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Method of thermal derivative gradient analysis (TDGA)

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Abstract

In this work a concept of thermal analysis was shown, using for crystallization kinetics description the temperature derivatives after time and direction. Method of thermal derivative gradient analysis (TDGA) is assigned for alloys and metals investigation as well as cast composites in range of solidification. The construction and operation characteristics were presented for the test stand including processing modules and probes together with thermocouples location. Authors presented examples of results interpretation for AlSi11 alloy castings with diversified wall thickness and at different pouring temperature.

Keywords: Solidification, Crystallization, Thermal derivative gradient analysis,

1. Introduction

The premise of the topic was to specify the information about heat flow kinetics during the liquid – solid phase transformation in relation to cast alloys and composites. The growing requirements for high quality castings are connected with a search for lower tolerance of shape, dimensions, chemical composition, structural characteristics and mechanical properties. In addition, there is a clear tendency to minimize the wall thickness of castings including super thin-wall castings. Particularly difficult cases to assess crystallization kinetics of thin-wall castings are composites with significantly diversified, in terms thermal properties, components and/or the metallic compounds of the matrix.

The method was developed based on the classical method of TDA $[1 \div 5]$ and based on the virtual and real experiments. The most important factor was to solve the problem concerned the geometry of casting probe. Multipoint temperature measurement allowing the designation of the thermal gradient can be achieved in many ways based on a single or multiple test castings of similar or different shapes - cylindrical, spherical wedge, etc. $[6 \div 8]$.

Selection of casting geometry should provide the required sensitivity of measurements - the measurements of temperature in the following points should give a sufficiently diverse results, to significantly exceed the accuracy of measurement of used thermocouples. Presented results are related to castings manufactured in sand moulds.

2. Method description

Cone shape probe was used with thermocouples situated outside the heat axis. The measuring points were placed on conventional side surface formed by rotation of angle bisector $\alpha/2$ of the cone probe and belong to the spatial curve of the spiral beginning at the apex of the cone. Casting geometry and thermocouples placement was shown in fig.1. Such shape was selected to designate the structural characteristics of castings with varying wall thickness. Temperature dependence of microstructure was provided by testing two castings in each experiment and assuming drop in pouring temperature.

Three variables such as: both temperature derivatives and the primary quantity – the temperature, should be considered as a function of time and direction of solidification. Concept of the analysis was based on three fundamental assumptions:



Fig. 1. Casting geometry and thermocouples placement

- 1. Crystallization kinetics evaluation for sample casting including varying of wall thickness and their mutual interaction with the riser.
- 2. Measurement of temperature located half-way between the heat axis and the wall of the mould.
- 3. A detailed analysis of the impact of pouring temperature by pouring following two castings at a minimum time interval for one melt.

The first two resulted from the desire to include cooperating parts of the casting characterized by different solidification modulus. For one-point measurement a single thermocouple is placed on the heat axis, preferably in the heat center which changes its position along the vertical axis as a function of time and is different for the isotherms of crystallization temperature and in the vicinity of the range. In this case, the location of thermocouple is fraught with a certain approximation. The low volume of approved sample casting - with a compact construction corresponds to the actual solidification modulus occurring in real state. The central location of a thermocouple exposes the measurement to errors related to the possible shrinkage and other discontinuities. Assuming the volatility of the heat center position in the range of crystallization, the temperature measurement is carried out with the assumption in the approximate center of heat. Hence it is assumed that the analysis of solidification kinetics should not consider the specific case, which is the heat center, but the regions placed between the heat axis and the mould wall. The third assumption is important mainly from technology point of view – the aim is to set quantitative differences in solidification kinetics caused by different pouring temperature. This is a basic, adjustable quantity in casting alloys technology - especially important in the rheology of composite liquid dispersion. Experimental studies and omitted in this work numerical simulations concern rate of crystallization of AlSi11 castings in range of: $0.12 \div 1.2$ [K / s], which corresponds to the thickness of casting wall $1.5 \div 45$ [mm]. The purpose of the preliminary numerical analysis was to verify thermal and geometric conditions of the real experiment and the analysis of isoliquidus and isosolidus surface movement in the volume of solidifying casting.

A preliminary verification of the method and the stand was conducted together with structural and mechanical studies of obtained sample castings [9] These results led to the final version of the method and the geometry of the probe.

3. Test stand

A multichannel analog-digital converter was designed and prepared [10]. Transformation of collected data was conducted with use of prepared software, which was an integral part of the test stand.

The test stand (fig. 2) consists of two probes and two modules for analogue signal processing, power supply, the control and registration module.



The system is protected against electromagnetic interference, thermal and power errors. 8 channel modules enable the implementation of measurements in both probes with 4 channels for additional measurements. This is an adaptive system - for the purpose of research, you can increase the number of measurement channels that are not multiple of eight channel modules, while maintaining the same accuracy of measurement. Fig. 3 shows multi-measurement module used for the analysis of solidification kinetics of test castings.



Fig. 3. A single multi-measurement module

Eight-channel thermoelectric measurement module TEMP8 allows you to measure the voltage from 15mV to 120mV and it is possible to install different thermocouples.

Modules and measurement channels are galvanically isolated, but each entry are not separated from each other. The modules communicate with the computer supervising the work with twoway bus standard RS422/RS485. It permits the connection of modules separated by up to a distance of 100-500m depending on the set of data transfer speed. For proper communication with the computer using a standard signal converter RS422 to RS232 and USB. The converter is built-in a transducer of power supply. Its correct operation is indicated by the controls.

The principle of communication with the modules is as follows:

- None of the modules does not send information unless it is asked by the computer controlling the work.
- Question posed by the computer must have a specific number of the module,
- Responding module provides a number for the unique identification equivalent.

Condition for the proper functioning of greater than one is to set the number of modules for each individual caller. Each module has a microprocessor controlling the work. It is responsible for testing the performance of the card after power on, synchronization of the measurement, recording and reading the data from the conversion of the measurement systems in accordance with the calibrating settings and for communication with the system - a computer.

Commands can be sent and received through the MLab on the [communication port Diagnostics], or any terminal that supports the serial port. Each command setting parameters of the module will save the settings in the memory chip which controls the work of the module. As a result, even after turning off, the data set is permanently stored, after enabling the module set according to the changes. Data about the workplace are updated immediately after their introduction to the module. The only exception is the change in speed of data transmission. This change is introduced only after turning off power and re-activated.

Technical documentation was prepared as well as the moulding instrumentation for probes. Conclusions on methodology of measurement were formulated. Inference was based mainly on DAS evaluation of eutectic AlSi11 alloy. (This alloy is characterized by high thermal capacity heat of crystallization).

4. Verification of TDGA method and the test stand

Tests were carried out to verify the method for a model, eutectic, unmodified AlSi alloy [9]. Registered as the result of direct measurement of temperature as a function of time in different points of the sample casting. Subsequently, the system software created the temperature derivatives after time and direction in function of time and direction of heat flow - dT/dt, dT/dl = f(t, l). Examples of obtained relationships are shown in fig. 4.



Fig. 4. An example of relationships obtained as a direct result of the TDGA method a) temperature dependence as a function of time, T = f(t), b) temperature derivative after time as a function of time dT/dt = f(t), c) temperature derivative after direction - the vertical component as a function of time dT/dz = f(t); The next curves correspond to different levels of measurement points and thus casting with a different wall thickness

They provide a basis for the analysis of phase transition mainly, in particular, provide information on the crystallization of phases depending on the thickness of the casting wall, cooling rate and pouring temperature.

Based on this information, there is the possibility of determining the characteristics of microstructural and mechanical dependencies using the example of commonly used in commercial simulation programs. However, according to the methodology of verification DAS of α solution was determined and on this basis, according to the example describing the microstructure as a function of the rate of vertical component of the gradient change before the first crystallization effect was set (Fig. 5). For each cooling rate the diversity of microstructure is registered, resulting from pouring temperature $\Delta T_{zal} = 4 \,^{\circ}$ C. The maximum difference in temperature recorded during the whole cooling of the casting is $\Delta T_{max} = 6 \,^{\circ}$ C, with a delay for casting poured at lower temperature of $\Delta t = 4$ s.



Fig. 5. Examples of DAS relations as a function of the gradient rate $v_{Gv} = G_v/dT = \Delta t/(\Delta t dl) [K/(cm s)]$ at different pouring temperature $\Delta T_{zal} = 4^{\circ}C$, a) $T_{zal} = 718^{\circ}C$, b) $T_{zal} = 714^{\circ}C$

It should be noted that the rate of change of temperature gradient is directly associated with the pouring temperature. Following values on the abscissa axis are designated for variable wall thickness corresponding to thermocouples locations along the vertical axis of the test casting.

5. Summary and conclusions

Most studies of crystallization kinetics is based on two variables describing the structural and mechanical properties. These are the rate of crystallization determined as a temperature derivative after time and direction ratio and the thermal gradient. Presented studies enabled formulation of following conclusions:

- The validity of the assessment of microstructural properties with application of TDGA method was shown.
- It has been shown that minimal difference in pouring temperature resulted in microstructure diversity.
- Proposed methodology for the analysis, provided statistically significant results.
- The method may be an alternative to other methods of alloys diagnosis.
- The proper way of recording and processing the data was confirmed, offered by research stand, which main component was the multichannel system.
- Studies carried out to verified the fulfillment of all assumed technical requirements.

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