Kuzmanov et al., J Nanomater Mol Nanotechnol 2019, 8:3

Journal of Nanomaterials & Molecular Nanotechnology

Research Article

A SciTechnol Journal

Study of the Influence of Modification by Nanocompositions both on the Process of Crystallization and on the Structure of Aluminum Alloy AlSi7Mg

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Abstract

A study of the cast alloy A356, modified by various types of nanoparticles has been carried out. SiC, AlN, TiN, clad by Cu, Ag and Al have been used. The cladding has been done by the following methods: currentless chemical method, extrusion of a composite rod, tableting and mechanical-chemical treatment in a planetary mill. The obtained nanocompositions (NCs) have been introduced into the crucible of the furnace. Homogenization has been conducted after that by using an impeller. The samples have been cast in thin-walled steel containers. The non-stationary temperature has been measured during cooling and crystallization. Data about the dependencies of the temperature on time have been obtained and the magnitude of overcooling for the cases with and without NCs has been determined. It has been established for the samples with NCs, decreasing of the overcooling and grain refinement, with an average diameter of the α-grains decreasing from 21% to 60%. For the sample, modified by NC, SDAS (Secondary Dendrite Arms Spacing) decreases by about 14%, while the micro-hardness increases by 7.7%, compared to the unmodified sample. These studies reveal new information about the influence of the NCs on the process of crystallization of A356 alloy.

Keywords

Nanocompositions; Crystallization; Aluminum alloy; AlSi7Mg

Introduction

The combination of thermal and structural research allows for an overall study of the interaction between various types of NCs and casting of an aluminum alloy, on the one hand, as well as of the process of crystallization of the alloy in their presence. Paper [1] considers the results from thermal and metallographic analyses of AlSi7Mg alloy before and after modifying it by two types of nanopowders (NPs)-nanodiamond (ND)+Ag (0.1 wt) and TiN+Al (0.03%-0.3 wt%). By means of the thermal analysis, the temperature changes during the different stages of crystallization and the values and the duration of overcooling in the result of the modification have been registered. The metallographic analysis has established that the modification by NCs leads to a significant presence of small-sized α-grains with cellular structure and to grain refinement of the silicon and intermetallic crystals. The average grain diameter decreases by 26%-28%, DAS values-up to 13%, while the micro-hardness increases by up to 15%. The influence of the cooling rate on both the process of solidification and on the properties of AlSi and AlMg alloys has been studied in [2]. From the cooling curves, obtained by means of an especially developed and constructed equipment, the cooling rate and the solid phase content in the biphasic zone for the different types of alloys have been defined, whereas improvement of the microstructure has been observed at high cooling rates. Paper [3] studies by thermal analysis the effect of the different amount of the classical modifier Al-5Ti-1B on both macro-and microstructure characteristics and the characteristic parameters of the cooling curve of aluminum alloy 319. Acceleration of the crystallization as a result of the introduction of the modifier has been observed and it has been established that the parameters of the process of solidification depend on the grain refinement. The influence of 0,2 wt% Ti and 0,03 wt% Sr on the temperature curves of cooling, as well as on the macro-and micro-structure of alloy A356 has been discussed in [4]. Changes in the character of the curves have been observed alongside macro-grains refinement as a result of introducing the additive.

The present paper describes a methodology, developed by the research team, together with a constructed experimental equipment for casting and thermal analysis of small samples of AlSi7Mg, modified by NCs. The way in which modification by nanoparticles influences both the parameters of the crystallization process and the structure of the alloy has been established.

Experimental studies

Casting alloy AlSi7Mg with the following composition: 6.65 wt% Si; 0.49 wt% Fe; 0.05 wt% Cu; 0,28 wt% Mg; 0.07 wt% Zn, determined by spectral analysis, was modified by NCs, based on nanopowders (NPs): SiC, TiN, TiCN and AlN. The metal cladding was carried out to improve the wetting of the nanoparticles by the metal melt. Different methods of cladding were applied.

By the method of mechanical-chemical treatment of the NPs in a planetary mill [5], the first type of NCs: 1 part TiN+1,5 parts Cu was obtained. For this purpose, the nanosized powder was mixed with micronized Cu particles. Thus, the metallization of the nanoparticle surface was accomplished.

Cladding by a currentless chemical method was used for obtaining the second type of NCs: SiC+Cu and SiC+Ag [6].

NC, made of: 4 parts AlN+1 part Cu+12 parts Al was obtained by the method of extrusion, developed by the authors. In this case, a mixture of nanoparticles and micronized aluminum and copper particles were homogenized, and then placed in a thin-walled container, made of pure aluminum, and extruded through a suitable nozzle.

By pressing a mixture of nanoparticles together with micronized Al particles briquettes of NC were obtained in a ratio of 1part SiC+4 parts Al.

A stainless steel thin-walled mould, equipped with an axial

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Received: June 25, 2019 **Accepted:** July 18, 2019 **Published:** July 26, 2019

fast-acting mantle thermocouple TCMI-K1x100G+3 m 800°C with hot solder SS316-type K, outer diameter of the working part 1 mm and length-100 mm (Figure 1a), was designed to cast the samples (Figure 1b). The thermocouple was placed in a thin-walled steel tube to protect it from the influence of the aluminum melt. The whole process was recorded by using a two-channel fast-running USB FLASH ARCHIVER MS DL-F2 v-2.0 produced by the Bulgarian company Mycrosist and enabling information at a time interval of 0.2 seconds about the temperature of the sample as a function of time. The melting of the aluminum alloy AlSi7Mg was carried out in an 11 kW electric resistance furnace, having a crucible with 1.0 kg capacity, mounted in it. The sequence of the experimental steps was as follows:

Melting the Aluminum Alloy

1. Degassing the melt at 730-740°C for 1 to 3 minutes by introducing a "Degasser" tablet, produced by "SM Region". A degassing method by purging with argon was also used
2. Packing the NC in an aluminum container; attaching it to the impeller; and introducing the impeller into the melt
3. Mechanical stirring for 3-5 minutes to melt the container and homogenize the melt at a rotational speed of about 150 revolutions per minute and melt temperature of 720-740°C
4. Immersing the mould into the melt, filling it, removing it from the furnace and fixing it by a stand until the melt is completely crystallized

A moment from the process of homogenization is shown in (Figure 2a). The temperature of the melt decreases in result of the homogenization and this imposes that reheating is carried out to reach the casting temperature of 720°C. The casting of a sample is done by immersing the mould and scooping up melt from the crucible. All experiments were conducted at close temperatures of the melt. For observing these conditions the melt temperature was continuously controlled by a corundum-protected thermocouple, dipped in the crucible (Figure 2b), the signal of which was visualized on the screen of the archiver. The mould, filled with crystallized alloy AlSi7Mg is shown in Figure 2c.

Results and Discussion

Temperature dependencies

Upon completion of each experiment, an array of data about the temperatures of both the melt and the sample were obtained and graphics were built with the dependences of the temperature on time during the crystallization. Figure 3 illustrates the dependencies T(t), obtained during the conducted experiments for the samples: a) without NC; b) with NC: 1 part TiN+1.5 parts Cu, at a concentration of nanoparticles 0.05 wt% TiN. Similar dependencies have been derived for all experiments. The tabular data about the temperatures, obtained during the experiments, serve for determining certain quantities, from which conclusions can be drawn about the process of crystallization after introducing NCs. These are: T_0-initial melt temperature starting temperature of the melt; T_L-liquidus temperature; $DT_L=T_L-T^L_{min}$-overcooling at the temperature of the liquidus; T_e-eutectic temperature; DT_e-overcooling at the temperature of the eutectic. A scheme for determining the quantities is shown in Figure 3c.

The modification by NCs does not influence the temperatures of both the liquidus and the eutectic of the samples decrease in the overcooling for both the eutectic and the liquidus of the samples has been established. The overcooling at the liquidus decreases from 3.51 for the unmodified sample to 0.2-2.74 for the modified ones. For the samples S6N, S9N and S10N, modified by NCs, containing TiN and SiC, the overcooling at both the eutectic and the liquidus is below 1°C. The obtained results confirm the influence of all modifying NCs, used in the present study, on the parameters of aluminum alloys crystallization (Table 1).

Macrostructural studies

For capturing and analyzing the macrostructure the samples were prepared according to a standard procedure ground by sandpaper numbers 120, 220, 400 and 600, and etched with a Poulton reagent. The macro-samples were photographed, using a Canon Power Shot G7 digital camera, together with part of a measurement line for scaling and quantitative analysis. The quantitative analysis was done using a licensed image analysis software Olympus Micro Image. The average diameter values of the macro-grains were defined (average diameter-the average length of the diameters, measured at 2-degree intervals and going through the center of the object).

The results about the average diameter of the macro-grains, obtained during the conducted quantitative analysis of AlSi7Mg alloy samples made of AlSi7Mg alloy are given in Table 2. Sample S2-1 is taken as a base for calculating the percentage change of the average diameters in D_{ave}. It can be seen that the average diameter of the macro-grains for all modified samples decreases between 21% and 60% in comparison with the unmodified samples, which proves the repeatability of the effect of nanomodification. The largest grain refinement of the structure, from 49.6% to 60.4%, was obtained with the S6N-1, S7N-1 and S8N-1 samples modified by NCs, containing correspondingly TiN, SiC and TiCN. This gives reasons to consider that the NPs, specified here, are the strongest refiners of the AlSi7Mg alloy macrostructure.

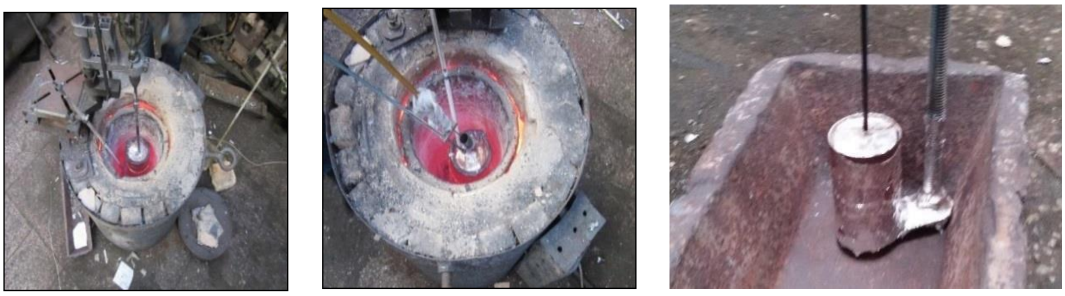
Microstructural studies

The modifying effect of NC: 1part TiCN+2 parts Ti, on the microstructure of the AlSi7Mg alloy was studied as well. Micro sections were prepared from the samples S2-1 and S8N-1. Their microstructure was photographed at a magnification of x50 (3 fields for computing SDAS) and of x200-for qualitative assessment of the eutectic. The micro-images are shown in Figure 4.

Table 3 gives the result of SDAS and the micro-hardness of the same samples. It can be seen that for the modified by NC sample

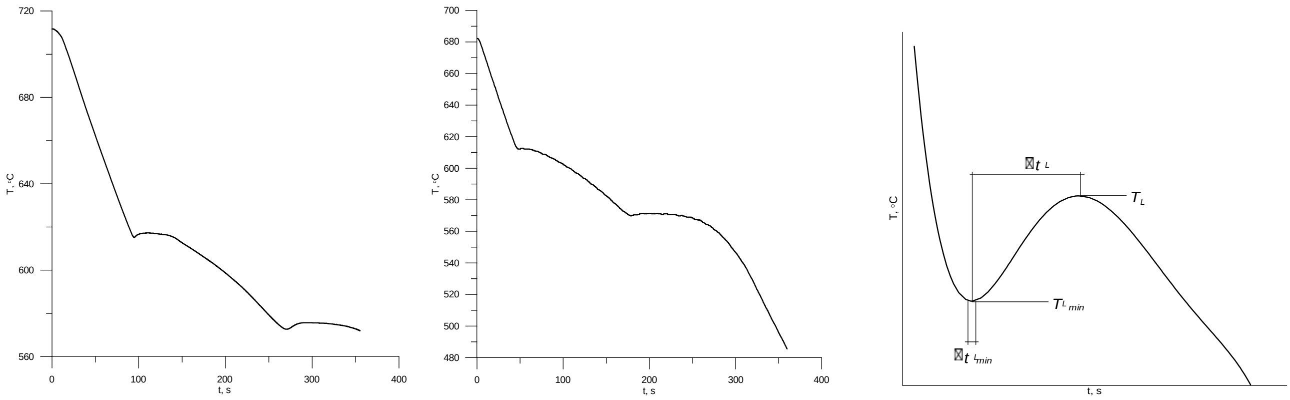
(a) **(b)**

Figure 1: (a): Mould with axial thermocouple; (b): General appearance of a mantle thermocouple.



(a) (b) (c)

Figure 2: (a): Homogenization of the melt; (b): Taking a sample; (c): A cast sample together with the mould.



(a) (b) (c)

Figure 3: (a): Dependence of the temperature on time for sample S1 without NC; (b): Dependence of the temperature on time for sample S6N with NC 0.05 wt% TiN; (c): Scheme for determining the quantities of the temperature dependencies.

Table 1: Crystallization parameters of AlSi7Mg alloy, modified with NCs.

No of the sample	Type of NC	T_0, °C	DT_L, °C	T_L, °C	DT_e, °C	T_e, °C
S2	Without NC	721	3.51	615	4.2	571.9
S2N	Currentless cladding, SiC+Cu, 0.03 wt% SiC	713.6	2.74	618.9	2.2	576
S5N	Extrusion, 4 parts AlN+1part Cu+12ч.Al, 0.05 wt% AlN	685	2.4	614.1	1.4	571.1
S6N	Planetary mill, 1part TiN+1.5parts Cu, 0.05 wt% TiN	682	0.5	612.7	0.7	570.4
S9N	Tableting 1part SiC+4parts Al, 0.04 wt% SiC	676	1.0	614.8	0.9	573.2
S10N	Currentless cladding, SiC+Ag, 0.03 wt% SiC	675	0.2	613.8	1.0	574.2

Table 2: Results about the average grain diameter in samples of AlSi7Mg alloy.

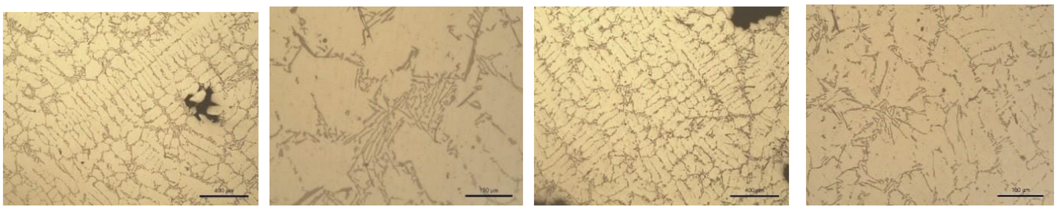
Sample No	NC used	Dave, mm	Change in Dave, %
S2-1	Without NC	2,812	-
S2N-1	Currentless cladding, 1 part SiC+4 parts Cu, 0.03 wt% SiC	2,206	-21,6
S5N-1	Extrusion, 4 parts AlN+1Cu+12 parts Al, 0.05 wt% AlN	2,034	-27,7
S6N-1	Planetary mill, 1 part TiN+1,5 parts Cu, 0.05 wt% TiN	1,416	-49,6
S7N-1	Extrusion, 4 parts SiC+1 part Cu+12 parts Al, 0.05 wt% AlN	1,2	-57,3
S8N-1	Planetary mill, 1 part TiCN+2 parts Ti, 0.04 wt% TiCN	1,114	-60,4
S9N-1	Tableting, 1 part SiC+4 parts Al, 0.04 wt% SiC	1,71	-39,2
S10N	Currentless cladding, SiC+Ag, 0.03 wt% SiC	1,83	-34,9

Table 3: Results about SDAS and the micro-hardness.

Sample No	SDAS, µm	Change in SDAS, %	Micro-hardness, MPa	Change in the micro-hardness, %
S2-1	54,83	-	727.26	-
S8N-1	47,17	-13,97	783.16	+7,7

(a) **(b)** **(c)** **(d)**

Figure 4: Macrostructures of samples-without and with NC (a): S2-1-without NC; (b): S6N-1-1part TiN+1.5 parts Cu, 0.05 wt% TiN; (c): S7N-1-composite rod: 4ч.SiC+1ч.Cu+12ч.Al, 0.05 wt% SiC; (d): S8N-1-1 part TiCN+2 partsTi, 0.04 wt% TiCN.



(a) **(b)** **(c)** **(d)**

Figure 5: Microstructure of samples S2-1 and S8N-1 (a): general view of the microstructure of sample S2-1; (b): eutectic in sample S2-1; (c): general view of the microstructure of sample S8N-1; (d): eutectic in sample S8N-1.

SDAS decreases by about 14%, while the micro-hardness increases by 7.7% in comparison to the unmodified sample. The type of eutectic stays the same (Figure 5b and 5d).

Conclusion

Studies of modification of aluminum alloy AlSi7Mg by different (both by composition and by the method of the cladding of the powders) NCs have been carried out. The used NCs are: (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). It was established for the samples with NC, in comparison to the ones, without NC, that the overcooling decreases both at liquidus and eutectic, grains refine, SDAS decreases, and micro-hardness increases.

Acknowledgement

The work is supported by a project under contract No DN07/20/15.12.2016, funded by the Scientific Research Fund.

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