

INFLUENCE OF NANOPARTICLES INTRODUCING IN THE MELT OF ALUMINUM ALLOYS ON CASTINGS MICROSTRUCTURE AND PROPERTIES

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Abstract

Three types of aluminum alloys such as AlSi7Mg, AlSi12Cu2MgNi and AlZn4 are refined by introducing nanoparticles of high melting temperature compounds—nitrides and carbides as well as diamonds in the melt. Their influence on microstructure, mechanical and some electrochemical properties of castings of these alloys is investigated. It is established that the microstructure is refined, the porosity is decreased, and some mechanical

and electrochemical properties such as protection potential in drinking water are increased. The mechanism of grain refinement and silicon modification is discussed.

Keywords: nanoparticles, casting, aluminum alloys, refining, mechanical and electrical properties

Introduction

Nanosized particles (NP) are used as inoculants for refining the metal microstructure and improving the mechanical properties of products in the last years.^{1–5} Powders of high melting temperature (2000–3000 °C) compounds, nitrides, carbides, borides, etc. with nanodimensions, are introduced in the molten metal. They are obtained usually by means of plasma chemical synthesis and are covered with metal protector. When a homogenous introduction of nanopowders is reached, a local overcooling and volume crystallization conditions are created and the nanoparticles become active centers of crystallization. The investigations of refined with nanoparticles metal alloys show that the mechanical properties (tensile strength and specific elongation^{1–5}) as well as the corrosion and abrasive resistance⁶ increase. Nanoparticles increase the part of the surface energy in the total energetical balance of an alloy which determines the phase transformations. Most of the nanoparticles have low wettability, and so it is difficult to

be wet by the melts. For this reason, they are clad with metal protector. Most of the existing studies are performed on Al alloys,^{1,5,7–10} Mg alloys,^{11,12} cast iron^{4–6,13} and some on steels with specific application.^{3–5} This work enriches the investigations on castings of three types of aluminum alloys refined with nanoparticles.

Aim: The aim of the present work was to investigate the influence of some types of nanoparticles on the structure and properties of aluminum alloys castings.

Experimental

Production of Nanopowders

High melting temperature compounds in form of nanopowders are obtained by plasma chemical synthesis. Nanopowders of TiCN, SiC, TiN and AlN are produced by Neomat CO, Latvija and the Institute of Theoretical and Applied Mechanics—Siberian Branch of RAS. They are passivated with oleic acid for atmospheric influence protection. In order to reach better wettability, the

Dedicated to his Eminence Photius—Bishop of Triaditsa on the Occasion of His 60th Birthday.

nanoparticles are clad with metal protector (Ni, Cr, Fe, Al, Cu, etc.). This is realized by mechanical and chemical processing in planetary mills or free-current method.¹⁴

For refining, aluminum alloys could be used also diamond nanopowders (ND). They are obtained by detonation technology and are clad with metal protector using free-current method.

NP of SiC clad with Cu by free-current method is shown in Figure 1. The presence of larger particles with diameter up to 600 nm as well as smaller ones with diameter up to 100 nm is observed.

Investigated Alloys Castings

Three aluminum alloys castings are investigated, namely “Boat” of AlSi7Mg alloy, piston of AlSi12Cu2MgNi alloy and anode, protector of AlZn4 alloy, as given in Table 1.

AlSi7Mg is refined with:

- 0.05 % AlN and cladding metals Al + Cu
- 0.1 % SiC and cladding metal Cu

AlSi12Cu2MgNi is refined with:

- 0.1 % ND + Ag
- 0.1 % SiC + Cu
- 0.1 % SiC + Cu + 0.03 % AlN + Al [ultrasonic dispersion (USD)]

AlZn4 is refined with:

- 0.05 % AlN + Al (USD)
- 0.1 % ND + Ag.

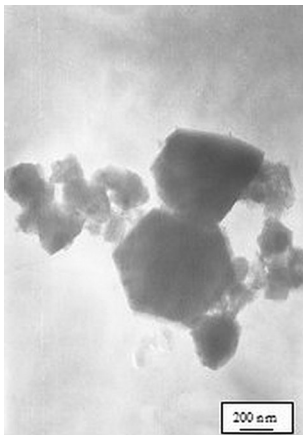


Figure 1. SiC particles clad with Cu.

Technology of Nanoparticles Addition and Casting with and Without Nanoparticles

The technology of NP introduction and casting “Boat” of AlSi7Mg alloy consists in:

- Melting the Al alloy in electric-resistance furnace with crucible capacity of 2.5 kg;
- Degassing molten metal at 730–740 °C by means of blow-through with argon through graphite tube with flow 1 l/min for 5 min;
- Introducing NP wrapped in aluminum foil and capsulated in aluminum cartridge in the melt at 720–740 °C and mechanical stirring with Ti-mixer for 3–5 min (rotational speed—up to 150 min⁻¹);
- Casting in metal mold.

Castings for pistons with 45 mm diameter and 0.17 kg weight of AlSi12Cu2MgNi alloy are produced by direct squeeze casting. This method assures high crystallization rate which means fine microstructure. The castings are designed for pistons with diameter 38–40 mm for racing motorcycles and carting engines with 50 cm³ volume, air cooling and velocity up to 12,000 rpm.

The castings with and without NP are produced in the following conditions:

- Melting the Al alloy in furnace with capacity of 6 kg;
- Temperature of the melt in the furnace 730–740 °C;
- Molten metal degassing by means of blow-through with argon through graphite tube for 3–5 min;
- Temperature of the mold and the punch 150–160 °C;
- Pressing pressure during crystallization—188 MPa;
- Time from the end of pouring up to applying pressure 3–4 s;
- Time of applying pressure 20 s.

The technology of introducing NP and casting anode—protector of AlZn4 alloy—consists in:

- Obtaining AlZn4 alloy in electric-resistance furnace with crucible capacity of 2.5 kg;
- Melt degassing at 730–740 °C by means of blow-through with argon through graphite tube for 3–6 min;
- Introducing NP, wrapped in aluminum foil and capsulated in aluminum cartridge in the melt at 740–750 °C and mechanical stirring melt with Ti-mixer for 3–5 min (rotational speed—up to 150 min⁻¹);

Table 1. Chemical Composition of Investigated Alloys, wt% (Al—the Rest)

Alloy	Si	Mg	Ti	Fe	Cu	Mn	Zn	Pb	Sn	Ni	Cd	In
AlSi7Mg	7.73	0.34	0.02	0.53	0.05	0.03	0.10	0.08	0.05	–	–	–
AlSi12Cu2MgNi	12.73	0.87	0.10	0.39	3.30	0.12	0.10	0.003	0.003	1.75	–	–
AlZn4	0.08	–	–	0.18	0.05	–	3.00	–	0.10	–	0.1	0.03

- The as-obtained alloy is cast according to¹⁵ in bi-seat metal mold at melt temperature 720–725 °C and mold temperature 150–160 °C.

Investigation of Microstructures, Mechanical and Electrochemical Characteristics of Samples from Castings

Metallographic samples are prepared by standard procedure consisting in wet grinding and mechanical polishing. The microstructure of AlSi7Mg and AlSi12Cu2MgNi samples is revealed with 0.5 % water solution of HF. The microstructure of AlZn4 samples is revealed by means of electrolytic etching. The microstructure is characterized by means of metallographic microscope PolyvarMet at magnifications up to 1000×. The quantitative metallographic analysis is carried out by automatic image analyzing system “Olympus MicroImage.” The dendrite arm spacing (DAS), domain and grain size are determined. Si crystals size control is performed. The microhardness of specimens is measured by means of the device MicroDuromat 4000 with load of 10 g, time for reaching the load 10 s and hold time 10 s.

The mechanical properties are determined by standard procedure.

The density d of the melts is measured using gravimetric method. The theoretical density ($d_0 = 2.7050 \text{ g/cm}^3$) of the melts is calculated via additive approach based on the densities and concentrations of components. The porosity P (%) is defined through the formula:

$$P = \frac{d_0 - d_{\text{mean}}}{d_0} 100 \%$$

The installation for obtaining polarization curves and thus estimating electrochemical properties is shown in Figure 2. The polarization curves present the dependence of potential on density of polarization current [$E = f(i)$].

The anode polarization potential is determined at a current density of 5 A/m^2

The investigations held at current density 5 A/m^2 gave a possibility to determine the anode current density at which starts and ends the fast polarization. As-received experimental data showed that at anode current density $i_a = 0.01 \text{ mA/cm}^2$ fast anode polarization starts. Based on

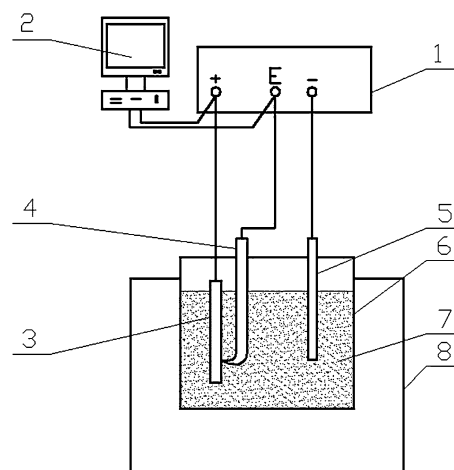


Figure 2. Installation for obtaining polarization curves. 1—Potentiostat 263A-1; 2—PC; 3—tested electrode; 4—comparative electrode (Ag/AgCl); 5—control electrode (Pt); 6—analytical cell; 7—electrolyte (drinking water); and 8—circulation thermostat UNIC-200.

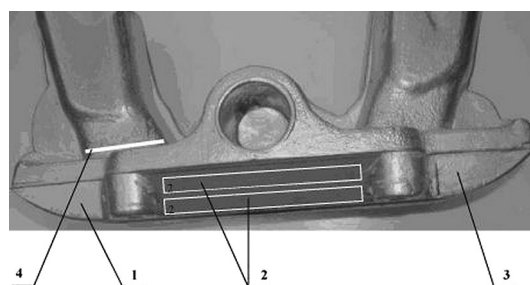


Figure 3. Casting “Boat”—detail of a power-transmission network and zones of casting samples: zone 1— from the side of the riser with cross section 6.6 cm^2 (for testing density and metallographic sample); zone 2— with cross section 12.1 cm^2 (for testing density and mechanical properties); zone 3—from the side of the runner (gate) with cross section 6.6 cm^2 (for testing density); and zone 4—section of the riser (for testing density and sample for macro-analysis).

these data, we chose as an anode current density $i_a = 12 \mu\text{A/cm}^2$. The testing temperatures are 20, 30, 40, 50, 60, 70, 80 and 90 °C.

After measuring the potential at a given temperature, the next higher temperature is set using a thermostat. The time

for reaching needed temperature is from 12 to 15 min. To make sure that the temperature in the thermostat and the voltaic cell is equalized as well as to reach the equilibrium potential, it is put on hold for another 45 min—i.e., the measured potential is per hour. The thermostat and galvanic cell temperature control is held via tarred and tested immersion thermocouple with precision of 0.5 °C.

The potential of anode alloys at a given current density which is equal or higher than the one taken for a minimum borderline potential ($E_{\min} = -980$ mV) could be used as protectors. It shall be noticed that according to international standards, the minimum necessary protective potential for low-alloyed steels is as follows: $E = -720$ to 760 mV at 20 °C and $E = -920$ mV at 85 °C.

Results and Discussion

Casting “Boat” of AlSi7Mg Alloy

The investigated casting called “Boat” is a detail of a power-transmission network, as shown in Figure 3.

The microstructure of samples from casting “Boat” before heat treatment is investigated in Dimitrova et al.⁹ In the present work, we have investigated the microstructure, physical and mechanical properties of casting “Boat” after heat treatment (HT). Castings without NP (Nr 1) and with NP (Nr 2 and 3) are treated according to regime T6:

- homogenization at 535 ± 3 °C—6 h
- water quenching at 25–40 °C

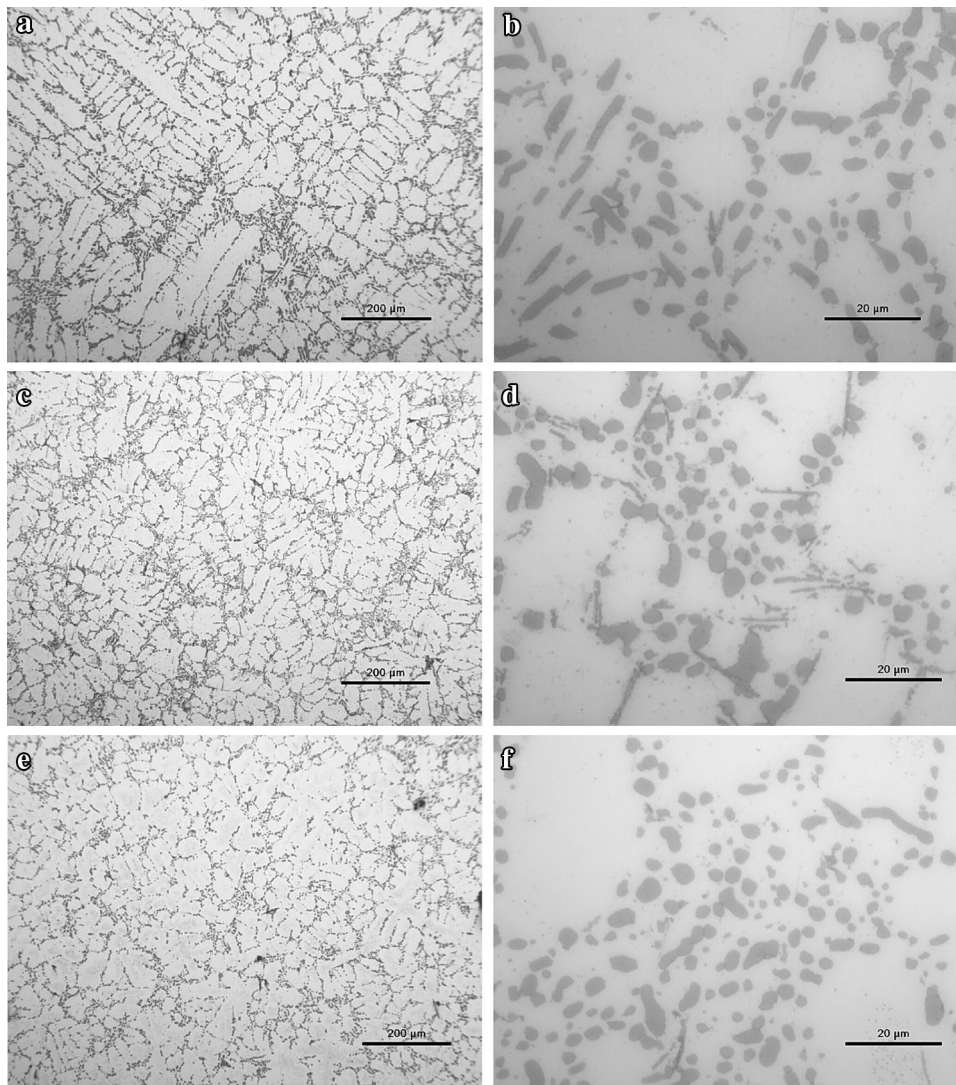


Figure 4. Microstructures of castings type “Boat” of AlSi7Mg after HT: (a, b) casting 1 without NP; (c, d) casting 2 refined with 0.05 % AlN + Al + Cu; (e, f) casting 3 refined with 0.1 % SiC + Cu; (a, c, e) general view of the microstructure; and (b, d, f) eutectic.

Table 2. Microstructure, Physical and Mechanical Characteristics of Refined and Unrefined Castings “Boat” (Alloy AlSi7Mg) After HT (Regime T6)

Casting no.	Type and concentration of NM	DAS (μm)	Length of Si particles (μm)	$R_{0.2}$ (MPa)	R_m (MPa)	A_5 (%)	Q (MPa)	d (g/cm^3 , zone II)	P (% zone II)
1	Without NP	23.90	3.52	240	296	4.0	386	2.6783	0.99
2	0.05 % AlN + Al + Cu	17.69	2.94	253	318	4.3	413	2.6956	0.35
	Alteration (%)	-26.0	-16.5	5.4	7.4	7.5	7.0	0.70	-64.6
3	0.1 % SiC + Cu	17.66	2.59	238	317	6.4	438	2.6950	0.37
	Alteration (%)	-26.0	-26.4	-0.8	7.1	60.0	13.5	0.62	-62.6

- aging at 160 ± 3 °C—6 h.

The microstructures are shown in Figure 4. They present dendrites of α -solid solution and eutectic of α -solid solution, Si particles and intermetallic compounds. The microstructure, physical and mechanical characteristics of initial and refined castings “Boat” after HT (regime T6) are given in Table 2. It could be seen that the nanoparticles introducing in the melt lead to microstructure refinement. Both modifiers reduce DAS values with 26 %. The length of Si particles is decreased with 16.5 % by AlN modifier and 26.4 % by SiC comparing with Si particles length of non-modified sample. It could be supposed that the refining effect of used in this case nanoparticles is based on their properties after the metal covering is melted in the molten metal. They have (1) high melting temperature, (2) low reactivity and (3) extremely high sedimentation stability because of their small size and specific surface.¹⁶ The energy of Brownian motion is sufficient to ensure the particles with size up to 1 micron to be in permanent movement and not to be settled under gravity.¹⁷ Thereby, the nanoparticles act as nucleation centers. According to the known and generally recognized principle of orientation and size matching formulated by Dankov,¹⁸ the crystalline lattice of the new phase is oriented relative to the initial phase so that the arrangement of atoms between lattices in both phases achieves maximum similarity and that the lattice atoms underwent minimal displacement. The crystal structure of SiC (β , which is used by us) is cubic with lattice parameter $a = 4.3596$ Å. The Al one is also cubic with $a = 4.046$ Å. The difference between lattice constants is $7.19\% < 10\%$, so the requirement for orientation and size matching is fulfilled and the SiC particles could be substrate on which the Al crystals generate. It could be assumed that this requirement is not obligatory for nanomodifiers since the AlN does not fulfill it, but the microstructure is refined. Such a finding is made in Stanev et al.¹⁹ where diamond nanopowder is used as modifier for AlSi7Mg, and although its crystalline constant differs with 12 % from Al one, the grains are refined. Proving or disproving this assumption requires further research on crystal lattice level. We suppose the Si particles are refined and rounded because of diminished volumes between dendrite

arms (DAS) where the particles grow. Nanoparticles have a double-modifying effect: firstly, they serve as centers of crystallization, and secondly, being very numerous in number and being a long time in a suspended state, they block the diffusion of the respective atoms to emerging and growing crystals that in the end contributes to a fine-grained structure formation.^{17,20} It is found that irrespective of the chemical composition of the NP, their crystal system, crystal lattice constants, density, melting temperature and other parameters, they all have similar modifying effect.²⁰

As a result of NP introducing in the melt of AlSi7Mg, microstructure refinement is obtained and mechanical properties are improved. In the case of AlN using as modifier, the mechanical characteristics are raised with several percent, as given in Table 2. The elongation increases mostly with 60 %. That is due especially to the Si particles length reduction with 26.4 %.

The influence of NP on porosity and quality index Q of casting “Boat” is presented in Figure 5. The quality index Q is calculated by the formula:²¹

$$Q = R_m + 150 \log A_5 \text{ (MPa)}$$

It is noted that both modifiers reduce the porosity significantly from about 1 to 0.4 %. The presence of a sufficient quantity of nanoparticles leads to the emergence of smaller crystals and thus assures greater density of the casting.

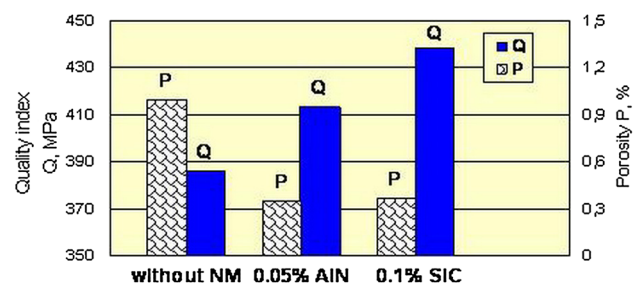


Figure 5. Influence of NP on the porosity and quality index Q of casting “Boat.”

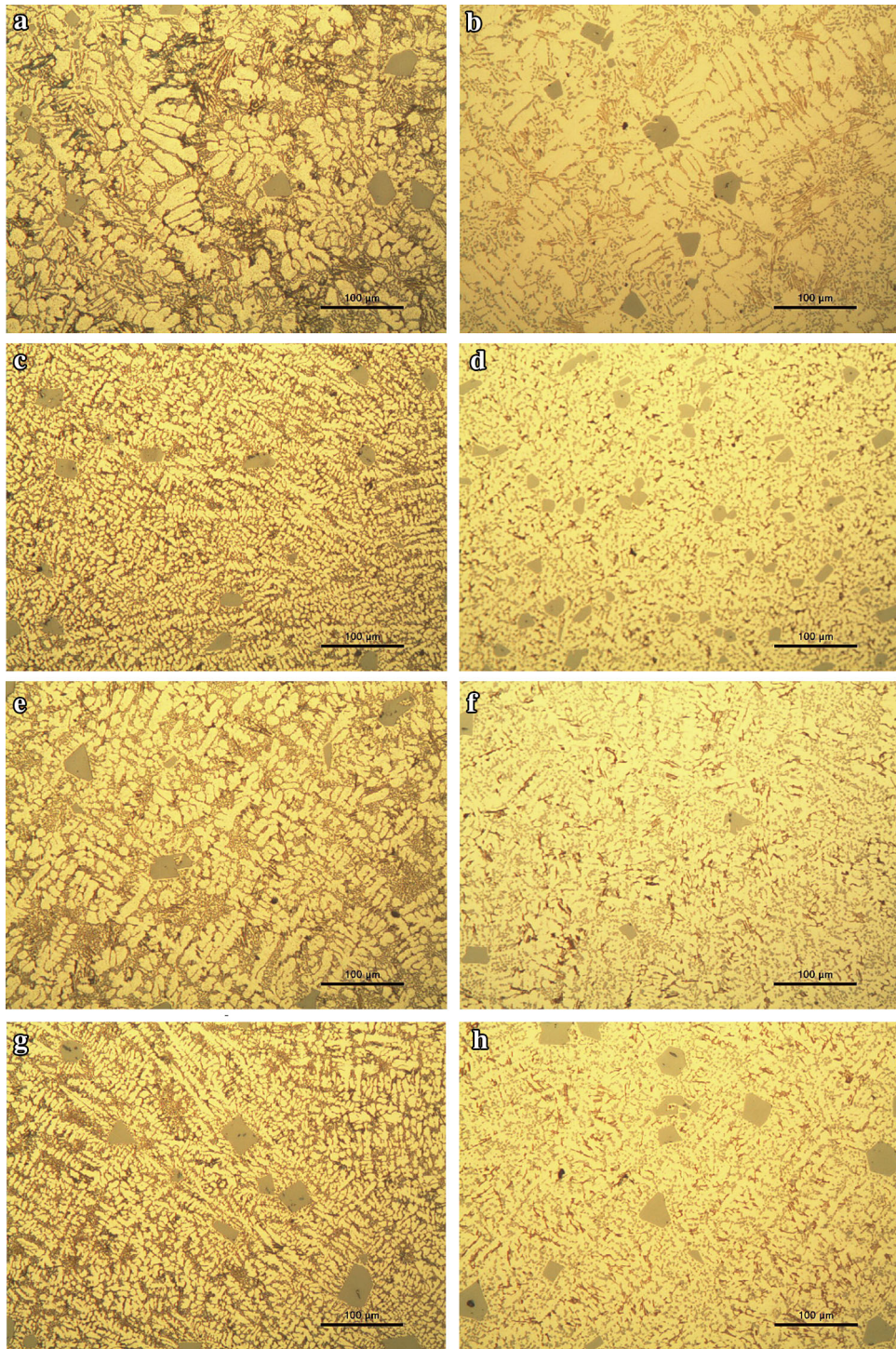


Figure 6. Microstructure of AISi12Cu2MgNi alloy: (a, c, e, g) in as-cast stay and (b, d, f, h) after heat treatment. (a, b) Samples without NP; (c, d) samples with 0.1 % ND + Ag; (e, f) samples with 0.1 % SiC + Cu; and (g, h) samples with 0.1 % SiC + Cu + 0.03 % AlN + Al.

Table 3. Testing Results of Pistons Before and After HT

Type and concentration of NP	DAS (μm)	d_{cp}^a (g/cm^3)	$R_{0.2}^a$ (MPa)	R_m^a (MPa)	A_5^a (%)	HB ^a
Without NM	10.56	2.7685/2.7689	243/367	266/388	0.4/0.5	114/154
0.1 % ND + Ag	8.67	2.7686/2.7692	233/369	273/393	0.7/0.6	118/154
Alteration (%)	-17.9	-	-4.1/0.5	2.6/1.3	75/20	3.5/0
0.1 % SiC + Cu	9.64	2.7694/2.7696	238/373	283/402	0.7/0.7	117/154
Alteration, %	-8.7	-	-2.1/1.6	6.4/3.6	75/40	2.6/0
0.1 % SiC + Cu + 0.03 % AlN + Al (USD)	7.92	2.7698/2.7700	245/371	281/404	0.7/0.8	115/152
Alteration (%)	-25.0	-	0.8/1.1	5.6/4.1	75/60	0.9/-1.3

^a The numerator values are corresponding to untreated castings; the denominator values—heat-treated castings (regime T6), DAS values are measured before heat treatment

Table 4. Potential—E in Drinking Water After 1-h Stabilization

Potential—E (mV) in drinking water after 1-h stabilization							
Nr 1—without NM		Nr 2.1—with 0.05 % AlN + Al (USD)			Nr 3.2—with 0.1 % ND + Ag		
T (°C)	-E (mV)	T (°C)	-E (mV)	ΔE (%)	T (°C)	-E (mV)	ΔE (%)
20	689	20	751	9.0	20	725	5.2
30	744	30	780	4.8	30	754	1.3
40	770	40	795	3.2	40	785	3.2
50	794	50	845	6.4	50	834	5.0
60	891	60	913	2.5	60	913	2.5
70	1005	70	995	-1.0	70	998	-0.7
80	1039	80	1023	-1.5	80	1027	-1.1
85	1049	85	1031	-1.7	85	1045	-0.4

Table 5. Potential—E (mV) in Drinking Water After 10-h Testing

No.	Treatment	Temperature of drinking water (°C)							
		20	30	40	50	60	70	80	90
1	Without NP	1167	1175	1179	1202	1219	1230	1235	1253
2.1	0.05 % AlN + Al (USD)	1175	1181	1193	1213	1236	1244	1255	1266
	ΔE (%)	0.7	0.5	1.2	0.9	1.4	1.1	1.6	1.0
3.2	0.1 % ND + Ag	1173	1181	1192	1210	1233	1241	1254	1263
	ΔE (%)	0.5	0.5	1.1	0.7	1.4	0.9	1.5	0.8

Table 6. Microstructure Characteristics and Density of AlZn4 Castings Designed for Protectors

Casting no.	Type and concentration of NM	Mean diameter of domains d (mm)	Mean grain diameter (μm)	Grain roundness	HV (kg/mm^2)	d (g/cm^3)
1	Without NP	1.96	61.18	1.42	38.5	2.7516
2.1	0.05 % AlN + Al (USD)	0.95	51.30	1.30	36.9	2.7556
	Alteration (%)	-51.5	-16.1	8.5	-4.1	0.15
3.2	0.1 % ND + Ag	1.23	47.05	1.33	41.2	2.7587
	Alteration (%)	-37.2	-23.1	6.3	7.0	0.26

The electrochemical characteristics in drinking water after 1-h stabilization and after 10-h testing are shown in Table 4, Table 5 and Figure 7, respectively

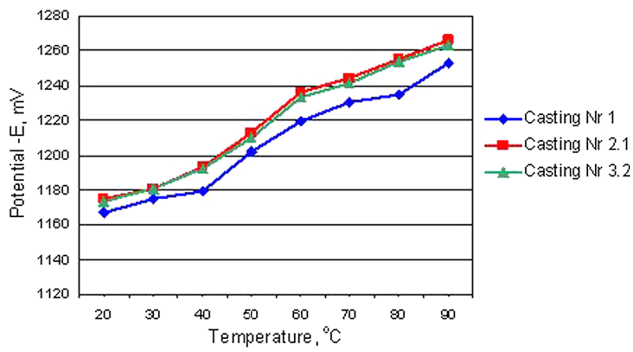


Figure 7. Increased potential during anodic polarization in dependence on the drink water temperature after 10-h testing.

After introducing 0.05 % AlN + Al + Cu and 0.1 % SiC + Cu in AlSi7Mg alloy, it could be seen:

- refining microstructure—DAS decreases with 26 %;
- decreasing porosity up to 70 %. The values are smaller than 0.5 % and the requirements for casting density are satisfied;
- increasing yield strength with 7 % and elongation up to 60 %;
- tensile strength changes less. More favorable is the influence of 0.1 % SiC + Cu addition;
- effect of NP influence on the microstructure and properties after HT is less compared to this one before the HT;

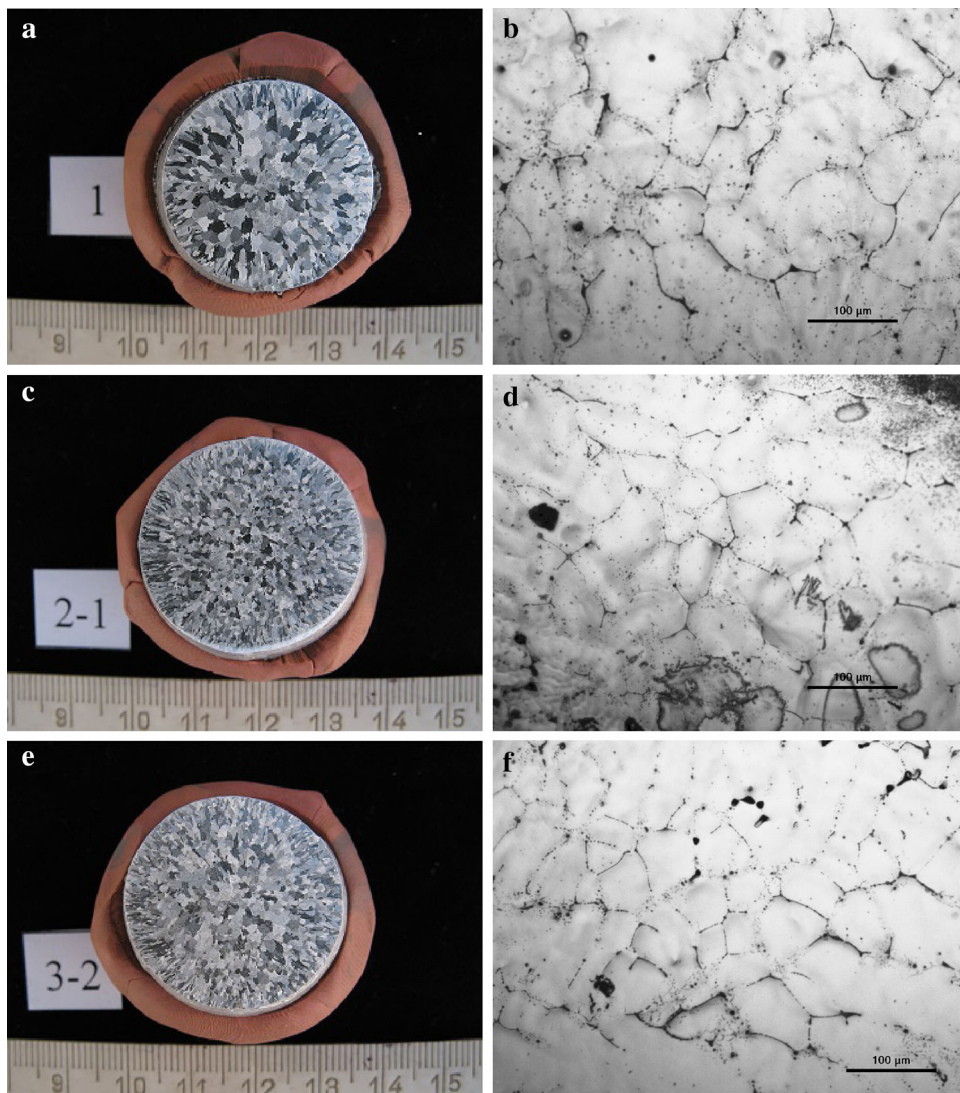


Figure 8. Macro- and microstructures of AlZn4 castings designed for protectors: (a) casting 1 without NP; (b) casting 2.1 refined with 0.05 % AlN + Al (USD); and (c) casting 3.2 refined with 0.1 % ND + Ag.

- HB > 80 in all zones of casting and meets the requirements;
- values of quality index Q increase with 7–14 % and surpass 400 MPa. This is sufficient to assure 10^7 cycles fatigue endurance at load 70–80 MPa, which is usually required for dynamic loaded parts and proves increased reliability of castings with NP addition.

All this means that nanopowder introducing in the melt of AlSi7Mg alloy leads to mechanical properties and total quality of castings improvement.

AlSi12Cu2MgNi Alloy

The microstructure of this alloy consists also of dendrites of α -solid solution, $Al_{15}(FeMn)_3Si_2$ phase—primary particles and eutectic of α -solid solution, $Al_{15}(FeMn)_3Si_2$ phase particles and intermetallic compounds,²² as shown in Figure 6. The results from measuring microstructure parameters and mechanical testing the pistons before and after heat treatment are shown in Table 3.

NP addition leads to decreasing DAS up to 25 %. In cast state, R_m increases with 3–6 % and A_5 with 75 %. After HT, the increasing is less: R_m is up to 4 % and A_5 is up to 60 %. The hardness changes insignificantly. After HT, HB > 150 and fulfills the requirements. SiC + AlN has stronger influence than other investigated nanoparticles.

AlZn4 Alloy

Investigation of Castings ‘Anode Protector’ of AlZn4 Alloy with NP Addition

The influence of nanosized AlN particles and nanodiamonds on the microstructure and electrochemical characteristics of ‘Anode protector’ AlZn4 casting is investigated. It is designed for corrosion protection of enamel tank water heater and hulls of ships.

It is seen that the nanosized powders, AlN and ND + Ag added in AlZn4 alloy, influence on the electric potential of the protector alloy increasing the potential electronegativity as follows:

- The influence of both NP in drinking water after 1-h stabilization occurs up to 50 °C (E increases with 5–9 %). Then the values reach the values of initial alloy. Since we do not know other publications related to the electrochemical behavior of nanomodified alloys, the explanation for the changes above 60° we could give after conducting further research in this direction;
- The protective potential E of alloys with NP addition after 10 h in drinking water is higher than

the initial one in whole temperature range (20–90 °C). Its increasing at 85 °C reaches 1.6 %;

- The protective potential E of all investigated alloys at 85 °C is higher than the accepted by us minimal limitrophe potential ($E_{min} = -980$ mV). It means that the cast anodes can serve as protectors.

The microstructure characteristics and density of AlZn4 castings designed for protectors are shown in Table 6.

Macro- and microstructures of AlZn4 castings designed for protectors are shown in Figure 8. It is visible that the structure is refined after NP addition. The values of mean diameter of the domains decrease with 37.2 % and the mean grain diameter with 23 % (Table 6).

The grains after NP addition are more rounded (1—ideal circle) that means more equal axed which is prerequisite for increasing protective potential. There is a tendency of raising density. The increase in density is also beneficial for the potential. The microhardness increases with 7 %. It could be concluded that the protective potential is structurally sensitive property.

Conclusions

1. After introducing NP in AlSi7Mg, it is established that microstructure is refined (DAS decreases with 26 %). Porosity is decreased (up to 70 %). The values are smaller than 0.5 % so they satisfy the requirements for casting density. Yield strength is increased with 7 % and specific elongation up to 60 %. Tensile strength changes less. Most favorable is the influence of 0.1 % SiC + Cu addition. NP influence on microstructure and properties after HT is less compared to the one before HT. HB > 80 in all zones of casting and requirements are met. Values of quality index Q increase (with 7–14 %) and surpass 400 MPa. This is sufficient for dynamic loaded parts.
2. NP addition leads to decreasing DAS (up to 25 %) in AlSi12Cu2MgNi alloy. In cast state, R_m increases (with 3–6 %) and A_5 too (with 75 %). After heat treatment, the increasing is less (R_m raises up to 4 % and A_5 up to 60 %). The hardness changes insignificantly. HB increases (over 150) and fulfills the requirements.
3. The influence of NP in AlZn4 alloy on its electrochemical characteristics as well as on the microstructure is established. There is a tendency towards an increasing of these characteristics. The increasing protection potential at 80–85 °C in drinking water reaches 1.5–1.6 %. The microstructure is refined. The mean diameter of domains decreases (up to 51.5 %) and the mean grain diameter too (up to 23 %). After nanodiamond

addition, the microhardness increases (up to 7 %). The density increases too.

- The carried out investigation affirms the statement that the nanoparticles introduced in the melt of aluminum alloys act as centers of crystallization refining the microstructure. As a result, the microstructure susceptible mechanical and electrochemical properties are improved.
- The mechanism of grain refining is discussed.

Acknowledgments

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