MODIFYING OF ALUMINUM ALLOY AlSi7Mg WITH NANOCOMPOSITIONS

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***Abstract**: A methodology was developed for the casting of samples of AlSi7Mg alloy modified with nanocomposites (NCs). The following NCs were used: SiC + Cu, AlN + Cu + Al, TiN + Cu, SiC + Al, SiC + Ag Cu, where Ag and Al are cladding metals. The introduction of the nanocompositions takes place in the crucible of a furnace. A thin-walled cylindrical casting mould is used on whose axis is installed a protected thermocouple to measure temperature variation as a function of time. The effect of NCs on overcooling and on sample structure is established.*

***Key words:** NANOPARTICLES, NANOCOMPOSITION, MODIFICATION, CRYSTALLIZATION*

1. Introduction

The combination of temperature and structural research allows a thorough study of the interaction of different types of nanocompositions (NCs) with an aluminum alloy melt and of the crystallization of the alloy in their presence. In [1] are presented the results of the thermal and metallographic analyses of AlSi7Mg alloy before and after modification with two types of nanoparticles: ND (nanodiamond) + Ag (0.1%) and TiN + Al (0.03-0.3%). Using thermal analysis, the temperature changes during the different stages of crystallization and the values and the duration of overcooling as a result of the modification have been registered. By means of metallographic analysis, it has been found that modifying with NCs results in a significant presence of small-sized α-grains with cellular structure and in the refining of the silicon and intermetallic crystals. The average grain diameter decreases by 26-28%, DAS values by up to 13%, and microhardness increases by up to 15%. The influence of the cooling rate on the hardening and the properties of AlSi and AlMg alloys is investigated in [2]. From the temperature curves obtained by a specially developed apparatus, the cooling rate and the solid phase content in the biphasic zone for the various types of alloys has been determined. Microstructure improvement at high cooling rates has been established. The effect of the varying amount of a classical modifier Al-5Ti-1B on the macro and microstructure characteristics and on the characteristic parameters of the cooling curve of aluminum alloy 319 is investigated by thermal analysis in [3]. Acceleration of the crystallization as a result of the introduction of the modifier has been obtained, and it has been found that the parameters of the hardening process depend on grain refinement. A study on the influence of 0.2% Ti and 0.03% Sr on the temperature cooling curves and on the micro and macro structure of alloy A356 shows a change in the character of the curves and refinement of the macrograins as a result of the introduction of the additive [4]. The present work offers a methodology and an experimental device, both developed by the research team, for the casting and thermal analysis of small samples of alloy AlSi7Mg modified with NCs. It shows how modification with nanoparticles influences the parameters of the crystallization process and the structure of the alloy.

2. Experimental Studies

Casting alloy AlSi7Mg with chemical composition: 6.65wt% Si; 0.49wt% Fe; 0.05 wt% Cu; 0.28 wt% Mg; 0.07wt% Zn, determined by spectral analysis, is modified with NCs based on nanopowders (NPs): SiC, TiN, TiCN and AlN. To improve the wetting of the nanoparticles by the metal melt, metal cladding is carried out. Various methods of cladding are applied.

The following NCs are obtained by the method of mechano-chemical treatment of the powders in a centrifugal planetary mill: (1part TiCN+2 partsTi) 0.04wt%TiCN and (1partTiN+1.5parts Cu) 0.05wt%TiN. For this purpose, nanosized powder is mixed with micron-size particles of Ti or Cu. Metallization of nanoparticle surface is achieved after treatment in the planetary mill.

Cladding by currentless method is used to obtain the following NCs: (SiC + Cu) 0.03 wt% SiC and (SiC + Ag) (0.03, 0.05, and 0.17) wt% SiC.

The following NCs: (4 parts AlN + 1part Cu + 12 parts Al) 0.05wt% AlN and (4 parts SiC + 1 part Cu + 12 parts Al) 0.05wt% SiC are obtained by extrusion. In this case, a mixture of NPs with aluminum and copper particles is homogenized, placed in a container and extruded through a suitable nozzle.

NCs of the type (1part SiC + 4 parts Al) of 0.04wt% SiC are introduced into the melt in the form of briquettes obtained by compressing a mixture of nanoparticles and micron-sized Al particles.

a) b)

***Fig.1** a) General appearance of the installation for the casting of cylindrical samples.*
b) Casting mould on whose axis is installed a thermocouple.

A methodology has been developed in which, by tilting the crucible removed from the melting furnace, the melt is poured directly into a thin-walled cylinder-shaped casting mould made of stainless steel. On the axis of the mould is installed a type K fast-acting mantle thermocouple TCMI-K1x100G + 3m 800^{o}C with outside diameter of the working part 1mm and a length of 100mm. At the bottom of the mould is a bushing made of cladded sand to reduce the heat flow (Fig. 1a). The thermocouple is mounted in a thin-walled steel tube to protect it from the aluminum melt. Figure 1 b) shows the gripping device of the thermocouple pressed against the protective thin-walled tube by means of a tension spring which provides the required pressure. This ensures a minimum thermal resistance on the boundary thermocouple-protective tube, resulting in minimum thermal inertia when registering the rapidly changing temperature. The sequence of the experimental steps is as follows: melting aluminum alloy in a crucible, melt degassing, introduction of NCs, homogenization using an impeller, removing the crucible from the furnace and casting the melt into the mould until it is full. Throughout all the procedures the temperature is recorded by an archiver, which makes it possible to obtain information of temperature change as a function of time with a step of 0.2 seconds. The melting is carried out in an 11 kW electric resistance furnace in which is installed a crucible with a capacity of 1.0 kg. The melt is degassed at a temperature of 730 to740 ° C by using tablets of *Degaser* (product of the firm "Region CM"). The tablets are introduced below the melt level with a special device. A degassing method with argon blowing of the melt is also used. The degassing time is from 1 to 3 minutes. Next, the respective NC, placed in an aluminum container attached to the impeller, is inserted. The impeller is immersed below the melt level. Mechanical stirring follows in order to melt the container and finally the melt is homogenized for 3-5 minutes at a rotation speed of about 150 revolutions per minute and a melt temperature of 720-740 ° C.

a)

Fig.2
a) Homogenization of the melt

b) *Sampling*

As a result of the homogenization of the melt, its temperature decreases and this necessitates reheating to reach the casting temperature of 720 ° C. A picture of the homogenization process is shown in Fig. 2a. The casting of samples is carried out by removing the crucible from the furnace with a special appliance and pouring into the mould. Fig. 2b shows the casting of a sample. All experiments were conducted at the same melt temperature, which is continuously controlled by a thermocouple immersed in the crucible and protected with a corundum tube (Fig. 2a). The signal from the thermocouple is visualized on the screen of the archiver.

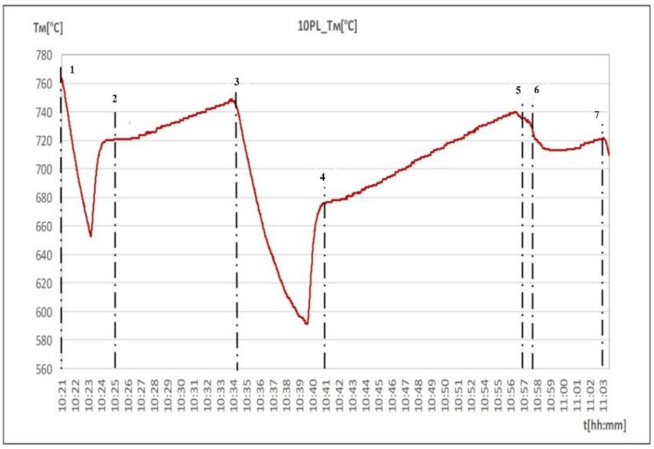


Fig.3 *Ddependence of the ttemperature of the melt in the furnace crucible on time; 1 - start of degassing, 2 - end of degassing, 3 - introduction of NCs and homogenization, 4 - end of homogenization, 5 - sampling.*

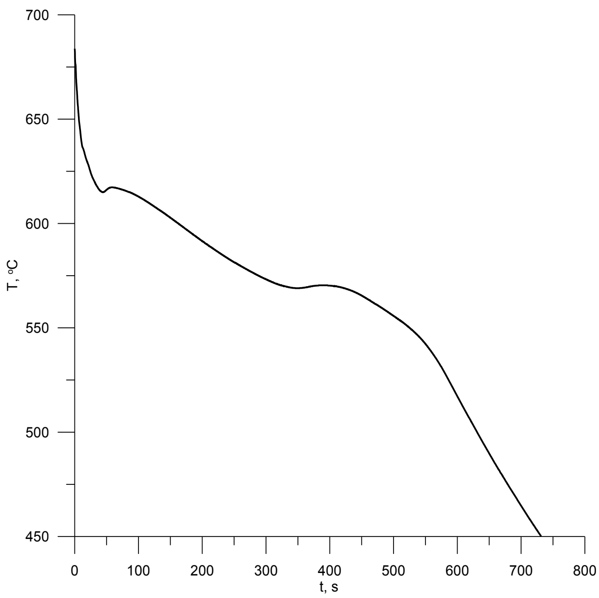
Fig.3 shows the dependence of the temperature of the melt in the furnace on time during the preparatory operations and during the casting of the experimental samples. Similar dependencies have been obtained for all casted samples.

Fig. 4 *Casted sample with the mould*

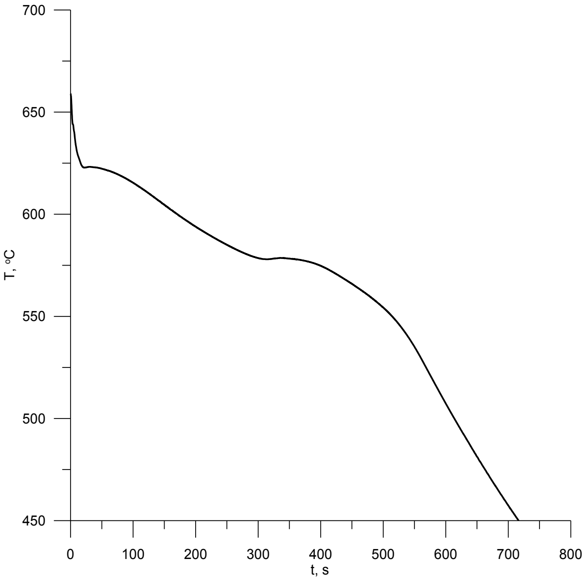
Fig. 4 shows the mould filled with crystallized alloy AlSi7Mg. After the completion of each experiment, an array of melt and sample temperatures data was obtained and time-dependent graphs of the temperature were built. For capturing and analyzing the macrostructure, the samples were prepared according to a standard procedure – grinding with Nos120, 220, 400 and 600 sandpaper, and etching with Poulton's reagent. The macro samples are photographed using a digital camera Canon Power Shot G7. A section of a measurement line is captured on the pictures for scaling and for quantitative analysis which is done using licensed Olympus micro imaging software. The average diameters of the macrograins are determined (the average diameter is the mean length of the diameters, passing through the centre of the object, and measured with a 2-degree step).

3. Results and discussions

3.1. Temperature dependencies

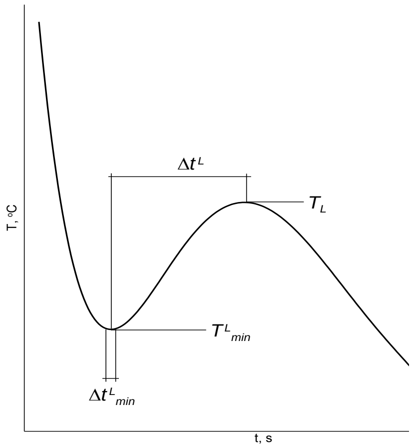


Фиг.5 *Temperature dependence on time: a) for sample G3 without NCs and b) for sample G6N with NC 0.05wt% TiN*
a)



b)

Figure 5 shows the experimentally obtained dependence of the temperature on time for a sample without NC (Fig. 5a) and for a sample with NC: 1 part TiN + 1.5 parts Cu with nanoparticle concentration of 0.05wt% TiN (Fig. 5b). Similar dependencies are obtained for each experiment.



Фиг.6 Schematic diagram showing how the quantities characterizing the alloy crystallization process are determined.

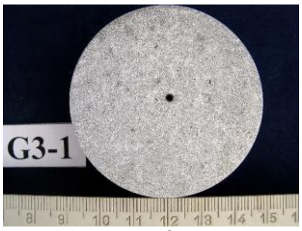
The data for the experimentally obtained temperature serve to determine some of the quantities characterizing the alloy crystallization process after the introduction of NCs. They are: T_0 - initial melt temperature, T_L - liquidus temperature, $DT_L = T_L - T^L_{min}$ - overcooling at the liquidus temperature, T_e - eutectic temperature, DT_e - overcooling at the eutectic temperature. Fig. 6 shows how these quantities are determined.

Table 1. Crystallisation parameters of AlSi7Mg alloy modified with NCs.

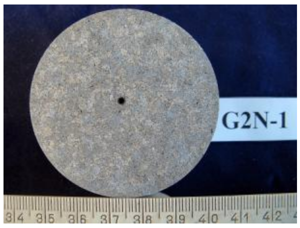
Sample No	NC type	T_0, ^0C	DT_L, ^0C	T_L, ^0C	DT_e, ^0C	T_e, ^0C
G3	without NCs	684	2.44	617.3	1.4	570.4
G2N	currentless covering, SiC+Cu 0,03wt%SiC	650	0.68	621.2	0.6	576.7
G5N	extrusion, 4 parts AlN+1 part Cu +12 parts Al 0.05wt%AlN	666	0.87	622.8	0.3	577.8
G6N	planetary mill, 1 part TiN+1.5 parts Cu 0.05wt%TiN	659.9	0	622	0.6	578.6
G7N	extrusion, 4 parts SiC+ 1 part Cu+ 12 parts Al 0.05wt% SiC	658	0.2	622.9	-	577.5
G8N	planetary mill, 1 part TiCN+ 2 parts Ti 0.04wt% TiCN	655	0.49	624.1	0.3	578.2
G9N	tableting, 1 part SiC+ 4 parts Al 0.04wt%SiC	654	0.58	620.56	0.3	575.8
G10N	currentless covering, SiC+Ag 0.03wt%SiC	669	0	622	0.1	579.1

Table 1 shows the types of NCs used. The processed data shown in Table 1 show a reduction of the overcooling for the NC-modified samples.

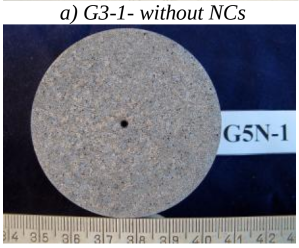
3.2 Study of the macrostructure



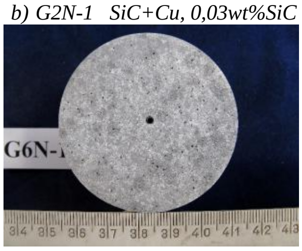
a) G3-1- without NCs



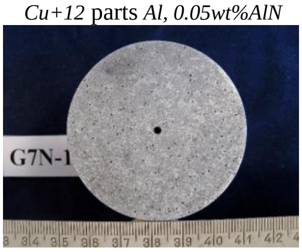
b) G2N-1 SiC+Cu, 0,03wt%SiC



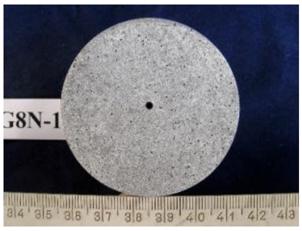
***c)** G5N-1 4 parts AlN+1* part *Cu+12* parts *Al, 0.05wt%AlN*



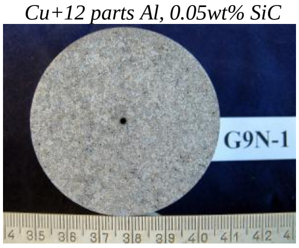
d) G6N-1 1 part*.TiN+1.5* parts *Cu, 0.05wt% TiN*



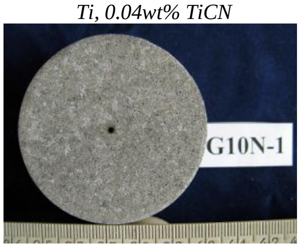
e) G7N-1 - 4 parts *SiC+1* part *Cu+12 parts Al, 0.05wt% SiC*



f) G8N-1 - 1 part *TiCN+2* parts *Ti, 0.04wt% TiCN*



***g)** G9N-1 1* part *SiC+4* parts *Al, 0.04wt%SiC*



***h)** G10N-1 SiC+Ag, 0.03wt%SiC*

Fig.7 Macrostructures of samples without and with NCs

In Fig. 7 are shown the numbers of the samples tested, the NCs used and the sample macrostructure.

Table 2. *Results for the average grain diameter of samples of AlSi7Mg alloy.*

Sample No	NCs used	Dave, mm	Dave change, %
G3-1	without NCs	2.089	-
G2N-1	currentless covering, SiC+Cu 0,03wt%SiC	1.87	-10.5
G5N-1	extrusion, 4 parts AlN+1 part Cu+12 parts Al 0.05wt%AlN	1.23	-41.1
G6N-1	planetary mill, 1 part TiN+1.5 parts Cu 0.05wt%TiN	1.028	-50.8
G7N-1	extrusion, 4 parts SiC+1 part Cu+12 parts Al 0.05wt% SiC	0.964	-53.9
G8N-1	planetary mill, 1 part TiCN+ 2 partsTi 0.04wt% TiCN	1.136	-45.6
G9N-1	tableting, 1 part SiC+4 parts Al 0.04wt%SiC	1.22	-41.6
G10N-1	currentless covering, SiC+Ag 0.03wt%SiC	1.79	-14.3

The results for the average diameters of the macrograins obtained from the quantitative analysis of AlSi7Mg alloy samples are shown in Table. 2. Sample No G3-1 is a basis for calculating the percentage change of the average diameters. Grain refinement of the macrograins for the samples modified with NCs (4 parts AlN + 1 part Cu + 12 parts Al), (1part TiN + 1.5 parts Cu), (4 parts SiC + 1 part Cu + 12 parts Al), (1part TiCN + 2 parts Ti) and (1 part SiC + 4 parts Al) is similar (41-54%), which proves the repeatability of the nanomodification effect. The reduction of the average diameters for all modified samples is between 10% and 54%.

4. Conclusions

The conducted studies of the modification of aluminum alloy AlSi7Mg with NCs (1 part TiCN+2 parts Ti); (1 part TiN+1.5 parts Cu); (SiC+Cu); (SiC+Ag) (4 parts AlN+1 part Cu+12 parts Al); (4 parts SiC+1 part Cu+12 parts Al) and (1 part SiC+4 parts Al) confirmed the reduction of the overcooling and grain refinement for the samples with NCs compared to those without NCs. As a result of the methodology proposed by the research team, sustainable results have been obtained with a very good repeatability of the modification effect, which is a good basis for the introduction of the method into industry.

5. References

1. Stanev S., R. Lazarova, S. Konstantinova, V. Manolov, Investigation of the Effect from AlSi7Mg Alloy Modification with Nanopowders by Thermal and Metallografic Analyses, 26th International Scientific Conference, 65 Years Faculty of Machine Technology, 13-15 September, Sozopol, Bulgaria

2. Marchwica, Paul, "Microstructual and Thermal Analysis of Aluminum-Silicon and Magnesium-Aluminum Alloys Subjected to High Cooling Rates" (2012). *Electronic Theses and Dissertations.* Paper 5572.

3. S.G.Shabestari, M.Malekan Assessment of the effect of grain refinement on the solidification characteristics of 319 aluminum alloy using thermal analysis Journal of Alloys and Compounds Volume 492, Issues 1–2, 4 March 2010, Pages 134-142.

4. Gutiérrez V., G. González A. García, Thermal Analysis of Grain Refinement and Modificati on of an A356 Cast Alloy, Chemistry and Materials Researchwww.iiste.org ISSN 2224-3224 (Print) ISSN 2225-0956 (Online) Vol.6 No.6, 2014

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