Effect of Ca and Zn alloying on the structure and properties of Al — 2.5%Mg alloy

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In an effort to optimize the composition of high-strength Ca-containing aluminum alloys, the dependences of the hardness of Al – 2.5Mg – (0...10)Ca – (0...14)Zn (wt.%) compositions upon the aging temperature have been studied. The structure of alloys and the composition of individual microstructure constituents have been investigated by means of light and scanning electron microscopy as well as the energy dispersive *X*-ray microanalysis (EDXMA) method. Determined are the concentrations of Zn in primary crystals and in a eutectic for all experimental alloys. It was fixed that the composition of primary crystals in Al – 2.5Mg – 4Ca – 12Zn, Al – 2.5Mg – 6Ca – 12Zn and Al – 2.5Mg – 10Ca – 14Zn alloys accords with the Al_3ZnCa phase. The hardness and elastic modulus of $(Al,Zn)_4Ca$ primary crystals and $[(Al) + (Al, Zn)_4Ca]$ eutectic in Al – 2.5Mg – 10Ca – (1...14)Zn alloys have been determined by the instrumented nanoindentation method. It was found that these mechanical properties are rising as the concentration of Zn in primary crystals increases. According to the study fulfilled, the best combination of mechanical and technological properties has been achieved in Al – 2.5%Mg alloys with the content of Ca varied from 3 to 4% and Zn from 8 to 10%.

Key words: aluminum alloys, eutectic, primary crystals, quenching, ageing, hardening, dispersoids, nanoindentation, hardness, elastic modulus.

DOI: 10.17580/nfm.2019.01.04

Introduction

Aluminum-magnesium alloys are characterized by wide industrial usage owing to their low density, high corrosion stability and manufacturability [1–4]. Magnesium combined with zinc alloying is used to strengthen an aluminium solid solution in 7xxx alloys [5–10]. Calcium adding leads to the corrosion stability increase, density lowering and significant growth of castability of aluminum alloys [11–25]. Aluminum-calcium alloys with considerable quantity of $[(Al) + Al_4Ca]$ eutectic (where (Al) — solid solution based on aluminum) compare well with silumins by the aggregate of casting characteristics [23]. Therefore, the Al – Ca – Zn – Mg system has been studied by the authors of [11–12, 23] in order to obtain the alloys, which blend high mechanical properties and processability. As yearly as in 1970–80th, the authors of [13–18] investigating the Al – Ca – Zn system have shown that zinc dissolving in the Al_4Ca phase forms more ductile $(Al,Zn)_4Ca$ intermetallic compound. These data have been confirmed in the later researches [11–12, 23], and in [22], on studying the Al – Ca – Mg alloys, there was established that magnesium in the Al_4Ca phase does not dissolve. The $(Al,Zn)_4Ca$ intermetallic compound has not yet been studied in more detail. However, on optimizing the composition of Zn-containing aluminum-calcium alloys, it is necessary to take into account the behaviour of this compound. It is important since, on the one hand, high castability is demonstrated by those alloys in which the eutectic share is big enough [26]; on the other hand, precipitation hardening at heat treatment is due to the structure transformations of solid solution. Hence, the more is the solid solution share, the stronger the alloy is as a whole. In this connection, the researchers of eutectic aluminum-nickel alloys (nickalins) have optimized the compositions so that the part of aluminum solid solution was as high as possible on insignificant castability deterioration [27–28]. Lowering the quantity of expensive nickel in the case of the composition optimization of nickalins is quite founded. In the case of $[(Al) + Al_4Ca]$ eutectic based alloys, the calcium content reduction may lead to density increase and corrosion stability decrease, as well as adversely affect casting characteristics. Therefore, in the present paper we have decided to study in detail the formation of the $(Al,Zn)_4Ca$ intermetallic compound as well as the effect of the (Al) solid solution and $[(Al) + (Al,Zn)_4Ca]$ eutectic ratio on the structure and properties of the Al – Ca – Mg – Zn alloys. In this connection, the following tasks were set:

– To plot the hardness dependences of Al – 2.5%Mg – (0...10)%Ca – (0...14)%Zn alloys on conditions of strengthening heat treatment;

– To study the composition and properties of primary crystals and eutectics in Al – 2.5%Mg – 10%Ca alloys with various zinc content;

– Select the alloys of optimal composition in terms of mechanical and casting properties.

Objects and methods of research

The alloys of Al – Mg – Ca – Zn system with a fixed content of 2.5%Mg (wt.%) have been the objects of experimental research; Ca and Zn content in these alloys are indicated in Table 1.

Melting has been carried out in a RELTEC IMS-100-2.4-0.06 (РЭЛТЕК УИП-100-2,4-0,06) induction furnace. Alloys have been prepared using A99 aluminum. Calcium metal wrapped in aluminum foil has been fed in four portions at aluminum melt at the temperature about 780 ^{o}C. Next, at the melt temperature of 730–740 ^{o}C, zinc and magnesium have been introduced in the form of two portions wrapped in aluminum foil. After complete dilution of charge materials, the holding for 5–10 minutes at 740 ^{o}C has taken place for equalizing the melt composition. Then, at the temperature of 720–740 ^{o}C the slag has been removed. After that, the metal was poured into a steel mould at the temperature of 710–720 ^{o}C to obtain flat ingots of 15×30×180 (mm) in size.

For all the alloys, a T6 thermal treatment have been fulfilled in a SNOL 8.2/1100 electric resistance furance: 450 ^{o}C, 3 h + 500 ^{o}C, 3 h followed by water quenching, ageing from 100 to 250 ^{o}C at 25 ^{o}C intervals and holding at each stage for 3 hours. The quenching and each stage have been followed by measuring the hardness HV. The flat sections were prepared by mechanical polishing and the electrolytic one at 12-V voltage in an electrolyte containing 75% of C_2H_5OH, 12.5% of $HClO_4$ and 12.5% of glycerin. The Vickers hardness (HV) has been measured at a load of 50N using a Wolpert 930N (Wolpert, Netherlands).

The microstructure of the cast and heat-treated samples has been studied on an Olympus GX51 light microscope (LM) and a TESCAN VEGA 3 scanning electron microscope (SEM). The TESCAN microscope completed by energy dispersive *X*-ray microanalysis (EDXMA) device manufactured by Oxford Instruments and Aztec software has used to compositions of microstructure constituents.

As in [29], measuring the hardness and elastic modulus of individual microstructure constituents has been fulfilled by selective [30] instrumented indentation (ISO 14577-1:2015) on a NanoHardness Tester (CSM Instruments SA, Switzerland) under the following conditions:

The maximal load applied .... 10 мН
The time of holding force on contact .... 5 s

Inside of each structural component, at least five indentations have been carried out with the imprints placed 12 ... 15 μm apart. The results of the measurements have been processed and averaged using an Indentation 3.83 software (CSM Instruments SA, Switzerland).

Results and discussion

The microstructure of cast alloys without Ca additions looks like dendrites of aluminum solid solution surrounded by the veins of $MgZn_2$ and T ($Al_2Mg_3Zn_3$) phases (Fig. 1, *a*). In alloys without Zn additions (Fig. 1, *b*) an eutectic looks somewhat coarser than that of quaternary alloys (Fig. 1 *c*, *d*). In hypereutectic alloys there are also primary crystals of $(Al,Zn)_4Ca$ phase.

The temperature of maximal hardening caused by precipitation out of (Al) the $MgZn_2$ and T ($Al_2Mg_3Zn_3$) dispersoids amounts to 150–175 ^{o}C. In Fig. 2, *a* the diagrams of the hardness dependence of an ageing temperature for alloys containing different Ca content at a constant Zn content of 10% are represented. It is evident, that the greater is Ca content, the lower is the hardness and less is the precipitation hardening effect. This is connected with augmentation of [(Al) + $(Al,Zn)_4Ca$] eutectic and diminution of share of the solid solution in the alloy. Such dependences have been observed at investigating the eutectic alloys based on Al – Si, Al – Ni, Al – Ce systems and the other ones [23]. The mentioned above alloys are additionally doped by Mg, Zn, Cu and some other traditional reinforces, which do not dissolve in eutectic intermetallic compounds.

An enhancement of the strengthening degree in course of ageing the alloys with 4% of Ca as their Zn content increases is demonstrated in Fig. 2, *b*. This is related to the increase of Zn concentration in aluminum solid solution, since zinc is redistributed between the (Al) and $(Al,Zn)_4Ca$ phase.

Further, the structure and composition of Al – 10Ca – 2.5Mg – (1...14)Zn alloys have been studied. The struc-

Table 1
Notation of experimental alloys depending on content of Ca and Zn

Element content, wt.%	Alloy notation															
	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Ca	15	10	10	10	10	10	6	6	6	–	–	–	7	6	4	4
Zn	–	1	2	4	8	14	4	8	12	8	10	12	–	–	8	10

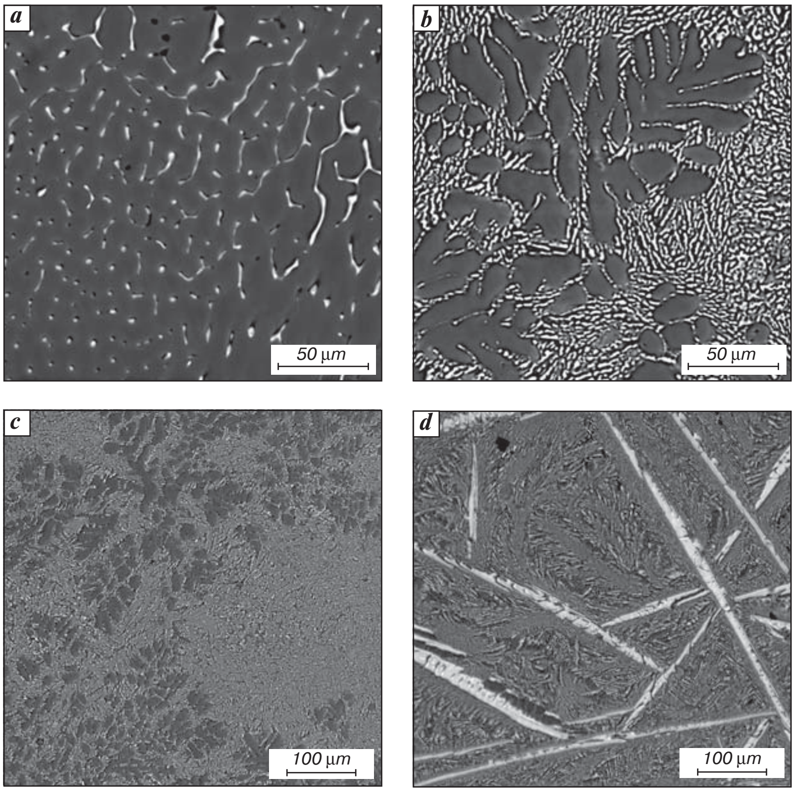


Fig. 1. Microstructure of alloys in as-cast condition, SEM: *a* — Al – 2.5Mg – 8Zn, ×1000; *b* — Al – 2.5Mg – 6Ca, ×1000; *c* — Al – 2.5Mg – 6Ca – 4Zn, ×500; *d* – Al – 2.5Mg – 10Ca – 1Zn, ×500

ture of alloys which contains up to 8% of Zn consists of primary crystals of the (Al,Zn)$_4$Ca compound in the form of rough large plates and the dispersed [(Al) + (Al,Zn)$_4$Ca] eutectic. The higher is the content of Zn in alloy, the greater is amount of (Al,Zn)$_4$Ca primary crystals and the less is their size. The composition of microstructure constituentsof experimental alloys, studied by EDXMA, is represented in Table 2.

Composition of primary crystals in Al – 2.5Mg – 4Ca – 12Zn, Al – 2.5Mg – 6Ca – 12Zn and Al – 2.5Mg –10Ca – 14Zn alloys conforms to the Al$_3$CaZn phase (Fig. 3, Table 3). That is, aluminum in the (Al,Zn)$_4$Ca compound is substituted by zinc, as long as composition of this compound would not be brought in correspondence with the Al$_3$CaZn formula.

In order to estimate the mechanical properties of Al$_4$Ca, (Al,Zn)$_4$Ca and Al$_3$CaZn compounds, an Al – 15Ca alloy consisting of primary crystals of the Al$_4$Ca phase either plate or rounded shape and the [(Al) + Al$_4$Ca] degenerate eutectic has been used as a base one. According to [31], the Al$_4$Ca phase has a body-centered tetragonal crystal lattice with parameters as follows: *a* = 4.36 A (0.436 nm), *c* = 11.09 A (1.109 nm) and its hardness is 200–260 HV. In [32] it is asserted that Al$_4$Ca phase have two modifications: an α-Al$_4$Ca with monoclinic lattice (the low-temperature modification with parameters as follows: *a* = 0.6158 nm, *b* = 0.6175 nm, *c* = 1.1180 nm, β = 88.9°) and β-Al$_4$Ca body-centered tetragonal crystal lattice of BaAl$_2$ type (the high-temperature modification with parameters as follows: *a* = 0.436 nm, *c* = 1.109 nm) and its hardness at room temperature corresponds to 170–220 HV, the Young's modulus is 30 GPa.

The experimental results of nanoindentation of some alloys are listed in Table 3. In Fig. 4 are shown the averaged experimental curves obtained on these alloys. Location of the imprints on sections of primary crystals of Al – 15%Ca and Al – 2.5Mg – 10Ca – 8Zn alloys is displayed in Fig. 5.

One can see from Table 3 and Fig. 4 that hardness and elastic modulus of both primary crystals and the eutectic monotonically grows with increasing Zn content in them. Smaller values of the indentation depth and smaller size of hardness impress correlate with higher hardness values. Mechanical properties reach the highest values in an Al – 2.5Mg – 10Ca – 14Zn alloy.

In present paper we have studied the properties of some microstructure constituents in hypereutectic alloys with the size of several tens of micron, which is quite comfor-

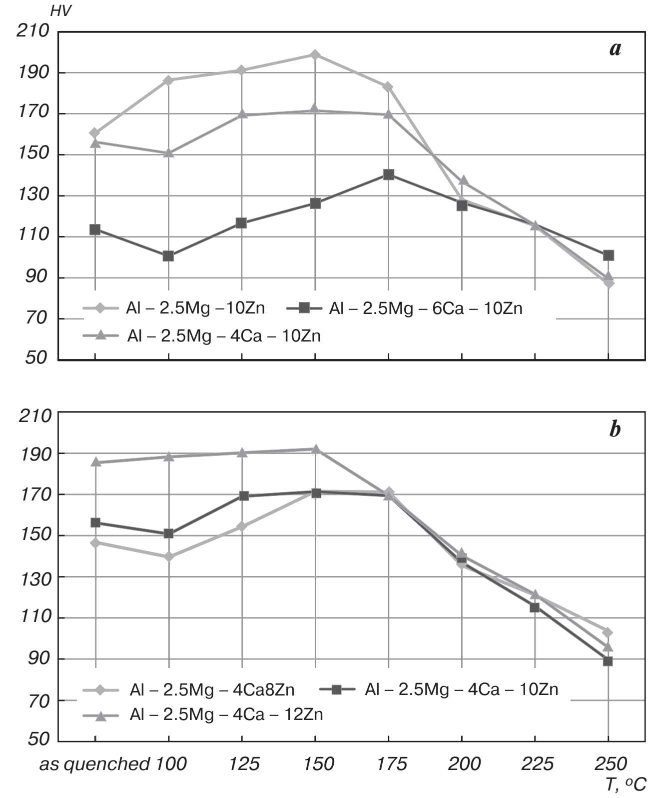


Fig. 2. Dependence of the hardness of experimental alloys of the aging temperature

Table 2
Chemical composition of microstructure constituents according to the EDXMA results

Alloy (notation)		Composition of eutectic [(Al) + (Al,Zn)$_4$Ca]				Composition of primary crystals (Al,Zn)$_4$Ca			
		Mg*	Al	Ca	Zn	Mg**	Al	Ca	Zn
Al – 2,5Mg – 10Ca – 1Zn (1)	wt.%	3.73	88.42	7.0	0.85	0.15	67.3	26.8	5.76
	at.%	4.24	90.58	4.83	0.36	0.19	76.58	20.53	2.70
Al – 2,5Mg – 10Ca – 2Zn (2)	wt.%	1.73	90.18	6.35	1.74	0.28	67.42	24.05	8.25
	at.%	1.97	92.88	4.41	0.74	0.36	77.20	18.54	3.90
Al – 2,5Mg – 10Ca – 4Zn (3)	wt.%	2.31	88.28	6.08	3.34	0.07	59.47	24.93	15.53
	at.%	2.66	91.66	4.25	1.43	0.09	71.88	20.28	7.75
Al – 2,5Mg – 10Ca – 8Zn (4)	wt.%	2.59	87.58	4.17	5.65	0.04	54.16	22.96	22.84
	at.%	3.01	91.61	2.94	2.44	0.05	68.48	19.55	11.92

*An excessive percentage of magnesium is bound up with the presence of Mg-containing phases of eutectic origin
**Presence of tenths shares of magnesium in intermetallic compounds may be related to the “highlight” from the matrix.

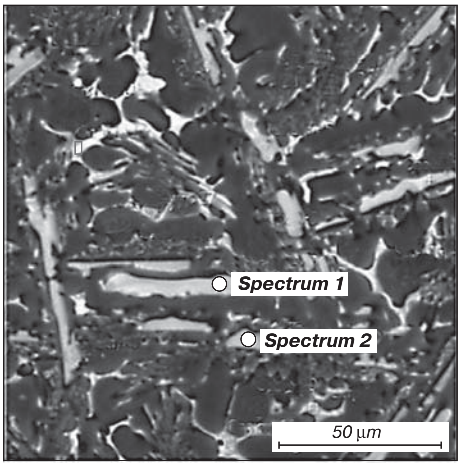
Table 3
Hardness and elastic modulus values of microstructure constituents of experimental alloys

Alloy	No.*	Primary crystals (Al, Zn)$_4$Ca		Eutectics [(Al) + (Al, Zn)$_4$Ca]	
		Hardness, *H* (GPa)	Modulus of elasticity, *E* (GPa)	Hardness, *H* (GPa)	Modulus of elasticity, *E* (GPa)
Al – 15Ca	0	2.3 ± 0.1	53 ± 6	0.9 ± 0.1	63 ± 1
Al – 2.5Mg – 10Ca – 1Zn	1	3.0 ± 0.2	62 ± 1	1.4 ± 0.1	75 ± 4
Al – 2.5Mg – 10Ca – 2Zn	2	3.2 ± 0.1	71 ± 1	1.6 ± 0.1	81 ± 3
Al – 2.5Mg – 10Ca – 4Zn	3	3.5 ± 0.1	80 ± 2	1.4 ± 0.1	86 ± 2
Al – 2.5Mg – 10Ca – 8Zn	4	4.3 ± 0.2	104 ± 6	1.7 ± 0.1	84 ± 4
Al – 2.5Mg – 10Ca – 14Zn	6	4.8 ± 0.3	107 ± 7	2.7 ± 0.1	89 ± 2

*The alloys are numbered according to Table 1.

table for investigating. The dependences obtained may be extended to hypoeutectic alloys, in which zinc is distributed between an eutectic an aluminum solid solution. It is known that elastic modulus is an additive quantity [1–4], therefore one can assume that the total elastic modulus of alloy grows as increasing is the amount of eutectic crystals reinforced by zinc. On the one hand, zinc is being “drawn out” of solid solution, this leads to diminution of the precipitation hardening effect, and the share of the solid solution to be strengthened also decreases. At the same time, the durability of an alloy may grow due to strengthening of (Al,Zn)$_4$Ca phase in eutectic.

Therefore, to increase the strength it is not necessary to reduce the amount of calcium and the share of eutectic in the alloy. The strengthening decrease due to lowering the (Al) share may be partially compensated by increase of durability of



Spectrum	Content of elements, wt.% / at.%				Sum	Phase
	Mg	Al	Ca	Zn		
spectrum1	0.07/0.10	45.01/61.15	22.45/20.53	32.47/18.21	100	(Al,Zn)$_4$Ca = Al$_3$CaZn
spectrum 2	0.07/0.11	44.83/61.13	21.80/20.02	33.30/18.74	100	(Al,Zn)$_4$Ca = Al$_3$CaZn

Fig. 3. Composition of primary crystals in an Al – 2.5Mg – 6Ca – 12Zn alloy (the EDXMA data)

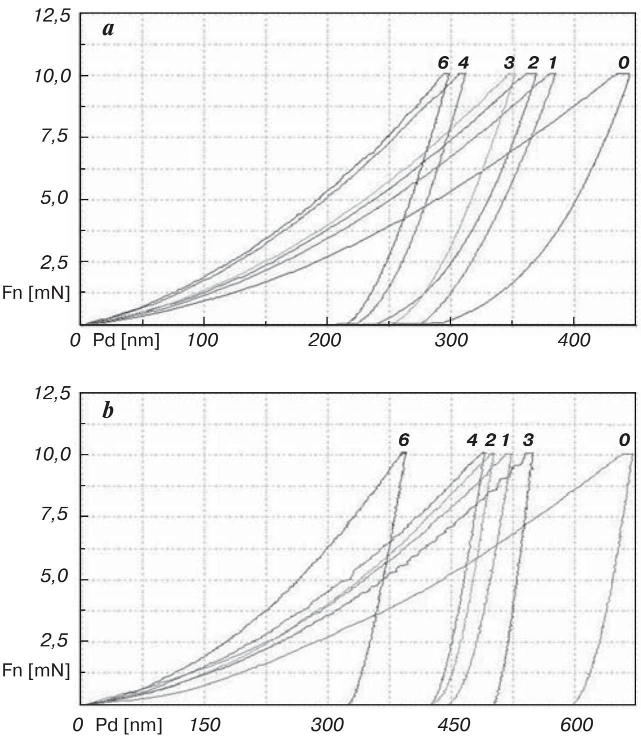


Fig. 4. The experimental averaged nanoindentation curves of primary $(Al,Zn)_4Ca$ crystals (*a*) and the $[(Al) + (Al,Zn)_4Ca]$ eutectic (*b*). The numbers of the curves correspond to the alloys from Table 1. On the diagrams: the vertical axis – the load applied (mN); the horizontal axis – the indentation depth (nm)

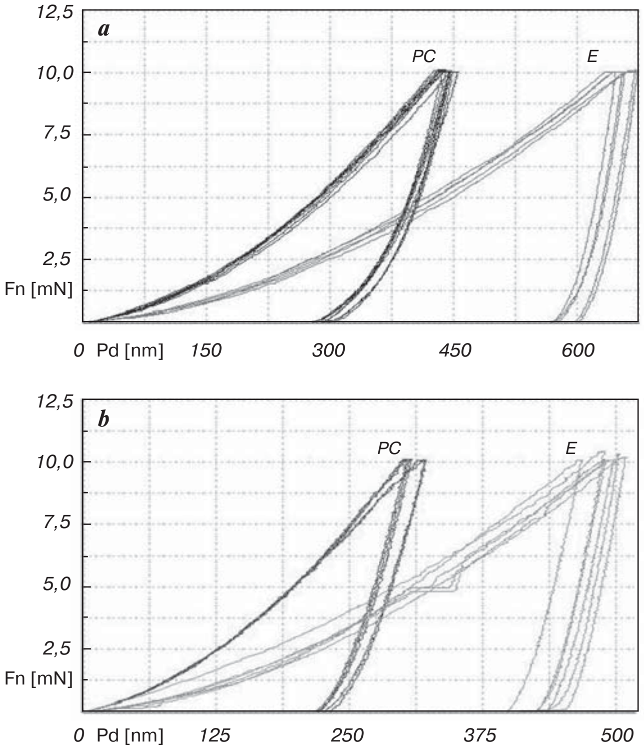


Fig. 5. Nanoindentation curves and photomicrograph of imprints at microstructure constituents of alloys (*PC* — primary crystals, *E* — eutectic):
a — Al – 15%Ca (spacing of imprints 12 μm);
b — Al – 2.5Mg – 10Ca – 8Zn (spacing of imprints 15 μm)

eutectic $(Al,Zn)_4Ca$ crystals. These deductions are confirmed by results of [12, 23], where high mechanical properties have been obtained for an alloy with 3.5% of Ca. At that, the amount of Zn should be not less than 8%, but no more than 10%. Hardness of an intermetallic compound at 8% of Zn is almost twice as high as that of Al_4Ca crystals (Table 2), but if the content of Zn exceeds 10%, the primary crystals appear which deteriorate plasticity of the alloy [11–12, 23].

In terms of hardness of alloys, the changes on alloying are well within the usual dependences, inherent to all eutectic alloys: hardening decreases as the (Al) share lessens. On our research it was obtained somewhat different dependences in alloys, doped by Ca and Zn. It was found the share of the Ca-containing phase in alloy may be kept high without losing the durability. This may be expected to provide high manufacturability on casting, density diminution and increasing the corrosion stability of alloys. [11–25].

Concluisons

– The dependences of hardness in Al – 2.5%Mg alloys with different amounts of calcium and zinc under different heat treatments were investigated. It is established that these dependences have the character peculiar to eutectic alloys based on other systems – Al – Si, Al – Ni, etc. That is, the degree of hardening of alloys decreases with a decrease in the share of aluminum solid solution.

– The composition of primary crystals and eutectic in alloys Al – 2.5Mg – 10Ca – (1...14)Zn is investigated. It is shown that with an increase in the of Zn content in the alloy, its share in the primary crystals increases. It was found that in alloys Al – 2.5Mg – 4Ca – 12Zn, Al – 2.5Mg – 6Ca – 12Zn and Al – 2.5Mg – 10Ca – 14Zn the composition of primary crystals corresponds to the phase Al_3CaZn.

– The hardness and modulus of elasticity of the individual structural components: primary crystals $(Al,Zn)_4Ca$ and eutectic $[(Al) + (Al,Zn)_4Ca]$ in alloys Al – 2.5Mg –10Ca – (1...14)Zn were determined. It is shown that with an increase in the Zn-content in primary crystals, these mechanical properties grow and at 8% of Zn become twice as high comparing with Al_4Ca phase of the binary Al – 15Ca alloy.

– According to the analysis of the results, the best combination of mechanical and technological properties has been achieved in alloys with the content of Ca from 3 to 4%, Zn from 8 to 10% at 2.5% of Mg.

Acknowledgement

This work was supported by Task No. 11.2072.2017/4.6 for the implementation of the project on the theme of "Development of technology for obtaining deformed semi-finished products from alumomatrix eutectic composites reinforced with $L1_2$ nanoparticles without hardening".

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