky State University of Nizhni Novgorod, 23 Garagina ave., Nizhny Novgorod, 603950, Russian Federation
² Physics and Technology Institute, National Academy of Sciences of Belarus, 10 Kuprevich st., Minsk, 220141, Belarus

Abstract. This paper presents the results of studying the thermal stability of the grain structure, mechanical properties, and electrical resistivity of the Al-0.5wt.%Mg-xSc submicrocrystalline (SMC) alloys with varying Sc content (x = 0, 0.1, 0.2, 0.3, 0.4, 0.5 wt.%). The SMC alloys were produced by equal-channel angular pressing (ECAP). The alloys with Sc concentration above 0.3 wt.% contained submicron Al₃Sc primary particles, the amount of which increased with increasing Sc concentration. High thermal stability of the SMC structure in the alloys was ensured by ECAP at 225 °C whereas the recrystallization onset temperature was 375 °C. The disintegration kinetics of the Al₃Sc particles in the SMC alloys was determined by the diffusion intensity along the dislocation cores in the lattice as well as at the grain boundaries.

1. Introduction

The Al–Sc alloys have been in the focus of researchers [1-5] and served as a base for a number of industrial Al alloys. Exceptional combination of high strength, weldability, and corrosion resistance make the Al–Sc alloys especially promising for applications in aircraft engineering, shipbuilding, petrochemistry, automotive and railroad industries, as well as in other Al-intensive sectors of general-purpose and special mechanical engineering. Sc is one of the most efficient doping elements for the Al alloys. The addition of a small amount of Sc (0.2–0.3 wt.%) provides 100-150 MPa higher strength in the deformed Al alloys, the maximum reduction of the cast grain structure (through to formation of a non-dendrite structure), markedly better weldability, and higher corrosion resistance. The nanoparticles of Al₃Sc intermetallic compound nucleating during annealing make the recrystallization slower significantly [6-8] that allows more than twofold increasing both the yield stress and the ultimate tensile strength. Moreover, adding Sc into the Al-Mg alloys ensures the formation of a fine-grained alloy structure and results in the alloy exhibiting superplastic behavior [9-11].

Further improvement of the mechanical properties of the Al-Mg-Sc alloys may be achieved through the formation of uniform submicrocrystalline (SMC) structure by means of severe plastic deformation (SPD). The formation of the SMC structure in the Al-Mg-Sc alloys ensures higher strength with retaining an adequate plasticity [12, 13], high-rate superplasticity [14-16], and higher fatigue strength [17, 18]. Note that, despite the reported successes in improving the thermal stability of the SMC structure in the Al alloys by means of doping with Sc, the authors have not been able to achieve the stability of the SMC structure and mechanical properties in the Al alloys at the temperatures, which would match...
the needs of industrial processing (above 300–350 °C) and correspond to the temperatures of superplastic deformation. This imparts a special relevance to studying the disintegration of the solid solution of Sc in the SMC Al-Mg-Sc alloys.

This paper aims at studying the thermal stability of the structure and mechanical properties of the SMC Al-0.5Mg-Sc alloys, which are promising for the electrical machinery applications such as high-strength conductors and studying the specific features of the disintegration of the Sc solid solution in the SMC alloys with low Mg content.

2. Materials and methods
The study was carried out on the Al-0.5 wt.% Mg alloys with varying Sc content (0.2, 0.3, 0.4, and 0.5 wt.%). Al-0.5 wt.% Mg alloy was taken as a reference. The Al alloys of the preset composition were produced by induction casting in an INDUTHERM casting machine. The SMC structure in the blanks was formed by means of equal-channel angular pressing (ECAP) carried out in Ficep HF400L press under the following process conditions: the ECAP temperature $T_{ecap} = 225$ °C, the deformation rate 0.4 mm/s, the number of ECAP cycles $N = 4$, and the deformation pattern Bc. The maximum time in the ECAP process channel was less than 8 min. Annealing was performed in forced-air furnace.

The axial homogeneity of the doping elements in the ingots was monitored through the specific resistivity $\rho$ was measured with SIGMATEST 2.069 eddy-current measuring instrument.

The structure of the alloys was studied with Leica DM IRM interference metallurgical microscope and Jeol JSM-6490 SEM with Oxford Instruments INCA 350 EDS spectrometer. The X-ray diffraction (XRD) investigations were carried out using DRON-3 diffractometer. The magnitudes of the internal stresses $\sigma_{int}$ were calculated using Williamson-Hall method. The microhardness $H_v$ was measured with HVS1000 tester.

3. Experimental results

3.1. Structure investigations
The cast Al-0.5Mg-Sc alloys in the initial state had a uniform coarse-grained structure. The average grain sizes decreased from 1000–1200 to 30-40 μm as Sc concentration increased from 0.2 to 0.5%. The structure of the alloys with 0.3-0.5 wt.% of Sc contained separate large light-colored particles with the average sizes of 0.35 – 1.3 μm. No primary Al₃Sc particles were found in the alloys with the Sc content less than 0.3 wt.%. A uniform SMC structure was formed after ECAP; the average grain size decreased slightly from 0.8–1 μm down to 0.5–0.5 μm with increasing Sc content from 0 to 0.5%. The internal stress $\sigma_{int}$ in the SMC alloys was 100–120 MPa.

Figure 1 shows the dependencies of the volume fraction of the recrystallized material and of the average grain size on the temperature of the 30-min annealing of the SMC alloys. The recrystallization (grain boundary migration) in the Al-0.5Mg alloy begins at $T_1 = 225$ °C: after heating up to 225 °C, the average volume fraction of the recrystallized material $f_R$ in the SMC Al-0.5Mg alloy was ≈56%, and the average size of the recrystallized grains $d_R$ was ≈3.1 μm. After heating up to 275 °C, the recrystallization occurs in the entire specimen volume ($f_R = 100\%$, $d_R = 12.6 \mu m$). With further heating of the SMC Al-0.5Mg alloy up to $T = 400$ °C, the grain sizes increased rapidly up to $d_R = 190.4 \mu m$.

Doping of the SMC Al-0.5Mg alloy with Sc resulted in a higher thermal stability: the recrystallization onset temperature in the Sc-doped SMC alloys was 375 °C. The average grain sizes and the volume fraction of the recrystallized material decreased with increasing Sc content: after annealing at 375 °C for 30 min, the average size of the recrystallized grains in the alloys with 0.2, 0.3, 0.4, and 0.5% of Sc were 4.5, 4.0, 3.4, and 3.1 μm, respectively. After 30-min annealing at 500 °C, the values of $d_R$ were 253, 8.5, 7.1, and 5.0 μm, respectively. Note that the alloys with increased Sc content (0.3–0.5%) contained the inclusions of the non-crystallized material even after heating up to 500 °C: the value of $f_R$ decreases slightly from 76 to 63% with increasing Sc content from 0.3 to 0.5%. The internal stress in the SMC alloys after annealing at 500 °C was $\sigma_{int} = 30–50$ MPa. This is an indirect evidence that the structure contains non-recrystallized inclusions.
3.2. Investigation of specific electrical resistivity

The specific resistivity of the cast Al-0.5Mg alloy increased gradually from 2.99 to 3.74 $\mu\Omega\cdot$cm with increasing Sc content from 0 to 0.4 wt.%. As the Sc concentration increased up to 0.5%, the value of $\rho$ decreased again down to 3.70 $\mu\Omega\cdot$cm. The non-monotonic dependence of $\rho$ on the Sc content suggests that when the Sc concentration exceeds 0.4 wt.%, some fraction of Sc nucleated into the primary particles during casting that agrees with the results of the structural investigations. The dependence of $\rho$ on the Sc content for the SMC alloy was similar; the resistivity of the SMC alloys was 0.01–0.04 $\mu\Omega\cdot$cm higher than the one of the cast alloys that corresponds to the impact of the electron scattering on the defects on the resistivity in the metals. This observation allows concluding that there was no disintegration of the Sc solid solution in Al during ECAP.

Figure 2 shows the dependencies of the specific resistivity on the temperature during annealing of the coarse-grained and SMC alloys. The figures demonstrate the dependencies $\rho(T)$ for the coarse-grained and SMC alloys to have three stages: no change in resistivity at lower temperatures ($T \leq T_1$), a rapid drop of the resistivity in the temperature range $T_1 < T < T_2$, and slight decrease in resistivity as the temperature increases further ($T \geq T_2$). As Sc content increased from 0.2 to 0.4-0.5%, $T_1$ for the cast alloys decreased from 275 to 225 °C, whereas $T_2$ remained unchanged ($\approx 400$ °C). Note that the resistivity of the cast Al-0.5Mg-Sc alloys heated up to $T_2$ decreased down to 3.03–3.06 $\mu\Omega\cdot$cm regardless to the Sc concentration that is close to the resistivity of the cast Al-0.5Mg alloy ($\rho = 2.98–2.99 \mu\Omega\cdot$cm). This leads to the conclusion that the disintegration of the solid solution (the nucleation of the second phase particles) was completed in the alloys after heating up to $T_2$.

No changes of the specific resistivity were observed during annealing of the coarse-grained Al-0.5Mg alloy. Annealing of the SMC alloy resulted in the decreasing of the resistivity from 3.01 down to 2.94–2.95 $\mu\Omega\cdot$cm. The observed decrease of the resistivity $\Delta \rho = 0.06–0.07 \mu\Omega\cdot$cm corresponds to usual decrease of the resistivity during the deformation and annealing of the deformed metals and indicates the observed decrease of the resistivity to be caused by the reinstatement and recrystallization during the annealing of the Al-0.5Mg alloy.
Figure 2. Dependencies of resistivity on temperature during 30-min annealing of coarse-grained (a) and SMC (b) Al-0.5Mg-Sc alloys with varying Sc content

Analyzing the ρ(T) dependencies for the SMC alloys (Figure 2b), one can see the disintegration temperature offset for the Al-Sc solid solution in the SMC alloys with 0.3-0.5% Sc to fall within the experimental uncertainty and to be close to T1 in the cast alloys, whereas the temperature of completing the solid solution disintegration T2 in the SMC alloys was 325–350 °C that was 50–75 °C lower than the values of T2 in the coarse-grained alloys. The resistivity ρ of the SMC alloys after annealing at 450 °C (30 min) was 3.09–3.12 μΩ·cm that was close to the ρ of the annealed Al-0.5Mg alloy.

Figure 3 show the dependencies of the specific resistivity on the isothermal annealing time at different temperatures for the coarse-grained and SMC Al-Mg-Sc alloys with varying Sc content. As the figures show, increasing annealing temperature resulted in increasing solid solution disintegration rate defined as $\partial \Delta \rho / \partial t$, whereas the resistivity decreasing rate in the SMC alloys was higher than that in the cast alloys (these results will be analyzed below).

3.3. Investigations of the mechanical properties

Analyzing the dependencies $H_v(\text{Sc})$, one can see that increasing the Sc content from 0 up to 0.5% in the cast alloys resulted in the increasing of $H_v$ from 300 to 380 MPa, whereas in the SMC alloys – from 500 up to 650 MPa.

Figure 3a shows the dependencies of the $H_v(T)$ in the coarse-grained and SMC alloys. The temperature dependencies $H_v(T)$ in the cast Al-0.5Mg-Sc alloys exhibited three usual stages originating from the nucleation of the second phase particles during annealing. Heating up within the temperature range $T < T_{(1)}$ didn’t affect the microhardness; annealing within $T_{(1)} \leq T \leq T_{(2)}$ increased $H_v$ rapidly, further heating at $T > T_{(2)}$ resulted in the strength degradation. The temperature $T_{(1)}$, which corresponds to the strengthening onset in the cast alloys, was in close agreement with the onset temperature of the specific resistivity decline $T_1$. The annealing temperature $T_{(2)}$, which corresponds to the maximum in the dependencies $H_v(T)$, was 325–350 °C and was 25–50 °C lower than $T_2$, which corresponds to completing of the rapid drop of the specific resistivity. We believe this is an indication that the loss of coherence and rapid growth of the Al3Sc particles in the cast Al-0.5Mg-Sc alloys begin already within the stage of solid solution disintegration, although at the end of this stage. Note that the scale of microhardness increased during the second stage of annealing $\Delta H_v$ increased from 410 MPa up to 555 MPa as the Sc concentration increased from 0.2 wt.% to 0.4 wt.% (see Fig. 3a). The maximum microhardness $H_{v(\text{max})} = 950$ MPa was found in the cast Al-0.5-0.4Sc alloy after annealing at 350 °C for 30 min.
The dependencies $H_v(T)$ for the SMC alloys were more complex (see Figure 3b). The strength of SMC Al-0.5Mg alloy began to decrease gradually at 125 $^\circ$C. A rapid degradation of $H_v$ took place at 275 $^\circ$C. During this stage, the hardness of the SMC Al-0.5Mg alloy decreased from 500 MPa down to 270 MPa and then decreases further slightly down to 230–235 MPa when heated up to 400 $^\circ$C.

The dependence $H_v(T)$ in the SMC alloys was qualitatively similar to the $H_v(T)$ dependences in the cast alloys. Note also that the maximum hardness values in the SMC alloys were achieved after annealing at lower temperatures (300 $^\circ$C, 30 min) as compared to the coarse-grained cast alloys. The scale of strengthening during the second stage of annealing $\Delta H_v$ was much smaller in the SMC alloys than in cast alloys: $\Delta H_v$ in the SMC alloys with 0.3-0.5% of Sc was 200–210 MPa whereas the scale of microhardness increase in the SMC Al-0.5Mg-0.2Sc alloy was quite smaller ($\Delta H_v = 15-20$ MPa, barely above the measurement uncertainty). Annealing at higher temperatures (above 300 $^\circ$C for 30 min) resulted in the alloy strength degradation, whereas the microhardness of the SMC alloys after annealing at 400 $^\circ$C for 30 min (460 MPa, 600 MPa, 645 MPa, and 700 MPa in the SMC alloys with 0.2, 0.3, 0.4, and 0.5 wt.% of Sc, respectively) was lower than the microhardness of the cast alloys (630 MPa, 760 MPa, and 780 MPa in the cast alloys with 0.2, 0.3, and 0.4-0.5 wt.% of Sc, respectively). We believe this result to indicate that the second-phase particles in the SMC alloys grow faster during annealing.

4. Discussion

In this section, the analysis of the specific features of the disintegration of Sc solid solution during annealing of the SMC Al-0.5Mg-Sc alloys are presented. First, we have calculated the theoretical values of resistivity in the investigated alloys and compared these values to the experimental observations. The theoretical resistivity $\rho_{th}$ was calculated on the base of the additivity rule:

$$\rho_{th} = \rho_{Al} + C_{Mg} \cdot K_{Mg} + C_{Sc} \cdot K_{Sc},$$

(1)

where $\rho_{Al} = 2.80 \ \mu\Omega\cdot$cm is the specific resistivity of pure Al, $C_{Mg}$ and $C_{Sc}$ are the Mg and Sc concentrations (at.%) in the alloy, respectively, $K_{Mg} = 0.49 \ \mu\Omega\cdot$cm/at.% and $K_{Sc} = 3.32 \ \mu\Omega\cdot$m/at.% are the contributions of Mg and Sc into the specific resistivity of the Al alloy, respectively [19]. One can see the increasing Sc content above 0.3% to result in the difference between the theoretical $\rho_{th}$ and experimental $\rho_{exp}$ resistivity $\Delta\rho_0 = \rho_{th} - \rho_{exp}$ exceeding the experimental uncertainty. Then, $\Delta\rho_0$ increased monotonically with rising Sc content: in the alloys with 0.3, 0.4, and 0.5%Sc, the values of $\Delta\rho_0$ were 0.08, 0.13, and 0.36 $\mu\Omega\cdot$cm, respectively. The difference between the theoretical and experimental resistivity values was attributed to the nucleation of the primary Al$_3$Sc intermetallics.

![Figure 3. Dependencies of resistivity on temperature during 30-min annealing of coarse-grained (a) and SMC (b) Al-0.5Mg-Sc alloys with varying Sc content](image-url)
The approach proposed in [20] can be applied to calculate the volume fraction of the second-phase particles. According to [20], the relation between the volume fraction of the second-phase particles \( f_v \) and the concentration of the doping elements \( C_r \) (at.%) (which the particles consist of) can be written as:

\[
 f_v = \frac{C_r(t)}{\alpha}, \quad (2)
\]

where \( \alpha \) describes the fraction of the doping elements in the particle (\( \alpha = 1/4 \) in the case of Al\(_3\)Sc).

Assuming the decrease in resistivity observed experimentally to be caused by lower concentration of the Sc atoms in the Al-Sc alloy \( \Delta \rho(t) = K_\text{Sc} C_\text{Sc}(t) \), equation (1) can be used to determine the maximum volume fraction of the Al\(_3\)Sc particles \( f_v(\text{max}) \). In the alloys with 0.2, 0.3, 0.4, and 0.5 wt.% of Sc, the values of \( f_v(\text{max}) \) were 0.12, 0.18, 0.24, and 0.30%, respectively. The alloys with 0.3, 0.4, and 0.5% of Sc manifested the following nucleation of primary Al\(_3\)Sc particles: 0.02%, 0.04%, and 0.11%, respectively. The presence of the primary particles in the Al-0.5Mg-Sc alloys was an apparent origin of smaller grain sizes in the cast alloys with increased Sc concentrations and of smaller strengthening in the coarse-grained Al-0.5Mg-0.5Sc alloy as compared to the cast alloy with 0.4%Sc.

Using equation (2) for the dependence of the specific resistivity change on the isothermal annealing time \( \Delta \rho(t, T = \text{const}) \), one can reformulate the dependence of the volume fraction of the second-phase particles on the annealing time \( f_v(t, T = \text{const}) \). According to Johnson – Mehl – Avrami – Kolmogorov equation \( f_v(t, T) = f_{v0}[1-\exp(-t/\tau^n)] \) [20], one can determine the activation energies \( Q \) and the numerical coefficients \( n \) to describe the disintegration mechanics [20]. These parameters can be determined from the experimental time dependencies of the specific resistivity plotted in the double logarithmic axes \( \ln[\ln(1-f_v(t)/f_{v(\text{max})})] - \ln t \). In the case of constant \( n \), these dependencies should be the linear ones, the slope of which gives the value of \( n \); and the offsets equal \( n \ln \tau \). The activation energy can then be determined from the slope of the dependence \( \ln \tau - \frac{T_m}{T} \).

Analyzing the dependencies in Figure 4, one can see that during the intensive change of resistivity the dependencies \( \rho(t, T = \text{const}) \) in the \( \ln[\ln(1-f_v(t)/f_{v(\text{max})})] - \ln t \) axes can be approximated with high accuracy by two straight lines with larger \( n_1 \) and smaller \( n_2 \) values of the disintegration intensity coefficient. The value of \( n \) obtained for the shorter annealing time was close to 0.3–0.5, and approached \( n_2 \approx 1 \) at medium annealing time. Further annealing resulted in the decrease of \( n \) back to \( \approx 0.3 \). Note also that the value of the effective coefficient \( n_{\text{eff}} \) depended on the annealing temperature is in a non-monotonously: Figure 4 shows that at lower annealing temperatures (200–225 °C) the SMC alloys exhibited the values of \( n_{\text{eff}} \approx n_1 \) at medium annealing temperatures (250–275 °C) \( n_{\text{eff}} \approx n_2 \), and at higher temperatures (above 300 °C) the effective value \( n_{\text{eff}} \) decreased again. (The effective (averaged) value of the disintegration intensity coefficient \( n_{\text{eff}} \) was determined by the slope of the whole dependence \( \ln[\ln(1-f_v(t)/f_{v(\text{max})})] - \ln t \) (not distinguishing the lower and higher values of \( n \)). The offset of such a linear dependence corresponds to the effective value \( n_{\text{eff}} \ln \tau_{\text{eff}} \).)

The dependence of \( n \) on the annealing temperature and the non-monotonous variation of its slope with increasing temperature and/or annealing time may be an indication of simultaneous action of different disintegration mechanisms, at least two. Assume that the volume fraction of a phase nucleated during the time \( t \) can be represented as a sum of two terms:
\[ f_1(t, T, n_1, Q_{\text{eff}}) = f_{v1}(t, T, n_1, Q), \]
\[ f_2(t, T, n_2, Q_{\text{eff}}) = f_{v2}(t, T, n_2, Q), \]
where \( f_{v1} \) is the volume fraction of the phase, the nucleation of which is regulated by the first mechanism described by its own set of parameters \((n_1, Q)\) and \( f_{v2} \) is the second mechanism with its own set of parameters \((n_2, Q)\). Depending on the annealing temperature and time, the ratios between the terms in (3) and, consequently, the effective values of the parameters \((n_{\text{eff}}, Q_{\text{eff}})\) can change.

For the purposes of further analysis, it is important to note that the effective activation energy of the solid solution disintegration \( Q_{\text{eff}} \) increased from 8.0 \( kT_m \) to 12.1–12.2 \( kT_m \) as the Sc concentration in the SMC Al-0.5Mg alloy increased from 0.2 to 0.5 wt.%. Note that dependences \( n_{\text{eff}} \approx T_{\text{eff}}/T \) for all SMC alloys can be approximated with a high degree of accuracy by a single straight line (without distinguishing between the lower value and the higher one). We believe this to mean that the activation energies for the processes with lower and higher \( n \) values are close.

According to the approach proposed in [20], low values of \( n_1 = 0.3–0.5 \) and \( Q_1 \approx 10 \( kT_m \) correspond to the nucleation of the particles at the lattice dislocation cores under the conditions of an unstable microstructure (during the development of the reinstatements and recrystallization), whereas high values of \( n_2 \approx 1 \) and activation energy \( Q_2 \approx 10 \( kT_m \) correspond to the nucleation of the \( \text{Al}_3\text{Sc} \) particles at the grain boundaries in the SMC alloys. The disintegration of the solid solution in the SMC Al-0.5Mg-Sc alloys can, therefore, be described as follows. At lower temperatures (below 225 °C \( \approx 0.53T_m \)), due to high density of the lattice dislocations in the SMC metals, the intensity of diffusion-based mass transfer via the dislocation cores is much higher than the one inside the grains or at the grain boundaries. Under these conditions, the disintegration kinetics is primarily determined by the dislocation core diffusion, i.e. \( f_1(t) \gg f_2(t); \) the volume fraction of the nucleated particles can be presented, in the first approximation, as: \( f_1(t) = f_{v1(\text{max})}[1 - \exp(-\frac{t}{\tau})] \). Note that sufficiently high annealing temperatures \((0.53–0.56)T_m \) induce intensive reinstatement processes in the SMC Al alloys, which result in lower densities of lattice dislocations \((\rho_s \neq \text{const}; \; d = \text{const})\). Here, the value of \( n_{\text{eff}} \) is close to \( n_1 \approx 0.25 \) in the first process.

One of possible reasons for the domination of the dislocation-type contribution at lower annealing temperatures may be an indirect impact of the micron-sized primary \( \text{Al}_3\text{Sc} \) particles. As has been demonstrated in [19, 21], the dislocation-related defects may occur in the disperse particles of the radius \( R \) distributed at the grain boundaries of the fine-grained materials. Each defect can be treated as a disclination loop of the radius \( R \) and power \( \alpha(t) \). The stress field in the vicinity of the boundaries, which contain the particles surrounded by the disclination loops, in the first approximation, can be estimated as:

\[ \sigma_1 = \alpha_1 G\omega(t)/\lambda, \]  

where \( \alpha_1 \) is a geometrical parameter. The formation of the disclination loops on larger (micron-sized) particles leads to additional strengthening of the SMC Al alloys, according to Orowan equation:

\[ \Delta\sigma(R) = \alpha_2 Gb\frac{r^{1/2}}{t} \]

where \( \alpha_2 \) is a numerical coefficient. This effect would also accelerate the nucleation of the secondary \( \text{Al}_3\text{Sc} \) nanoparticles in the vicinity of larger primary particles.

Lower dislocation density results in fewer places to accommodate the nucleation of the second-phase particles, lower value of \( f_{v1(\text{max})} \) (the volume fraction of these particles approaches its limit \( f_{\rightarrow} \rightarrow f_{v1(\text{max})} \)), and the disintegration controlled by the diffusion via the lattice dislocation cores almost ceases. The nucleation kinetics is controlled further by the diffusion via the grain boundaries with higher values of \( n_2 \approx 1 \) and \( Q_2 \approx 10 \( kT_m \). This factor leads to the increase of \( n_{\text{eff}}(T) \) with increasing temperature: the temperature increase from 200–225°C to 250–275°C resulted in the increase of \( n_{\text{eff}} \) from 0.3–0.5 to ~1. The nucleation of the \( \text{Al}_3\text{Sc} \) particles at the grain boundaries results in high thermal stability of the SMC structure in the Al-0.5Mg-Sc alloys produced by ECAP: the recrystallization onset temperature during the 30-min annealing was 375 °C that is markedly higher than the one in the SMC Al-Mg-Sc(Zr) alloys [6].

The origins of lower \( n_{\text{eff}} \) at higher annealing temperatures (300–325 °C) are less obvious. We believe the main reason for \( n_{\text{eff}} \) lowering to 0.3–0.5 to be the onset of the grain boundary migration...
The investigations of the thermal stability of the mechanical properties have shown the dependencies $H_v = H_v_0 + K_0 d^{-1/2}$. Further increase of the Sc concentration up to 0.5% resulted in the decrease of $K$ down to 0.53-0.60 MPa$^{-1/2}$. The dependence of $K$ on the Sc content is an indication of a considerable fraction of the Al$_3$Sc particles to be located at the grain boundaries in the SMC Al-0.5Mg-Sc alloys after ECAP that coincides indirectly with the conclusions on the nucleation of the second-phase particles drawn from the conductivity analysis as described above. The non-monotonous behavior of the dependence of $K$ on the Sc content and, in particular, the decrease of $K$ in the SMC alloys with 0.4 and 0.5% of Sc confirm the above conclusion that the volume fraction of the primary Al$_3$Sc particles increases in the alloys with high Sc concentrations with the associated decrease in the Sc concentration in the solid solution in the SMC Al-0.5Mg-Sc alloys.

5. Conclusions

1. The average grain size in the cast and SMC alloys decreases with increasing Sc concentration in the alloy. The average grain size in the SMC Al-0.5Mg-Sc alloys was 0.4–0.5 μm. The structure of the cast and SMC alloys contained the primary Al$_3$Sc particles of the submicron sizes (0.3–1.2 μm), the amount of which increased at higher Sc concentrations. The analysis of the specific electrical resistivity has shown that the volume fraction of the primary particles reached 0.04% and 0.11% in the alloys with 0.4 and 0.5 wt.% of Sc, respectively.

2. The SMC alloys were shown to have high mechanical properties: increasing Sc concentration in the SMC Al-0.5Mg-Sc alloys from 0 to 0.5% resulted in the increase of the hardness from 500 up to 650 MPa. The investigations of the thermal stability of the mechanical properties has shown the dependencies $H_v(T)$ in the cast and SMC alloys to exhibit a regular three-stage pattern. The maximum hardness $H_v(max)$ was 950 MPa and was observed in the cast Al-0.5-0.4Sc alloy after annealing at 350 °C for 30 min. The maximum hardness values in the SMC alloys were achieved after annealing at lower temperatures (300 °C, 30 min) as compared to the coarse-grained cast alloys, and the scale of strengthening during the second stage of annealing Δ$H_v$ was much smaller in the SMC alloys than in the cast ones. The annealing of the SMC alloys at higher temperatures resulted in a rapid strength degradation. The hardness of these annealed SMC alloys were lower than the ones of the cast alloys. We believe this result to indicate a fast growing of the particles Al$_3$Sc in the SMC alloys during annealing.

3. The activation energy for the Al$_3$Sc particle nucleation was found to be close to the one of diffusion via the lattice dislocation cores and grain boundaries, to depend on the Sc content, and to increase from 8.0 $kT_m$ up to 12.1–12.3 $kT_m$ with increasing the Sc concentration from 0.2 up to 0.5%. The value of disintegration intensity coefficient $n_{eff}$ in Johnson – Mehl – Avrami – Kolmogorov equation depended on the annealing temperature non-monotonously (with a maximum). The value of $n_{eff}$ It has been demonstrated to corresponded to the nucleation on the lattice dislocation cores at lower temperatures and to the nucleation of the Al$_3$Sc particles at the grain boundaries at higher temperatures.

4. The nucleation of the Al$_3$Sc particles at the grain boundaries of the SMC metals before the recrystallization (grain boundary migration) onset ensures a high thermal stability of the structure and mechanical properties of the SMC Al-0.5Mg-Sc alloys. The recrystallization onset temperature during
30-min annealing was 375 °C that is markedly higher than the published values for the thermal stability range for the SMC Al-Mg-Sc alloys.

5. The dependence of the microhardness on the grain sizes in the SMC alloys can be described by Hall–Petch relation. The Hall–Petch coefficient in the SMC alloys has been demonstrated to depend on the Sc content non-monotonic (with a maximum). The maximum values of the Hall–Petch coefficient were achieved in the SMC alloy with 0.3%Sc that corresponds to the maximum concentration of Sc in the Al-Sc solid solution after casting and ECAP and, consequently, to the maximum volume fraction of the nucleated Al3Sc particles.

Acknowledgements
The authors thank the Ministry of Science and Higher Education of the Russian Federation (Grant No. 11.1884.2017/4.6) for support.

References
[1] Zakharov V V 2003 Materials Science and Heat Treatment 45 246-253.
[2] Davydov V G, Rostova T D, Zakharov V V, Filatov Yu A, Yelagin V I 2000 Materials Science and Engineering A 280 30-36.
[3] Filatov Yu A, Yelagin V I, Zakharov V V 2000 Materials Science and Engineering A 280 97-101.
[4] Elagin V I, Zakharov V V, Rostova T D 1992 Materials Science and Heat Treatment 34 37-45.
[5] Zakharov V V, Rostova T D 2014 Materials Science and Heat Treatment 55 660-664.
[6] Chuvil’deev V N, Nokhrin A V, Makarov I M, Lopatin Yu G, Sakharov N V, Melekhin N V, Piskunov A V, Smirnova E S, Kopylov V I 2012 Russian Metallurgy (Metally) 2012 415-427.
[7] Nokhrin A, Shadrina I, Chuvil’deev V, Kopylov V 2019 Materials 12 316.
[8] Dobatkin S V, Zakharov V V, Vinogradov A Yu, Kitagawa K, Krasil’nikov N A, Rostova T D, Bastarash E N 2006 Russian Metallurgy (Metally) 2006 533-540.
[9] Mikhaylovskaya A V, Kotov A D, Kishchik M S, Prosviryakov A S, Portnoy V K 2019 Metals 9 33.
[10] Mikhaylovskaya A V, Yakovtseva O A, Cheverikin V V, Kotov A D, Portnoy V K 2016 Materials Science and Engineering A 659 225-233.
[11] Nieh T G, Hsiung L M, Wadsworth J, Kaibyshev R 1998 Acta Materialia 46 2789-2800.
[12] Malopheyev S, Kulitskiy V, Kaibyshev R 2017 Journal of Alloys and Compounds 698 957-966.
[13] Mogucheva A A, Dubina A V, Kaibyshev R O 2012 Reviews on Advanced Materials Science 31 54-61.
[14] Perevezentsev V N, Chuvil’deev V N, Kopylov V I, Sysoev A N, Langdon T G 2002 Annales de Chimie: Science des Matériaux 27 36-44.
[15] Furukawa M, Utsunomiya A, Matsubara K, Horita Z, Langdon T G 2001 Acta Materialia 49 3829-3838.
[16] Lee S, Utsunomiya A, Akamatsu H, Neishi K, Furukawa M, Horita Z, Langdon T G 2002 Acta Materialia 50 553-564.
[17] Vinogradov A, Washikita A, Kitagawa K, Kopylov V I 2003 Materials Science and Engineering A 349 318-326.
[18] Zhemchuzhnikova D, Mironov S, Kaibyshev R Metallurgical and Materials Transactions A 48 150-158.
[19] Chuvil’deev V N, Nokhrin A V, Smirnova E S, Kopylov V I 2012 Russian Metallurgy (Metally) 2012 985-993.
[20] Chuvil’deev V N, Smirnova E S, Kopylov V I 2012 Russian Metallurgy (Metally) 2012 612-624.
[21] Chuvil’deev V N, Nokhrin A V, Smirnova E S, Kopylov V I 2013 Russian Metallurgy (Metally) 2013 676-690.