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Investigation of the structure and properties of eutectic alloys of the Al – Ca – Ni system containing REM

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This study investigated the eutectic alloys based on aluminum containing small amount of rare earth metals (REM): Al – 6Ca – 3Ni – 2Ce; Al – 6Ca – 3Ni – 2La; Al – 6Ca – 3Ni – 2Pr. The compositions of the alloys were selected on the basis of previous studies of ternary Al – Ca – Ni and Al – Ni – Ce systems, taking into account the similarity of the structure of the Al – REM binary systems. Melting was carried out in an induction furnace by RELTEC. Alloys were prepared on the basis of aluminum A99. Annealing of the samples at 550 °C for three hours was carried out in SNOL 8.2/1100 and SNOL 58/350 muffle electric furnaces. Calculation of Al – Ca – Ni – Ce systems at 6% Ca by means of Thermo-Calc (databases TTAL5, TCAL4), showed that primary crystals of the Al_3Ni phase should be formed in the alloys of the selected compositions, however these crystals were not present. Using optical and scanning electron microscopy, the structure of alloys in the as-cast and heat-treated states was studied. It is established that in the process of non-equilibrium crystallization, the boundary of the phase region of existence of the aluminum solid solution significantly expands. Using micro-*X*-ray spectral analysis (MRSA), it was determined that during the equilibrium crystallization conditions in the Al – Ca – Ni – Ce system, rather than the binary Al_3Ni the ternary Al_9Ni_2Ca phase is formed. The possibility of applying hot rolling to Al – Ca – Ni based alloys additionally alloyed with Ce, La and Pr has been established, and the mechanical properties of hot-rolled samples have been obtained. Hot rolling was carried out at 500 °C. Rolling was carried out in five passes, the total degree of deformation in all cases was about 70%. Samples of the Al – 6Ca – 3Ni – 2Ce alloy were additionally rolled at a temperature of 550 °C. On the basis of a comparison of the mechanical properties and the microstructure of rolled products, it is assumed that the best mechanical properties are possessed by the samples of those alloys in which intermetallics have the smallest dimensions and are most evenly distributed in an aluminum solid solution. In particular, this demonstrates the Al – 6Ca – 3Ni – 2La alloy rolled at 500 °C and the Al – 6Ca – 3Ni – 2Ce alloy rolled at 550 °C.

Key words: eutectic alloys, rare earth metals, ternary Al_9Ni_2Ca phase, intermetallics, microstructure, hot rolling, mechanical properties

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Introduction

In recent decades, there has been a steady increase in demand for the application of aluminum alloys [1–2]; hence, development of alloys having improved set of mechanical and technological properties is an urgent task.

It is known that alloys with greater fraction of eutectic phases in the structure have good casting properties [3]. According to [4–6], the mechanical properties of the alloys tended to improve as the fraction of eutectic structure increases. Eutectic alloys on the basis of the Al – Si system (silumines) are traditionally used in industry. However, current market demands more durable and heat-resistant materials. Since the 90-s of the last century, studies on the other eutectic systems, such as Al – Ni [7], Al – Ce [8], Al – Ni – Fe [9], Al – Ce – Cu [10] and so on, have been undertaken. It has been revealed that these eutectic alloys possess finer structure than the silumines, thus providing enhanced technological and mechanical properties. Recent study [8] investigated the alloys of the Al – Ce – Ni system having ternary eutectic (Al) + Al_4Ce + Al_3Ni structure. Comparison of low- and high-temperature mechanical properties, as well as foundry characteristics, showed that the experimental Al – 12% Ce – 5% Ni – 0.5% Zr alloy perform better than the known high-temperature Al5Cu (AL 19), Al12SiCuMgNi (AL30) and Al5C cast alloys.

Foreign researchers started publishing research works on aluminum alloys modified with rare earth metals (REM) more and more often [11–12]. The best combination of strength and heat resistance has been demonstrated for the alloys modified with cerium and nickel; however, as these alloying elements are costly, their content in industrial alloys should be limited. On the other hand, the structure of alloys with high content of eutectic intermetallics is similar to that of composite materials, which has a combination of high mechanical and other performance properties [13]; however, since these alloys are produced using traditional foundry technologies, they are considerably cheaper than composites. In recent works on the study of alloys of the type “natural composites” we drew attention to calcium, which also forms a eutectic system with aluminum [14–16]. Alloys with calcium have not only good casting properties, high corrosion resistance, but also proved to be very technological in the process of hot and cold rolling [17–23]. Therefore, in this study, the following tasks were set:

– To study the structure of the selected alloys in the cast and heat-treated conditions;

– Investigate the possibility of applying hot rolling to Al – Ca – Ni-based alloys additionally doped with Ce, La and Pr;

– Investigate the structure and properties of the obtained deformed semi-finished products.

Methods and materials

The objects of the experimental study were quaternary alloys of the following compositions: Al – 6Ca – 3Ni – 2Ce; Al – 6Ca – 3Ni – 2La; Al – 6Ca – 3Ni – 2Pr.

Melting was carried out in an induction furnace by RELTEC. Alloys were prepared using aluminum A99 and Al – 20% Ni master alloys and pure cerium, lanthanum, praseodymium and calcium. Pre-calculated amounts of aluminum A99 and Al – 20% Ni master alloys were placed in the crucible and the temperature of the crucible was raised to about 730–740 °C. Upon complete melting, cerium, lanthanum and praseodymium were introduced into the melt. Then, to add metallic calcium, the melt temperature was raised to about 780 °C. After that, the molten metal was allowed to have complete dissolution of the materials charged into the melt. The melt was then held for 5–10 minutes at 740 °C to homogenize the alloy composition. At a temperature of 720–740 °C, slag was removed. After that, the metal was cast into a graphite mold at a temperature of 710–720 °C, which allowed obtaining flat castings with dimensions of 15×30×180 mm. These conditions for the production of experimental ingots correspond to the production of industrial ingots with a diameter of 100 mm, obtained by the method of twin rolling casting [2].

The samples were heat treated in SNOL 8.2/1100 and SNOL 58/350 muffle electric furnaces with a temperature accuracy of about 3 °C. The chromel-alumel thermocouples were used to measure the temperature. Annealing was performed at 550 °C for 3 hours.

The microstructure of the as-cast and heat-treated samples was examined using an Olympus GX51 (OM) optical microscope and a TESCAN VEGA 3 scanning electron microscope (SEM). The TESCAN microscope equipped with an energy dispersive attachment-microanalyzer manufactured by Oxford Instruments and AZtec Software, was also used for microenew and spectral analysis (MRSA). To prepare the sections, both mechanical and electrolytic polishing was used, which was carried out at a voltage of 12 volts electrolyte containing 75% C_2H_5OH, 12.5% $HClO_4$ and 12.5% glycerol.

Hot rolling was carried out on a laboratory mill 260. Type of mill Duo, reversible, maximum rolling width 250 mm, rolling speed 0.2 m/s.

The tensile test was carried out according to State Standart 1497–84 using the universal machine INSTRON at a loading rate of 10 mm/min.

Results and discussion

The composition of the alloys was defined and set on the basis of the studies undertaken earlier on the ternary Al – Ca – Ni [23] and Al – Ni – Ce [8] systems by considering the similarity of the structure of binary Al – REM systems [15–16]. All alloys have a pre-eutectic structure with a quite small fraction of the aluminum solid solution (hereinafter (Al)) and eutectic colonies of different degrees of dispersion (Fig. 1). Calculation of Al – Ca – Ni – Ce systems at 6% Ca by means of Thermo-Calc (databases TTAL5, TCAL4), showed that primary crystals of the Al_3Ni phase are expected to be formed during soli-

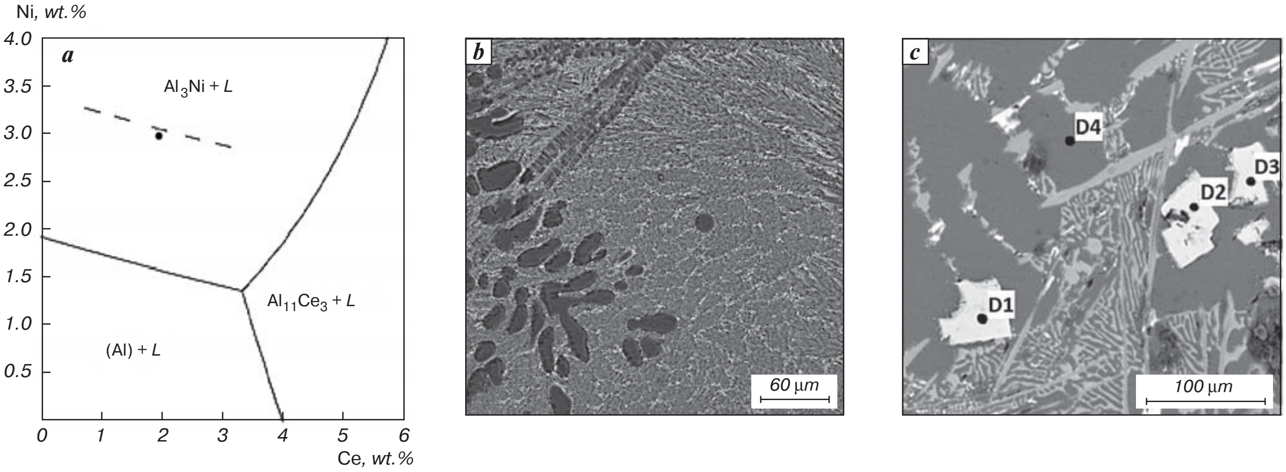

Fig. 1.

a — Projection of the liquidus surface of the Al – 6Ca – Ce – Ni system (the dashed line shows the displacement of the boundary of the phase regions at a high cooling rate); *b* — Structure of the Al – 6Ca – 2Ce – 3Ni alloy in the as-cast state (graphite, cooling rate 10 °C/s); *c* — Structure of the Al – 6Ca – 2Ce – 3Ni alloy in the as-cast state (cooling with the furnace, cooling rate 0.1 °C/s)

dification of the experimental alloys; however, microstructural investigations revealed no evidence of the formation of Al_3Ni phase in experimental alloys. This implies that the phase boundary under fast cooling conditions shifts, thus expanding the region of existence of (Al) (Fig. 2, *a*).

We assume that the phase diagrams of all the investigated systems have a similar form; therefore, in this paper we consider only the Al – Ca – Ni – Ce system, which is the most promising for the development of industrial alloys.

In a graphite mold, all alloys were solidified with the cooling rate of about 10 °C/s. Then, the Al – 6Ca – 3Ni – 2Ce alloy was slowly cooled in a graphite-chamotte crucible inside the oven (cooling rate of about 0.1 °C/s), which allows refining the boundary of the primary crystallization of (Al) and Al_3Ni phases. The structure obtained under slow cooling conditions is shown in Fig. 1, *c*. It consists of a fairly large number of light equiaxed crystals identified as Al_9Ni_2Ca by the MRSA method. A small amount of cerium is dissolved in this compound. It was confirmed that none of the eutectic-forming elements dissolves in an aluminum solid solution. Since the Thermo-Calc program does not take into account the existence of ternary eutectic phases, the liquidus surface in the region of the aluminum corner should be different than what is shown by calculation.

The summary table, at.%

Spectrum No.	Al	Ca	Ni	Ce	Total	Phase
D 1	74.44	7.80	16.73	1.05	100	Al_9Ni_2Ca
D 2	75.49	7.67	15.68	1.16	100	Al_9Ni_2Ca
D 3	74.98	7.81	16.10	1.12	100	Al_9Ni_2Ca
D 4	100	0	0	0	100	(Al)

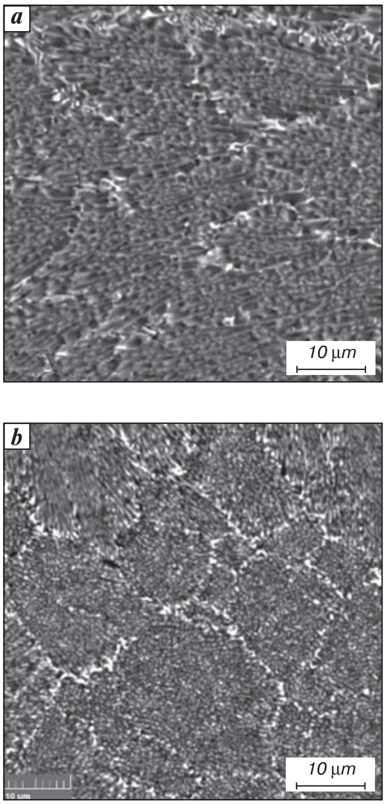

Fig. 2. Microstructure of alloys in the as-cast state, SEM, ×5000: *a* — Al – 6Ca – 3Ni – 2Ce alloy; *b* — Al – 6Ca – 3Ni – 2La alloy

Thus, under the cooling conditions observed in this study, alloys having very thin and almost completely eutectic structure with high proportion of intermetallic compounds (about 35 mass %) were obtained. Prior to hot rolling, all samples were annealed to increase the ductility: Al – 6Ca – 3Ni – 2Ce and Al – 6Ca – 3Ni – 2La at 500 °C for 3 hours, and Al – 6Ca – 3Ni – 2Pr at 500 °C and 600 °C for 3 hours. Plasticity is enhanced as annealing process caused the eutectic intermetallics to become more rounded (Fig. 3). The annealing regime of 500 °C, 3 hours was found to be optimal for eutectic alloys with REM [23].

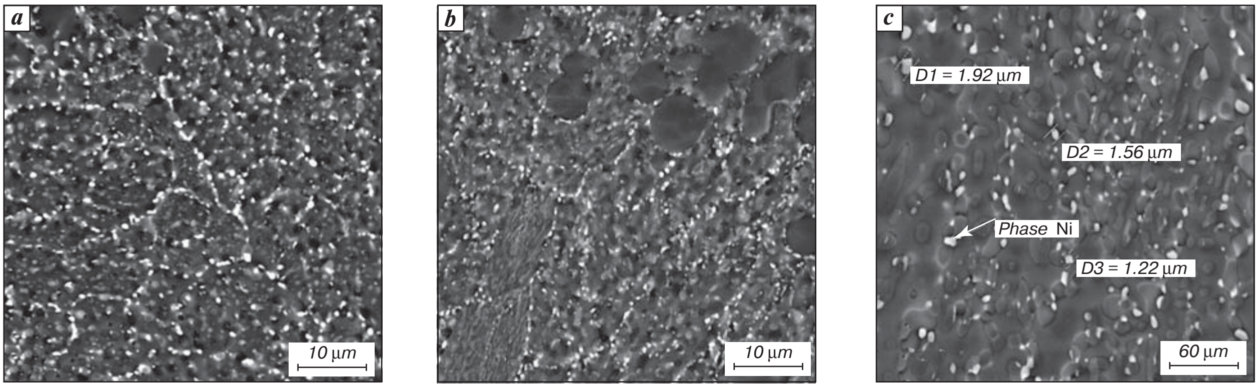

Fig. 3. Microstructure of alloys in heat-treated condition, SEM, ×5000:
a — Al – 6Ca – 3Ni – 2Ce alloy, annealing 550 °C, 3 h; *b* — Al – 6Ca – 3Ni – 2Pr alloy, annealing 500 °C, 3 h; *c* — Al – 6Ca – 3Ni – 2Pr alloy, annealing 600 °C, 3 h

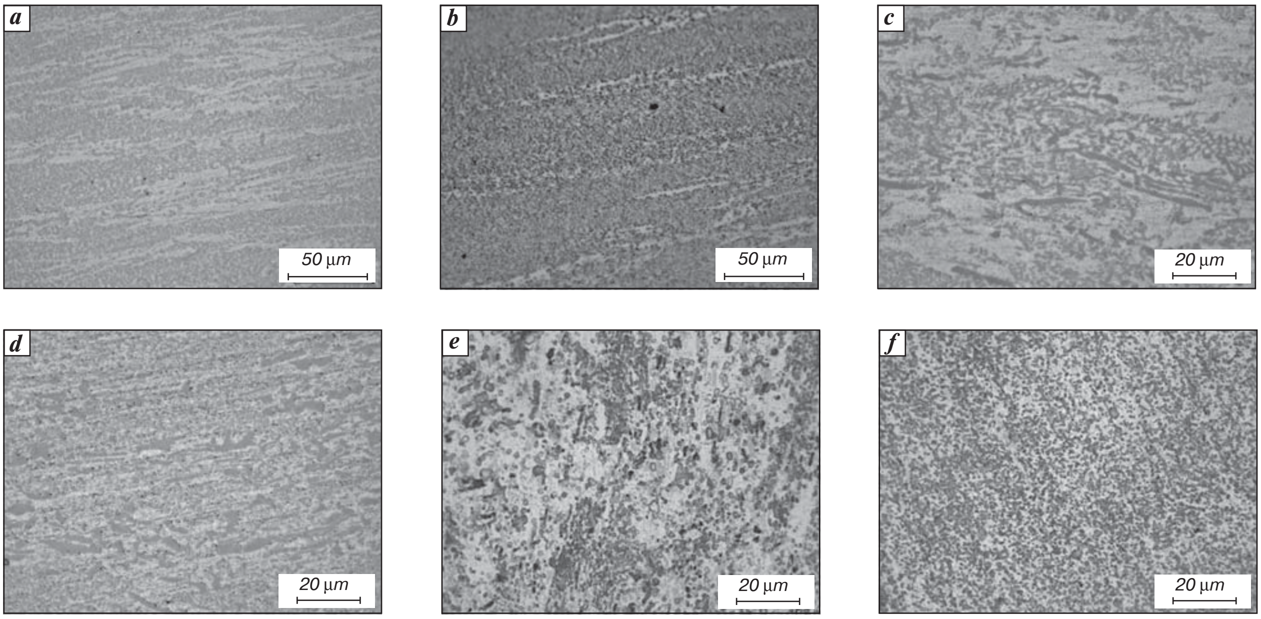

Fig. 4. Microstructure of hot rolled steel obtained at 500 °C:
a — Al – 6Ca – 3Ni – 2Ce longitudinal direction; *b* — Al – 6Ca – 3Ni – 2La longitudinal direction; *c* – Al – 6Ca – 3Ni – 2Ce transverse direction; *d* – Al – 6Ca – 3Ni – 2La transverse direction; *e* — Al – 6Ca – 3Ni – 2Ce in the plane of the sheet; *f* – Al – 6Ca – 3Ni – 2La in the plane of the sheet

For the alloy with the addition of praseodymium, two annealing regimes were used to evaluate its thermal stability. Al11Pr3 phase particles with increasing annealing temperature rise slightly (by 0.5–1.0 microns at 500 °C to 1.0–2.0 micron at 600 °C), which indicates the stability of alloy structure (Fig. 3, *b*, *c*).

Hot rolling was carried out at 500 °C. The initial thickness of the samples was 14 mm. Rolling was carried out in five passes, all samples were rolled up to 4.0–4.2 mm, the total degree of deformation in all cases was about 70%. Samples of the Al – 6Ca – 3Ni – 2Ce alloy were additionally rolled at a temperature of 550 °C. Strength properties of hot rolled products are reflected in Table 1.

The most durable and ductile were the sheets from Al – 6Ca – 3Ni – 2La alloy. A study of the microstructure of rolled products (Fig. 4) in different directions showed that the particles of the intermetallide $Al_{11}La_3$ (Al_4La) are smaller than the intermetallides of other alloys and are distributed more evenly in the aluminum solid solution.

Table 1
Mechanical properties of hot-rolled samples

Alloy	Rolling temperature, °C	Deformation level, %	σ, MPa	$\sigma_{0.2}$, MPa	δ, %
Al – 6Ca – 3Ni – 2La	500	71.4	303	203	5
Al – 6Ca – 3Ni – 2Ce	500	70.4	201	172	0.5
	550	86.4	258	216	2.9
Al – 6Ca – 3Ni – 2Pr	500	70	187	146	0.8

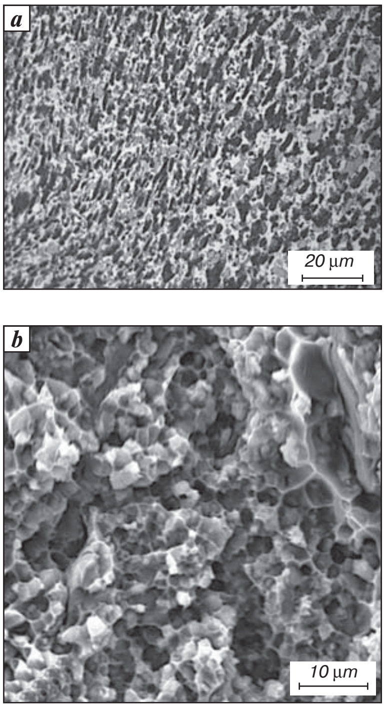

Fig. 5. The structure of the sample from the alloy Al – 6Ca – 3Ni – 2Ce, rolled at 550 °C:
a — Hot rolling; *b* — Fracture of a hot-rolled specimen

The strength properties of samples of the Al – 6Ca – 3Ni – 2Ce alloy rolled at 550 °C are much higher (Table 1), the intermetallide particles are finer and more evenly distributed in (Al) as compared to the sample rolled at 500 °C (Fig. 5, *a*) about 3%, fracture is viscous, pitting (Fig. 5, *b*).

Conclusions

1. It is established that in the process of non-equilibrium crystallization, the boundary of the phase region of existence of the aluminum solid solution expands. As a result, finely-dispersed pre-eutectic structure with a high volume fraction of eutectic intermetallics is formed in the experimental alloys instead of the hypereutectic structure.
2. It has been determined that rather than the binary Al_3Ni phase the ternary Al_9Ni_2Ca formed during equilibrium solidification of Al – Ca – Ni – Ce system, which indicates the necessity for additional experimental studies to refine the structure of the Al – Ca – Ni – Ce system in aluminum side.
3. It has been established that experimental alloys having the structure with more than 35 wt.% of intermetallic compounds can be subjected to hot rolling with deformation levels of 70 to 86%.
4. The best mechanical properties are the samples of those alloys in which the eutectic intermetallics have the smallest dimensions and are most evenly distributed in an aluminum solid solution.

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Powder technology for manufacturing compact blanks of Ti – Nb – Ta, Ti – Nb – Zr alloys

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Presented in the paper are the results of investigating the consolidation process (compacting, sintering, hot isostatic pressing – HIP) of calcium hydride powder of low modulus Ti – Nb alloys doped by tantalum: Ti – 30.1 wt.% Nb – 17.4 wt.% Ta (Ti – 22 at.% Nb – 6 at.% Ta), zirconium: Ti – 33.2 wt.% Nb – 8.6 wt.% Zr (Ti – 22 at.% Nb – 6 at.% Zr) and estimating their mechanical properties. It is shown that metal powders are notable for good compactability on both single-action compacting and isostatic forming. Cold isostatic forming under pressure of 200 MPa permits to obtain briquettes with relative density of 65–68%. Sintering the briquettes at a temperature of 1873 K provides blank formation with porosity of 16 and 8% for Ti – 30.1Nb – 17.4Ta, Ti – 33.2Nb – 8.6Zr (wt.%) alloys, respectively. Sintering in vacuum of 1.33 Pa leads to formation of a gas-filled layer with heightened microhardness to a depth of 8 mm. Sintering in vacuum of $1.33 \cdot 10^{-2}$ Pa allows to avoid this phenomenon. Hot isostatic pressing of the sintered blanks at a temperature of 1193 K and pressure of 150 MPa guarantees obtaining practically porousless material (1% of pores). It is determined that Ti – 30.1Nb – 17.4Ta, Ti – 33.2Nb – 8.6Zr (wt.%) are characterized after sintering by the following values of the yield stress and the Young’s modulus: $\sigma_{0.2} = 444 \pm 7$ MPa, $E = 57 \pm 5$ GPa and $\sigma_{0.2} = 570 \pm 29$ MPa, $E = 62 \pm 5$ GPa, respectively. After HIP: $\sigma_{0.2} = 791 \pm 16$ MPa, $E = 87 \pm 4$ GPa and $\sigma_{0.2} = 750 \pm 50$ MPa, $E = 81 \pm 1$ GPa, respectively.

Key words: titanium alloys, low modulus alloys, compacting, sintering, hot isostatic pressing, porosity, yield stress, the Young’s modulus.

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