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Influence of thermo-derivative analysis conditions on microstructure of the Al-Si-Cu alloy

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Abstract
Microstructure change of the metals and alloys as a result of variable crystallisation conditions also by mind of cooling rate change influence the mechanical properties. In this work there are presented the interdependences between the cooling rate, chemical composition and microstructure of the cast aluminium alloy Al–Si–Cu as a result of the thermo-derivative analysis, using the UMSA (Universal Metallurgical Simulator and Analyzer) device. An important tool for the microstructure evaluation of the Al type AC-AlSi7Cu3Mg alloy was the light and electron scanning microscopy technique.

Keywords: Aluminium alloy, Thermo-derivative analysis, UMSA, Microstructure, Properties

1. Introduction

Properties and appliance of the cast alloys are connected to the primary alloy structure, which depends on the crystallization kinetics. The crystallization kinetics is characterized by changes of following parameters:

- metal temperature,
- solidification rate,
- cooling rate,
- emission rate of the hidden crystallization heat,
- density of nucleus formation,
- fraction slid of the crystallized metal,
- the concentration of the compounds in the remaining liquid,
- characteristic distances as well values describing shape and size of the structural compounds.

All this parameters are variables of the crystallisation time and geometric coordinates of the cast. A full characteristic of the crystallization kinetics is achieved after the unification of the crystallization equations with the heat transfer equations, and the emitted crystallization heat has the function of the link between these equations and depends on the fraction of the out crystallised structure compounds [1,2].

The solidification of the liquid alloy goes from the liquid state – the liquidus line, as the beginning of the crystallization process, after that follows the crystallization of the eutectics and intermetallic phases until the solid stated is achieved – the solidus line, according to the equilibrium diagrams [3].

For this reason, on the crystallization curve there are present some inflexion points coming from the exothermic or endothermic reactions of the transformations of the out crystallised phases and eutectics. It is difficult to determine unequivocally the phase crystallization temperature on the cooling curve. The determination makes it possible to interpret the first derivative of the cooling curve in relation to time, this first derivative is often known as the differential curve (ATD called also derivative curve.

On the basis of the literature data it can be found that the temperature holds present on the cooling curve are caused by heat

source of the occurred phase transitions. The crystallising metal is emitting the heat into the surrounding in a constant way, a lack o f temperature change or a momentary temperature growth bear fitness to an additive crystallisation heat amount existing inside the crystallised metal, and the differential curve describes the kinetics of his heat emission.

For determination of the baseline on the basis of the thermo-derivative curve diagram - known also as the calorimetric curve - in such a way that the area between the f_b' (t) and f'(t) curves determines the whole crystallization heat, there can be described the kinetics of the hidden crystallization heat emission [4].

The baseline of the crystallisation process describes the change of emitted heat into the surrounding through the sampler without any hidden crystallisation heat. Without occurrence of crystallisation there is fulfilled the following condition:

$$\frac{dT}{dt} - \left(\frac{dT}{dt}\right)_b = 0 \tag{1}$$

When crystallisation heat emission occurs, that means when crystallization is going on, then the momentary heat effect is described by the following dependence:

$$\frac{dQ}{dt} = c_p(t) \cdot m \cdot \left[\frac{dT}{dt} - \left(\frac{dT}{dt}\right)_b\right] \tag{2}$$

where: Q – crystallisation heat, c_p – heat capacity, m – mass of the out crystallised metal.

Calculating the heat effect of the crystallization in time t there should be taking into account the heat capacity, which depends on the ratio of the crystallized metal to the remaining liquid.
The total crystallization heat amount can be determined by integral calculation of the term:

$$\frac{dQ}{dt}:$$

$$Q = c_p \cdot m \cdot \int_{t_N}^{t_{Sol}} \left[\frac{dT}{dt} - \left(\frac{dT}{dt}\right)_b\right] dt \tag{3}$$

Temperature change, solidification rate, cooling rate as well as fraction solid can be determined experimentally using thermo analysis or thermo-derivative analysis.

At present in industry practice there are applied two methods of the thermo analysis:

- Ordinary thermo analysis, which makes it possible to determine the temperature of the physical and chemical transformations during constant heating and cooling process on the basis of registration of temperature and time,
- differential thermo analysis DTA *(Differential Thermal Analysis*), which includes the measurement of the temperature difference between the investigated sample and the reference sample where any heat transformation doesn't occur during heating or cooling. The sample is heated or cooled at the same time in similar environmental conditions. Such a measurement makes it possible to found heat effects which are coexistent with toe physical and chemical transformations.

Depending of the cast process conditions there are produced materials with very miscellaneous microstructure caused by different crystallisation process of the liquid metal. Experimental results confirm, that there is a huge interdependence between the mould shape, the heat transmission coefficient and the dendrite arm spacing, and that fore also onto the alloy properties [5].

2. Material and experimental procedure

For determination of the interdependence between the results of the thermo-derivative analysis and the microstructure of the cast aluminium alloy AC-AlSi7Cu3Mg (EN 1706:2001) (Table 1), cooled with different cooling rates, following investigations were performed:

- alloy microstructure using light microscope MEF4A supplied by Leica with a special software for image analysis and scanning electron microscope using the Zeiss Supra 35 device, with high resolution mode,
- thermo-derivative analysis using the UMSA metallurgical simulator.

Table 1. Chemical composition of AC-AlSi7Cu3Mg aluminum alloy

Mass fraction of the element, %							
Si	Cu	Mg	Mn	Fe	Ti	Zn	Ni
7,5	3,5	0,3	0,25	0,36	0,11	0,13	0,04

Thermo analysis of the investigated alloy was performer using the UMSA device. The scheme of the heating and cooling system, of the thermocouple placement was presented on Fig. 1.

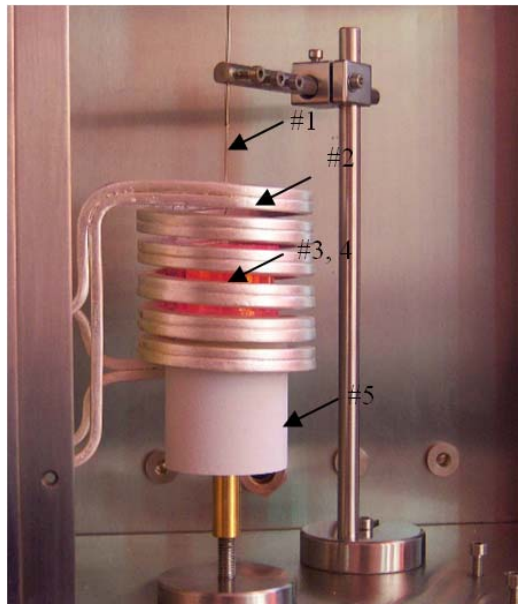

Fig. 1. Scheme of the heating and cooling system of the UMSA device as well the placement of the thermoelements: 1 – thermocouple, 2 – heating coil – cooling nozzles, 3 – steal foil, 4 – sample, 5 – sampler isolation

For achieving oh proper cooling rate, the samples were cooled with compressed gas supplied through the cooling nozzles placed in the copper coil.

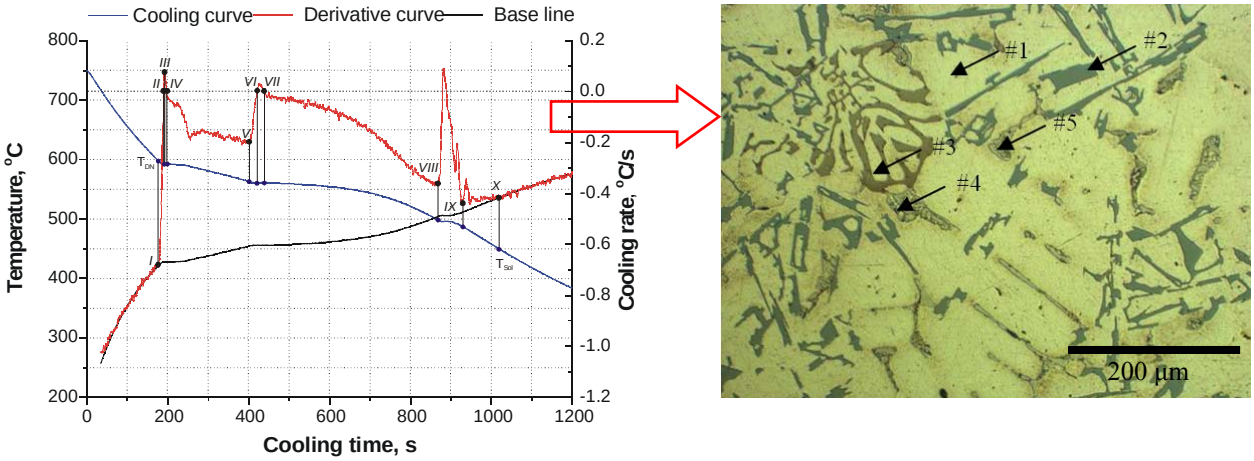

Fig. 2. Derivative curie, cooling curve, baseline as well the related microstructure for the cylindrical sample cooled with a cooling rate of 0,2°C/s, 1 - α, 2 - β, 3 – $Al_{12}(FeMn)_3Si$, 4 – Al_2Cu, 5 – Mg_2Si

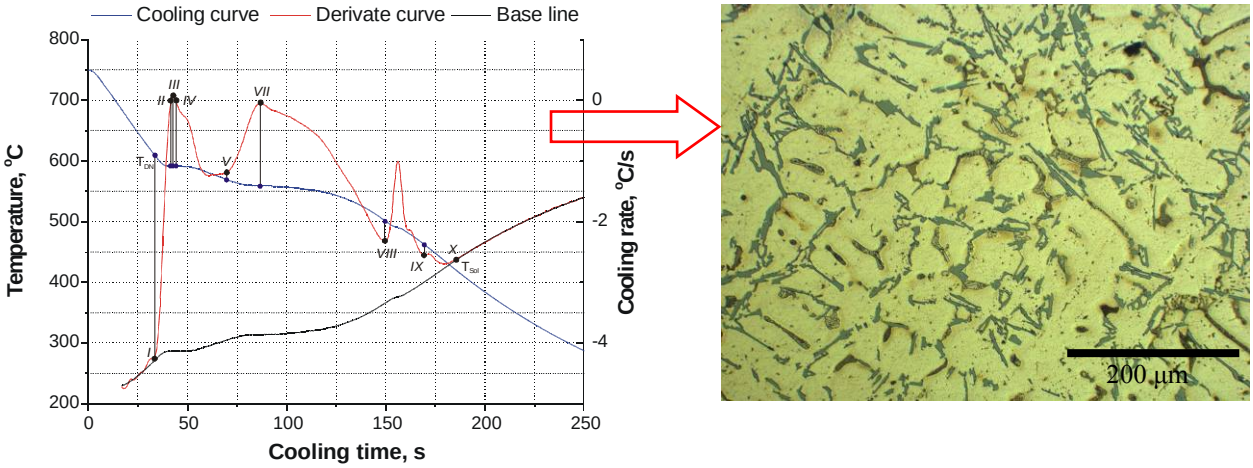

Fig. 3. Derivative curie, cooling curve, baseline as well the related microstructure for the cylindrical sample cooled with a cooling rate of 1,0°C/s

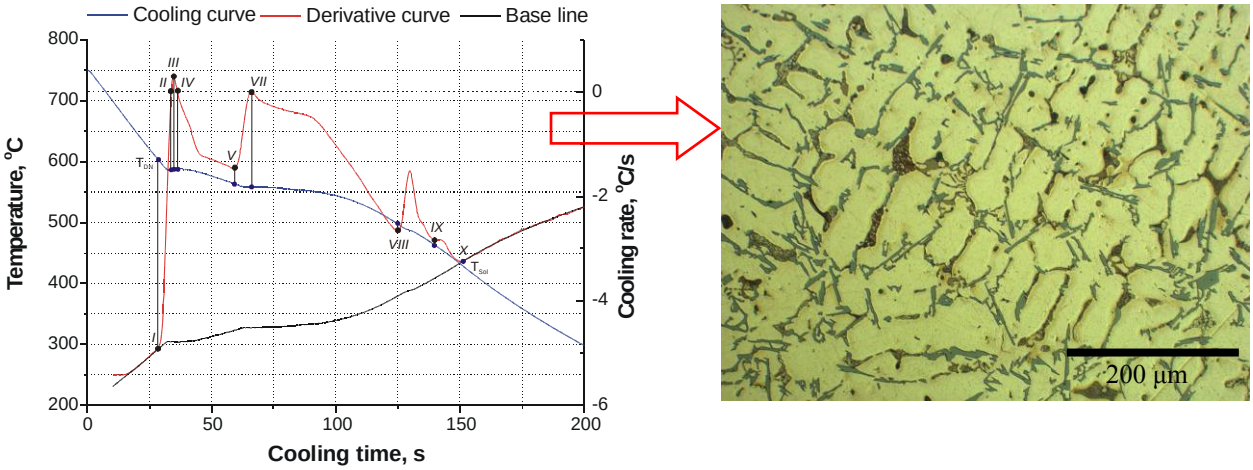

Fig. 4. Derivative curie, cooling curve, baseline as well the related microstructure for the cylindrical sample cooled with a cooling rate of 1,25°C/s

The pressure of the cooling gas was regulated using a rotameter. The flow rate of the cooling gas was regulated in such a way, that following cooling rates were achieved:

- ≈0,2 ºC/s, sample cooled with the furnace without any forced cooling,
- ≈1 ºC/s.
- ≈1,25 ºC/s, for cylindrical samples.

For the temperature measurement the K-type thermocouple was used. The tests were performed several times for each of the cooling rate for statistical evaluation of the investigation results.

For investigation the bulk cylindrical shaped samples were used with a diameter of 40 mm as well hollow cylindrical samples with a outer diameter of 40 mm, inner diameter of 16 mm and high of 40 mm.

3. Discussion of the experimental results

For the reason of the determination of the dependence between the results of microstructure investigations, alloy properties as well the cooling rate the thermo-derivative analysis was performer, the microstructure investigations including also computer aide image analysis for evaluation of the percent amount of the present phases In the microstructure as well for the determination of the primary and secondary dendrite arm length.

The performed investigation has allow it to determine the crystallization sequence of the phases and eutectics, which is presented in table 2.

Table. 2. Description of the characteristic points on the cooling curie from Fig. 2-4

Point on the graph	Description
I	T_{DN} nucleation temperature
II	T temperature of the beginning of the crystal growth (α phase dendrites)
III	Dendrites (α phase) growth temperature
IV	Temperature of αphase dendrite growth and (Al+Fe+Mn+Si) precipitation growth
V	Nucleation temperature of the α+β eutectics
VI	Temperature of the α+β eutectics growth
VII	Temperature of the stable eutectic growth α+β, in this point occurs the thermal equilibrium of the crystallized phases
VIII	Crystallization temperature of Cu phase and α+β eutectic
IX	Crystallization temperature of Mg, Cu phase and α+β multiphase eutectic
X	T_{Sol} temperature of the crystallization end

On Figs. 2-4 there are presented the thermo-derivative analysis diagrams including cooling curves, derivative curves and baselines [6,7] achieved for the investigated alloy cooled with different cooling rates.

For samples made of the investigated alloy cooled with chosen cooling rates the computer image analysis was performed [8], presented on Figs. 2-4. Visible microstructure refinement causes an increase of mechanical properties. Results concerning chemical composition and phase determination of the microstructure compounds were published in work [9,10].

4. Conclusion

Appliance of thermo-derivative analysis for investigations of the changes occurring during the crystallisation process of the alloys as well the microstructure analysis including quantitative and qualitative analysis, as well mechanical properties tests allows it to make a correlation between the alloy properties and a certain chemical and phase composition, of an alloy cooled with different cooling rates.

Result of this investigations makes it possible to determine the properties of the metals and its alloys based on derivative curves. This type investigations allows it to make an prediction of alloy properties as a result of microstructure modelling.

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