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Investigation the Effect of Modification with Nanopowders on Crystallization Process and Microstructure of some Alloys

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Abstract. Modified with nano-powders (NP), AlSi7Mg aluminum alloy, P265GH steel and GG25 gray cast iron, have been investigated. Thermal and metallographic analyses have been made. For modified AlSi7Mg alloy, reduction of overcooling and duration of crystallization at the initial crystallization and their increase at eutectic crystallization have been found. For cast iron GG25, reduction of overcooling at crystallization was established and for P265GH steel, overcooling was not recorded, only a change in the slope of the temperature dependence. The thermal effects obtained in the crystallization correspond to the refinement of micro- and macrostructures. A mathematical model for crystallization of samples for thermal analysis has been developed and solved.

INTRODUCTION

The method for introducing into metal melts of small amounts of nano-powders (NP) of refractory compounds (nitrides, carbides, borides, etc.) specially treated with cladding metals, represents an alternative to the classical modification. In the works [1, 2], studies have been carried out on the impact of NP on the structure and properties of metal alloys. It is achieved a grain refinement, a change in the morphology of the crystalline structure and the improvement of the mechanical and exploitation properties of the alloys and the castings made of them.

In the present work aluminum AlSi7Mg alloy, P265GH steel and GG25 cast iron have been modified with modifying compositions from NP. Aimed at a more profound investigation of the crystallization process, there have been developed methods and designed devices for simultaneous investigations of “temperature-time” dependence during the crystallization process and the microstructure of the obtained experimental samples.

USED METHODOLOGIES

Methodology for Thermal Analysis

AlSi7Mg

The thermal investigations have been carried out with a device (Fig. 1) designed by us and containing the following basic elements: a container for casting and crystallization of the sample, a thermocouple arranged along the axis of the cylindrical container, and a device for archiving of the temperature data. The thermocouple is with Ni-CrNi electrodes of 0.25 mm diameter and a non-protected hot junction. The thermocouple's electrodes are

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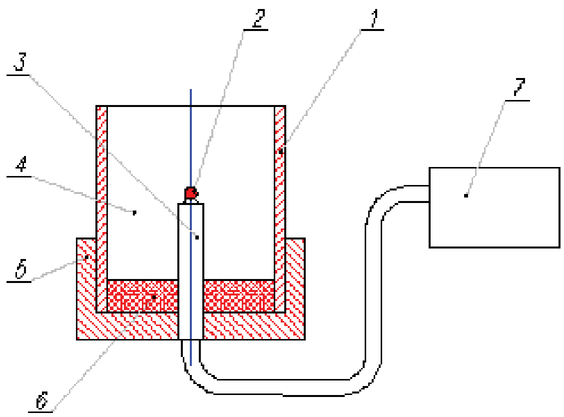
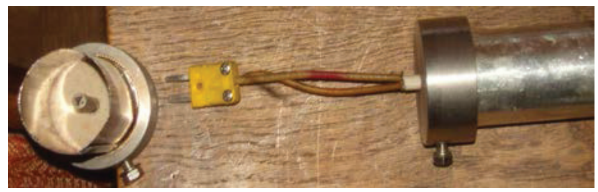
protected from contact with the melt through their insertion into a ceramic two-channel pipe. A thermo-insulating pad of coated sand is formed on the bottom. Thus, are provided conditions for the heat transfer of the melt, only through the walls of the container and through the upper free surface.





(a) (b)

FIGURE 1. Device for thermal analysis: (a) *1*-container, *2*-thermocouple, *3*-ceramic tube, *4*-specimen, *5*- bottom plate, *6*-thermo-insulating pad; (b) container with thermocouple

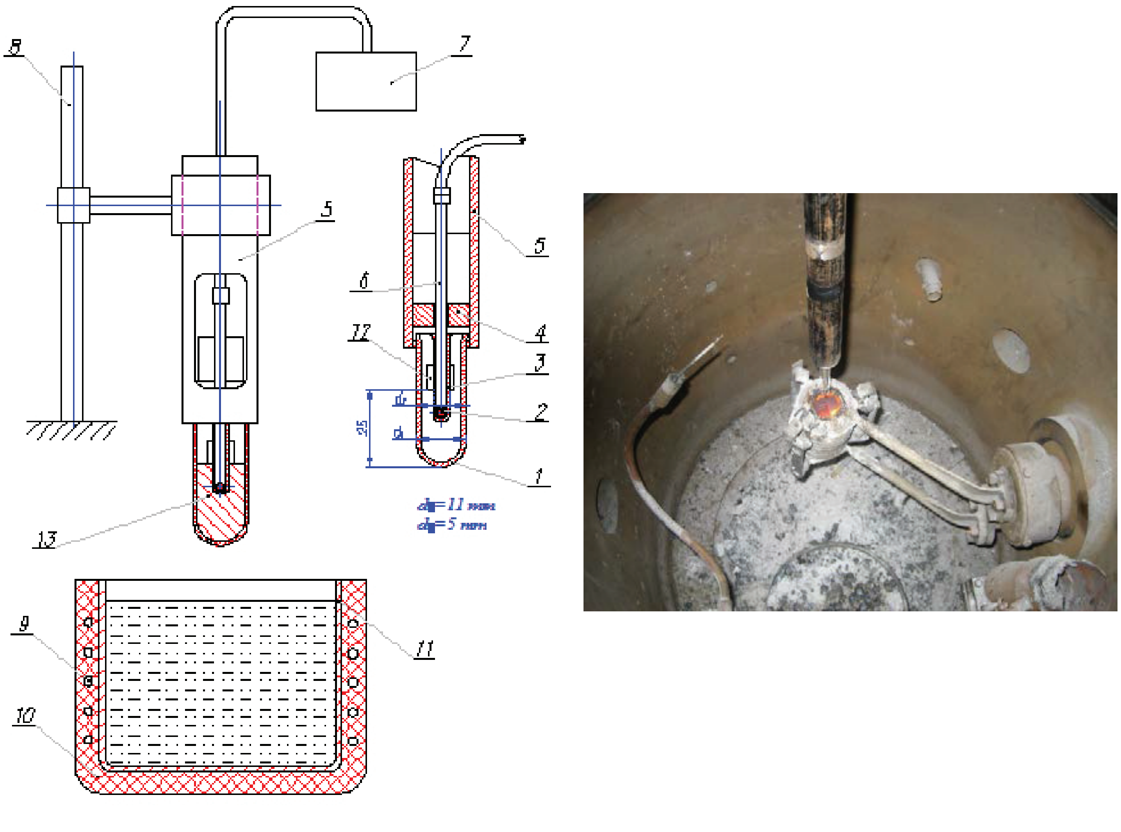
The methodology for thermal analysis includes the following steps:

1. Melting of the investigated aluminum alloy in a crucible located in the working space of the electric resistance furnace;
2. Blowing of the melt with Ar;
3. Measurement of the temperature of the melt;
4. Casting of the sample without NP by charging of a pre-heated foundry spoon with a required quantity from the melt;
5. Introduction of NP into the melt (0.05% AlN, cladded with Al) by the help of an aluminum cartridge;
6. Stirring for homogenization of the melt, by means of an agitator made of Ti alloy;
7. Casting of samples with NP;
8. Recording the temperature of the sample until its full crystallization.

P265GH Steel and GG25 Cast Iron

For investigation of the dependence "temperature-time" during the process of crystallization of P265GH steel and GG25 cast iron, have been developed a method and the device (shown in Fig. 2). The investigations have been conducted in Laybold Heraeus induction furnace. The sequence of the experiment is:

1. Melting the studied alloy;
2. Measuring the temperature of the molten metal;
3. Take a sample without NP by immersing the sampling unit (sampler) in the furnace at a depth sufficient to fill it;
4. Returning of the sampler to a starting position;
5. Recording the sample temperature from the moment of sampling to full crystallization.
6. Mounting of a new sampler.
7. Introduction into melt, with the help of a thin-walled steel cartridge, of 0.3 wt. % mixture of NP: TiCN (2 parts). + Y_2O_3 (1 part), cladded with Cr (1 part) + Fe (6 parts).
8. Homogenization of the melt for 1 minute, using the mixing effect resulting from the operation of the induction furnace.
9. Taking a sample with the NP by immersing the sampler in the furnace.
10. Returning the sampler to the starting position.
11. Recording the temperature of the sample.



(a) (b)

FIGURE 2. (a) Scheme of the sampler with Laybold Heraeus induction furnace: Sampling unit (1), Thermocouple PtPtRh (2); Quartz tube (3); Centering ring (4), Carrier (5), Ceramic tube (6), Device for archiving the temperature data (7), Metal stand (8), Inductor (9), Thermally insulating padding (10), Template (11), Opening for sampling (12), Sample of liquid metal (13); (b) A moment of sampling from the Laybold Heraeus furnace, using the device

The melts have been carried out under the following conditions:

1. Weight of molten metal in Laybold Heraeus furnace – 270 g or 420 g;
2. Duration of the melt, including melting of the charge, melting of the ferroalloys, homogenization of the molten metal and temperature measurement: 15-20 minutes (for the crucible of 270 g) and 20-30 minutes (for the crucible of 420 g);
3. Time for melting of the container with the nanomodifier -15 s;
4. Holding for homogenization, after introduction of the nanomodifier into the melt - 1min.

Methodology for Macro- and Microstructural Investigation of Samples of AlSi7Mg Alloy, GG25 Cast Iron and P265GH Steel

The preparation of the samples for metallographic analysis is carried out according to a standard procedure. The samples have been wet-grinded and mechanically polished. AlSi7Mg alloy samples have been treated with 0.5% aqueous solution of HF and those of GG25 cast iron and P265GH steel with 4% solution of nitric acid in ethyl alcohol. The metallographic analyzes have been made by the help of optical microscopes Reichert MeF2 and PolyvarMet at magnifications up to 1000 x. The quantitative assessment of the microstructure is made using system for quantitative analysis of the images – Olympus MicroImage. The average diameter of the grains and the parameters of the microstructure have been determined for all alloys.

EXPERIMENTAL RESULTS

AlSi7Mg Alloy

Figure 3 presents the results of experiments for determination the temperature dependencies of the samples without and with 0.05% AlN + Al. For the sample without NP, overcooling rate is $\Delta T = 1°C$, and with NP $\Delta T = 0°C$. The temperature of releasing of hidden heat of crystallization is 615-616°C and the duration of releasing $t = 9$ s. No overcooling is found for the sample with NP but only a change in the slope of the temperature curve in the interval [613°C; 600°C]. For the sample without NP there is no overcooling at the eutectic temperature. In the case of added NP the overcooling rate is $\Delta T = 40°C$ and $t = 41$ s.

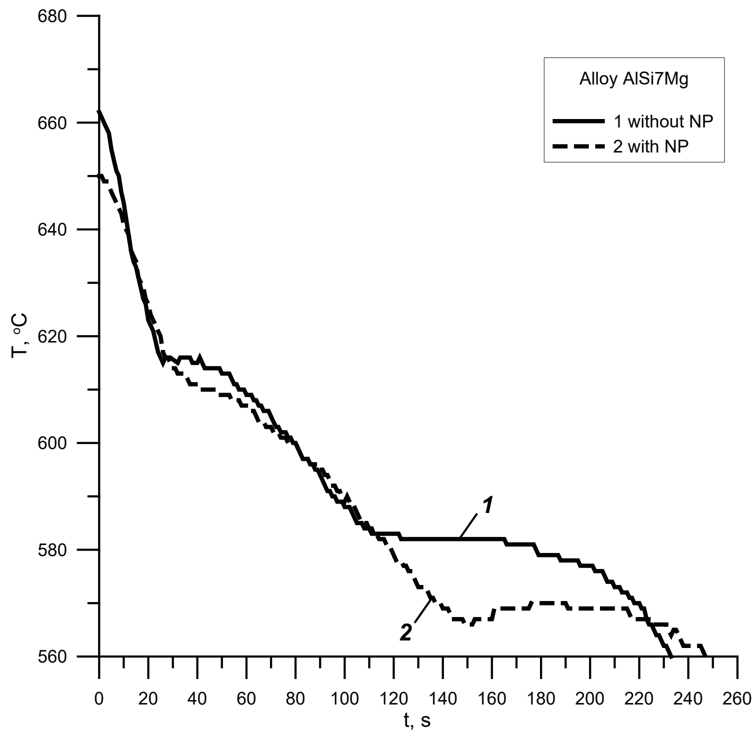


FIGURE 3: Temperature dependencies for alloy AlSi7Mg

Macro-structures of unmodified - (a) and modified with 0.05% AlN + Al - (b) samples are shown in Fig. 4. By macrostructure analysis, the average grain size is determined: without NP – 1.44 mm, with NP – 0.62 mm. It is achieved grain refinement of 57%.

(a) (b)

FIGURE 4. Macrostructure of samples of alloy AlSi7Mg: (a) - without NP; (b) - with NP

The microstructure study shows that the adding of NP leads to a change in the shape of α-grains from dendritic to cellular, and the volume part of the spherical α-grains in the refining casts increase almost twice.

Cast Iron GG25

The obtained temperature dependencies are shown in Fig. 5. For the sample without NP the overcooling rate is ΔT = 20°C, the temperature interval of releasing of hidden heat of crystallization is [1156°C; 1136°C] and the duration of releasing t = 5 s. No overcooling is found for the sample with NP but only a change of the slope of the temperature curve in the interval [1112°C; 1096°C].

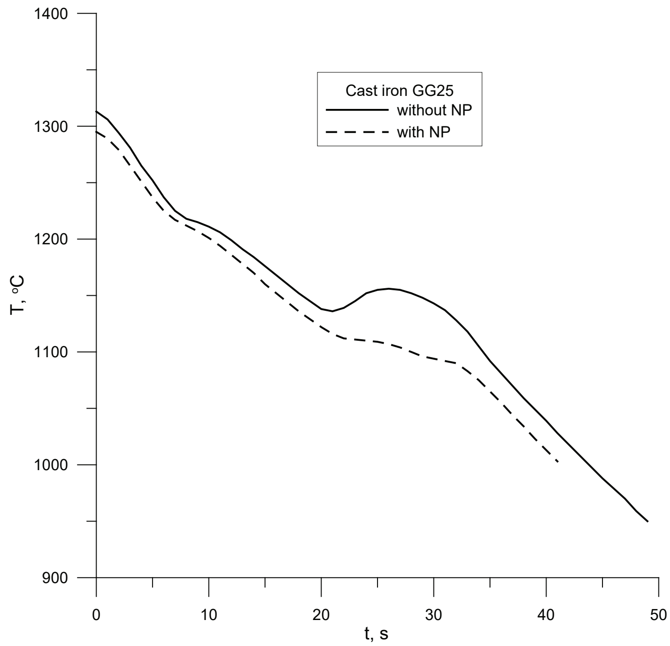
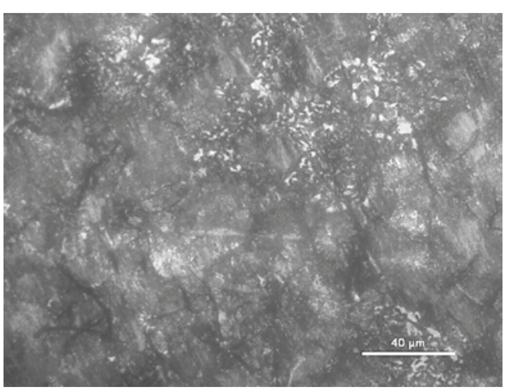
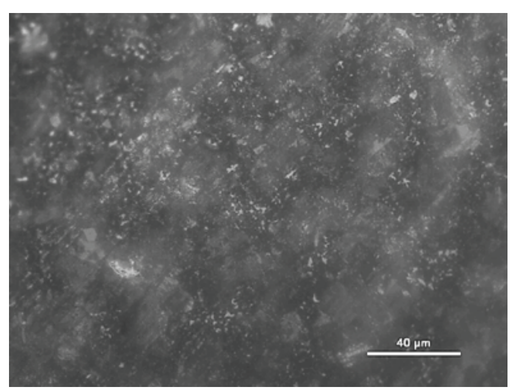


FIGURE 5. Temperature-time dependencies for cast iron GG25 samples

Microstructures of samples without NP – c) and with NP – d) are shown in Fig. 6. The average diameter of the grain in the sample without NP is 22.4 μm, whereas with NP is 9.8 μm, i.e. 56% smaller. The average length of the graphite inclusions is also measured: for the sample without NP is 3.6 μm and for the sample with NP is 1.4 μm, i.e. graphite inclusions refinement is 61%.



(a)



(b)

FIGURE 6. Microstructure of cast iron GG25 samples, respectively: (a) - without NP, (b) - with NP

Steel P265GH

The temperature dependencies are shown in Fig. 7. In this case no overheating was registered but only a change in the slope of the temperature curves in the intervals: for the unmodified sample [1477°; 1436°C] and for the sample with NP [1503°C; 1436°C].

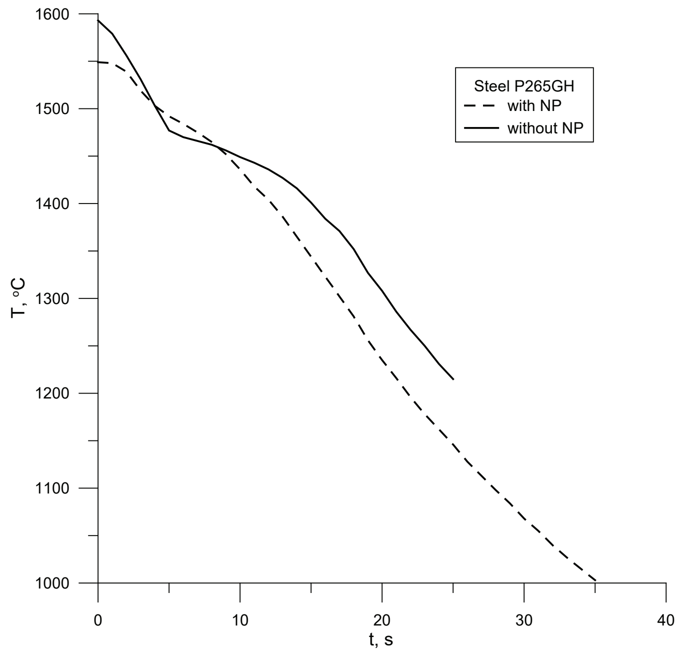
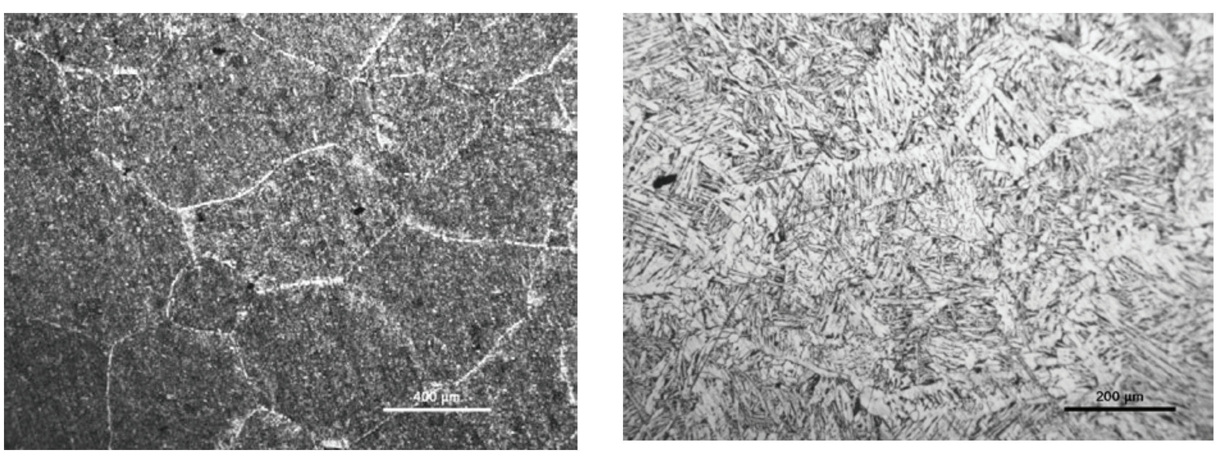


FIGURE 7. Temperature-time dependencies for samples of steel P265GH

Microstructures of steel P265GH samples are shown in Fig. 8: a) without NP and b) with NP in an amount providing the introduction of 0.03wt% TiCN in the melt. Both samples have ferrite-pearlite microstructure with separation of vanilla ferrite at the grain boundaries. The only significant difference that is observed is in the grain size. In the sample without NP, the average grain diameter is 482μm and in the sample with NP - 349μm. Refinement due to the introduction of NP is 27.6%.



(a) (b)

FIGURE 8. Microstructure of steel P265GH samples: (a) – without NP, (b) – with NP

MATHEMATICAL MODELING OF CRYSTALLIZATION OF SAMPLES FOR THERMAL ANALYSIS

A mathematical model of the crystallization of AlSi7Mg alloy is created and solved. This model is shown below.

$$\begin{cases} c(u)\rho(u)\dfrac{\partial u}{\partial t}=\dfrac{1}{r}\dfrac{\partial}{\partial r}\left(r\lambda(u)\dfrac{\partial u}{\partial r}\right)+\dfrac{\partial}{\partial z}\left(\lambda(u)\dfrac{\partial u}{\partial z}\right), r\in(0;R), z\in(0;H), t\in(0;T_0], \\ u(r,z,0)=u_0(r,z), r\in[0;R], z\in[0;H], \\ r\lambda(u)\left.\dfrac{\partial u}{\partial r}\right|_{r=0}=0, z\in[0;H], \\ \lambda(u)\left.\dfrac{\partial u}{\partial r}\right|_{r=R}=-\alpha\left(\left.u\right|_{r=R}-u_{amb.}\right), z\in[0;H], \\ \lambda(u)\left.\dfrac{\partial u}{\partial z}\right|_{z=0}=\tilde{\alpha}\left(\left.u\right|_{z=0}-u_{amb.}\right), r\in[0;R], \\ \lambda(u)\left.\dfrac{\partial u}{\partial z}\right|_{z=H}=-\bar{\alpha}\left(\left.u\right|_{z=H}-u_{amb.}\right), r\in[0;R], \\ \lambda(u)\left.\dfrac{\partial u}{\partial z}\right|_{z=h-0}=\lambda(u)\left.\dfrac{\partial u}{\partial z}\right|_{z=h+0}=\alpha_k\left(\left.u\right|_{z=h+0}-\left.u\right|_{z=h-0}\right). \end{cases} \tag{1}$$

The physical parameters $\rho(u)$ and $\lambda(u)$ we consider as constants and define in the next manner:

$$\rho(u),\lambda(u)=\begin{cases}\rho_1,\lambda_1 \text{ for the sand}\\ \rho_2,\lambda_2 \text{ for the metal alloy.}\end{cases} \tag{2}$$

Depending on where the temperature value is located regarding the liquidus temperature u_L and the solidus temperature u_S at a fixed moment of time, the heat capacity coefficient $c(u)$ is defined as

$$c(u)=\begin{cases} c_{\text{liquid}}, & u>u_L \\ c_{\text{solid}}-L\psi'(u), & u_S\le u\le u_L \\ c_{\text{solid}}, & u<u_S \end{cases} \tag{3}$$

Here c_{liquid} and c_{solid} denote the heat capacity of the alloy in the liquid and the solid state, respectively.

The function $\psi(u)$ is different for each concrete alloy and can be written in explicit form using the equilibrium diagram. It is continuous piecewise polynomial function. Here we consider it to be fractional-linear.

The mathematical model (1) at the additional assumptions (2), (3) is solved numerically via fully implicit locally one-dimensional difference scheme with local truncation error of order $O(h^2+\tau)$ [3], [4]. The values of the physical parameters and grid steps used for all calculations are: R = 0.017 *m*, H = 0.061 *m*, h = 0.01 *m*, c_1 = 1,08.10^3 *J/(kg.°C)*, c_2 = 1.05.10^3 *J/(kg.°C)*, λ_1 = 1,28 *W/(m.°C)*, λ_2 = 104 *W/(m.°C)*, ρ_1 = 1,65.10^3 *kg/m*^{3}, ρ_2 = 2.45.10^3 *kg/m*^{3}, u_L = 619°*C*, u_S = 580°*C*, $u_{amb.}$ = 20°*C*, u_0 = 665°*C*, $\alpha = \tilde{\alpha} = \bar{\alpha}$ = 90 *W/(m*^{2}*.°C)*, и α_k = 10^4 *W/(m*^{2}*.°C)*, h_r = 0,004, h_z = 0,00025, τ = 1. The model is verified in the following way: a thermo pair measuring the levels of the temperature for a period of 150 seconds is located in the point (r; z) = (0; 0.025), along the cylinder axis. So obtained experimental data set is validated against the numerical results extracted after solving the difference scheme. The comparison is plotted in Fig. 9. The relative error of the exact values versus the approximation is 0.7786%. This fact confirms the good coincidence between the theoretical estimation and experiment.

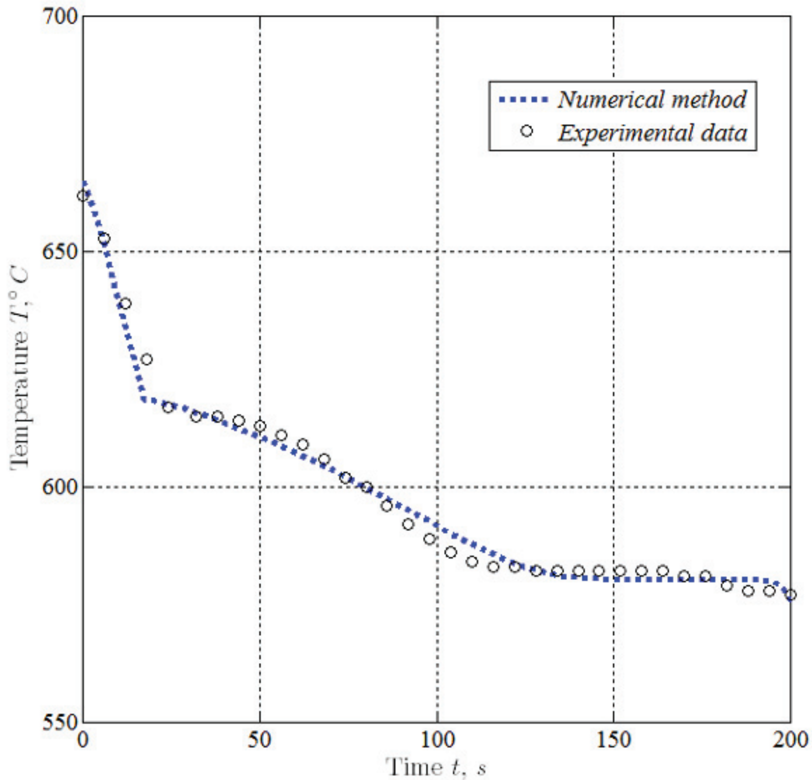


FIGURE 9. Validation of the numerical results against experimental data set

CONCLUSION

Methodologies and apparatuses are developed in order to study the crystallization process of various alloys with introduced nanoparticles. Information on temperature-time dependencies and microstructure parameters are obtained. The numerical solution of the mathematical model is compared against experimental data.

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