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Examples of thermal derivative gradient analysis (TDGA) application

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Abstract

The study presents the possibility of TDGA method based on multipoint measurement of temperature during crystallization of two test castings poured from different temperature. Sample castings have conical shape. Temperature measurement is carried out in six cone cross-sections between the heat axis and the surface of the mould cavity. The multichannel recording device allows describing the thermal derivative after time and direction. Presented examples of the assessment of kinetics of crystallization for alloys: AlSi11, CuSn8 and GJL 200 cast iron. Based on quantitative analysis of the microstructure (DAS, fig. 1 and tab.1) examples of relations were prepared, describing the mechanical and structural properties in function of crystallization kinetics parameters - DAS = f(v_{chl}) DAS = f(Δ(dT/dt)/dt) HV = f(G_y) Rm = f(Δ(dT/dt)/dt) as shown in fig. 2 and 4. The evaluation of crystallization process was also conducted for AlSi11 skeleton castings. Pointed out was the universality of the method with respect to the mathematical interpretation of the results and its limitations due to data registration extracted from multiple thermocouples. Based on the conducted studies, the method can be applied in the industry as well as in the experimental practice.

Keywords: **C**rystallization, Thermal derivative gradient analysis, AlSi, CuSn, Cast iron, Cast skeleton

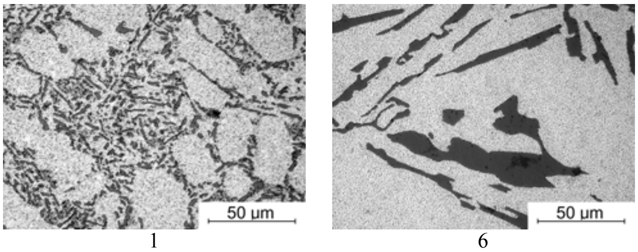
1. Introduction

The presented examples of application of the TDGA method are designed to indicate the possibility of its use in practice and the experimental production and testing of cast alloys and composites, which characteristic feature is the variable wall thickness. The method may prove particularly useful in the design of technology of thin-walled castings. The main problem is to predict the crystallization of cast microstructural features as a result of kinetics of heat movement. Most of the studies points to the dependence of microstructure and the mechanical properties of the cooling rate, crystallization rate and thermal gradient [1 ÷ 27]. Premises for the development of the method are the availability and versatility of digital measurement methods together with digital processing of data and increasing the quality of castings. It also stressed the possibility of widespread use of simulation programs, however, like the experimental method they have some limitations. Hence, the attempt to develop alternative and complementary methods for the diagnosis of experimental alloys and composites using multi-temperature measurement. Concept and basis for TDGA method was presented in [28 ÷ 34].

2. Materials, methodology and studies results

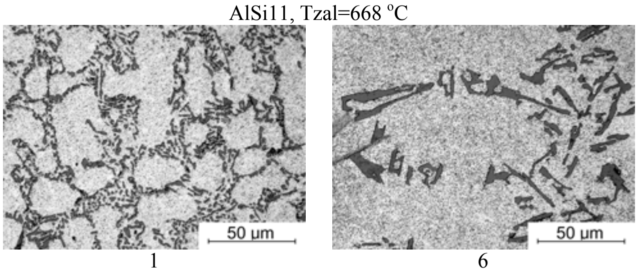
In order to validate the test method and instrumentation analyzed were following alloys: AlSi11, CuSn8 and GJL 200 cast iron. Such alloys were selected in order to achieve the basic objective of the work, i.e. determining the suitability of the method and instrumentation. AlSi11 alloy is essentially a two-component eutectic alloy, with a narrow crystallization range, exhibiting the characteristics of a model alloy, but with great practical significance. The CuSn8 alloy has clearly dominant α phase, with small quantity of eutectoid. Attempts made for the cast iron GJL 200 have practical importance, since it is produced in specific conditions of Centrozap Defka Foundry. Because of the production profile - e.g. bodies, pumps and valves for high-pressure hydraulics, the quality of the production of cast iron must be high.

The method was applied to determine the crystallization kinetics of AlSi11 skeleton castings, characterized by high and diverse cooling rates typical for thin-walled castings. Quantitative analyses of microstructure were carried out in the corresponding to thermocouples locations of casting microregions. Dendrite arm spacing (DAS) parameter was determined. Fig. 1 shows selected micrographs of investigated alloys.



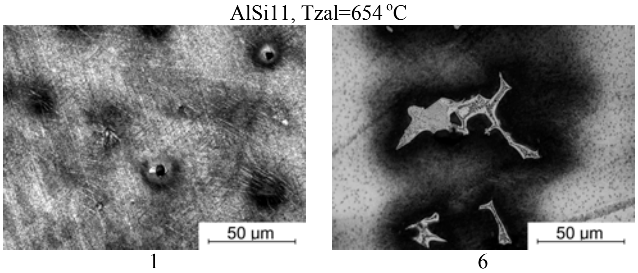
1 6

AlSi11, Tzal=668 °C



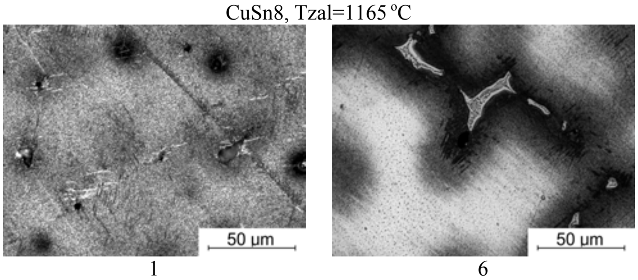
1 6

AlSi11, Tzal=654 °C



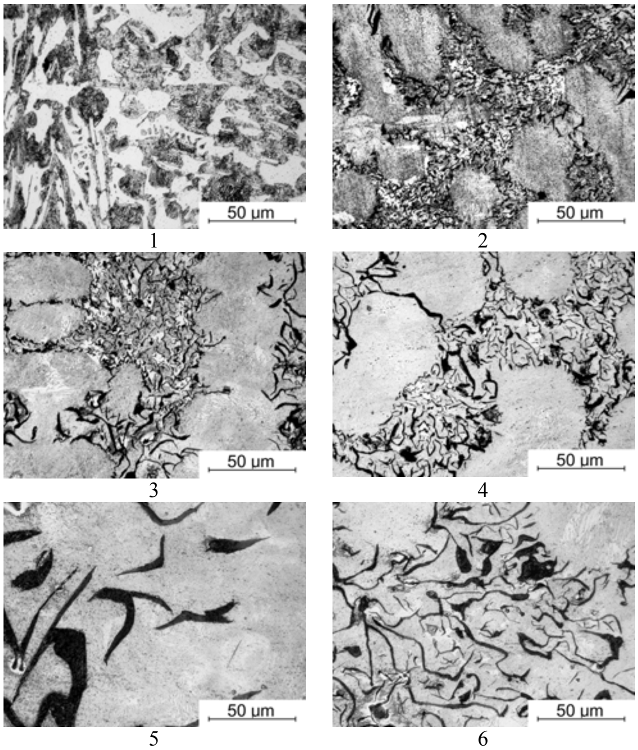
1 6

CuSn8, Tzal=1165 °C

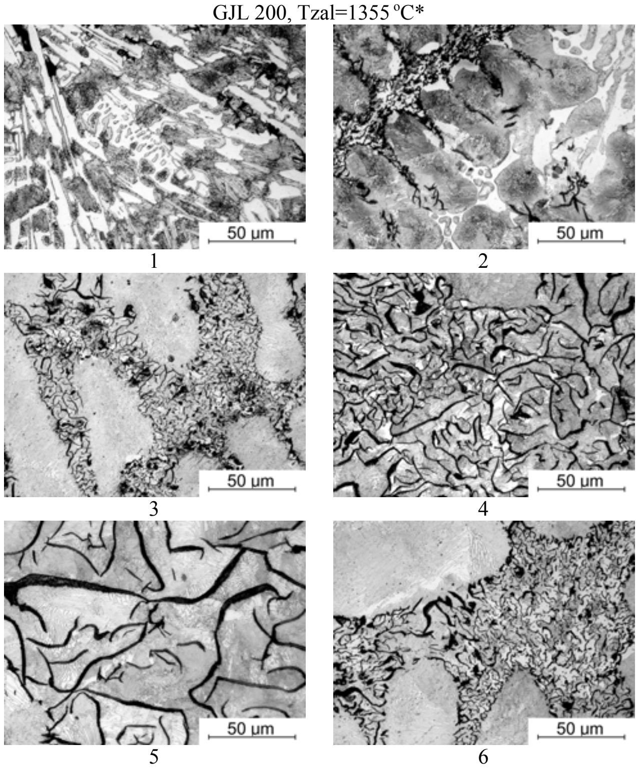


1 6

CuSn8, Tzal=1140 °C



GJL 200, Tzal=1355 °C*



GJL 200, Tzal=1335 °C

Fig. 1. Microstructure of alloys tested. 1 ÷ 6 indicates the thermocouple location in which microsection was observed. 1) corresponds to the highest rate of cooling, and 6) the lowest rate of cooling - at the base of the cone sample casting

For AlSi11 and CuSn8 alloys only examples of the extreme variation in microstructure were shown, corresponding to the location in areas with the highest and lowest cooling rate. Microstructure of other microregions was characterized by proportional degree of refinement.

In the case of cast iron all 6 microregions are shown, occurring in the areas of temperature measurement in order to accurately depict the structural changes, in particular, the morphology of graphite.

Table 1 provides examples of results of DAS determination in the characteristic cross-sections of the sample castings prepared in pairs, with different pouring temperature.

Table 1.
The results of secondary DAS measurements

Oznaczenie próbki	Miejsce pomiaru od czoła próbki mm	Odległość wtórnych osi dendrytów, µm			
		Min.	Maks.	Śr.	Odchstand
AlSi11, Tzal=668 °C	20	8,4	42,0	23,2	6,4
	41	13,2	52,2	26,8	8,7
	57	43,9	143,4	70,6	20,2
	78	68,3	167,8	104,6	22,8
	99	94,0	285,7	139,1	33,0
	115	102,4	488,9	182,4	60,5
AlSi11, Tzal=654 °C	19	11,2	32,6	21,2	4,9
	41	16,8	64,4	30,2	10,2
	59	56,2	149,1	94,7	21,9
	81	74,5	255,3	148,1	34,7
	101	118,0	311,6	219,7	47,0
	117	155,8	538,5	302,1	77,8
CuSn8, Tzal=1165 °C	18	20,8	69,4	44,4	10,8
	40	33,5	145,0	76,0	21,1
	58	57,3	202,2	115,2	33,3
	78	81,0	279,6	166,6	42,9
	98	82,9	335,5	193,5	54,1
	117	112,5	497,1	307,8	71,0
CuSn8, Tzal=1140 °C	21	12,4	49,7	33,0	7,7
	41	43,5	143,0	70,6	17,7
	60	25,6	137,5	92,3	19,4
	81	74,8	149,1	101,6	17,3
	99	84,5	325,7	210,4	45,4
	118	250,4	591,2	367,9	68,0
GJL 200, Tzal=1355 °C*	16	13,3	45,6	27,6	6,4
	38	27,9	85,0	43,8	8,9
	54	62,4	159,2	100,2	23,4
	74	125,8	268,9	178,0	25,6
	96	120,6	336,8	213,4	40,0
	115	140,5	477,8	285,0	69,0
GJL 200, Tzal=1335 °C*	20	13,4	59,2	27,6	9,4
	41	23,1	95,4	56,4	13,8
	60	61,2	198,1	129,8	29,5
	79	108,5	295,1	160,2	37,5
	102	141,6	369,2	224,1	44,5
	117	200,3	511,1	364,6	60,9

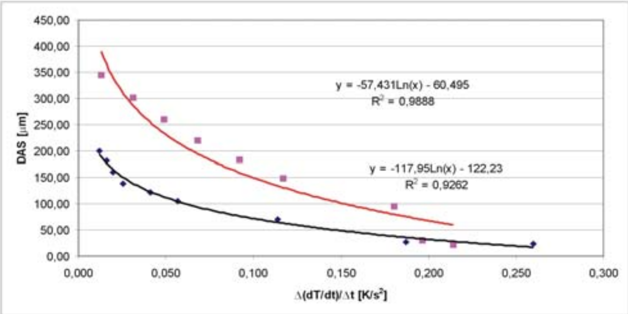
Lowering the pouring temperature is accompanied by increase of DAS and the reduction of its diversity at both high and low cooling rates. The size of the standard deviation of measurement within the scope of about 25 ÷ 30% was the result of spatial orientation of dendrites and their quantitative surface analysis. With the decreasing spatial orientation of dendrites, deviation increases.

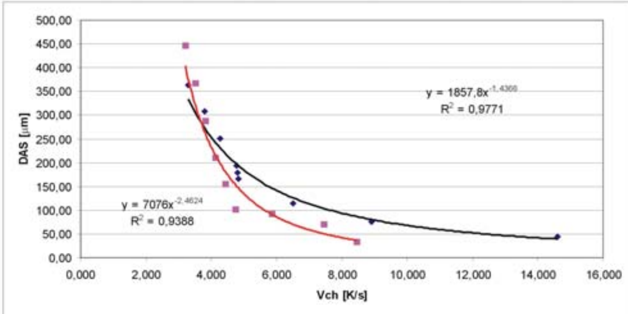
The direct and primary result of the measurement method is temperature for six thermocouples recorded as a function of time ($T\ 1 \div 6 = f(t)$).

Based on the temperature dependencies the derivatives were prepared, according to time and direction as a function of time and direction of heat movement, what was omitted in this work. Placing the thermocouples symmetrically between the heat axis and the wall of cavity creates conditions for the determination of the temperature gradient vector. Examined was the impact of the vertical gradient component, in conjunction with the temperature derivative of the time. Analyzed were the temperature increases and the component of the vertical gradient (ΔT and ΔGv) in time range (Δt), corresponding to an extreme extent of thermal effects of crystallization. Such approach avoids the impact of measurement error for temporary extreme temperatures. Analysis of the tangent of the angle of inclination of the measured value based on several points gives smaller error than building a relationship on the basis of a single extreme value.

Fig. 2 shows the relationship between the secondary dendrite arms (DAS) in [µm] and the cooling rate (v_{chl}) in a liquid state to T_{likw} in [K/s], the increase of temperature derivative after time before the first effect of crystallization ($\Delta(dT/dt)/\Delta t$) in [K/s^2] and the maximum value of temperature gradient vertical component (G_y) in [K/s] before the first crystallization effect in relation to the adopted temperature difference of pouring.

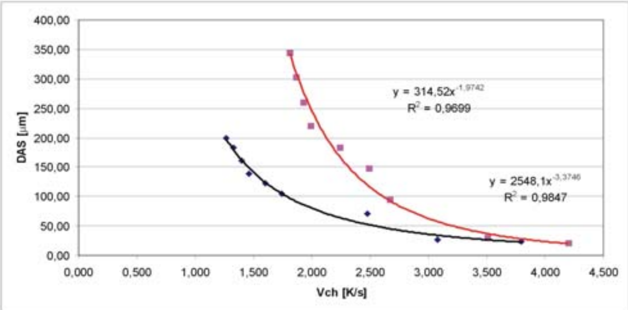
Basing on measurements of structural and mechanical properties results were obtained, which graphic interpretation was shown in fig. 4. Fig. 3 shows the cooling rate dependence of the thickness of the wall in AlS11 casting, $T_{zal} = 668^{o}C$. For industrial practice presented in fig. 2 relations can be set directly as a function of the wall thickness, using a dependency similar to those described in fig. 3. It should be noted, that, like other compounds also this dependence is related to temperature of pouring.

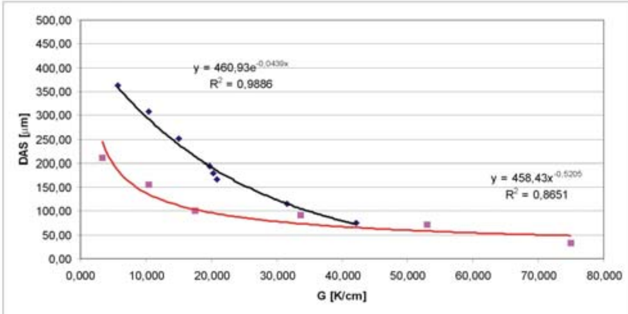




AlSi11 - DAS dependence = f (Δ(dT/dt)/dt), difference of pouring temperature ΔT_{zal} = 14 K

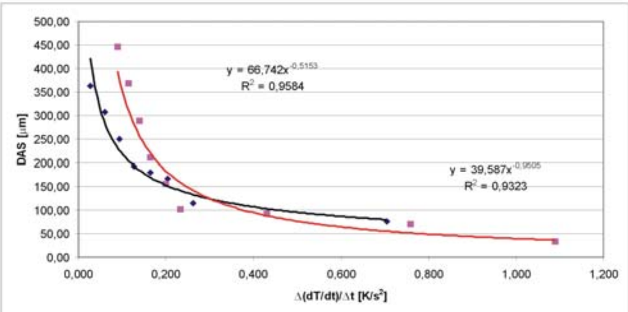
CuSn8 - The DAS = f ($v_{chł}$); difference of pouring temperature ΔT_{zal} = 25 K

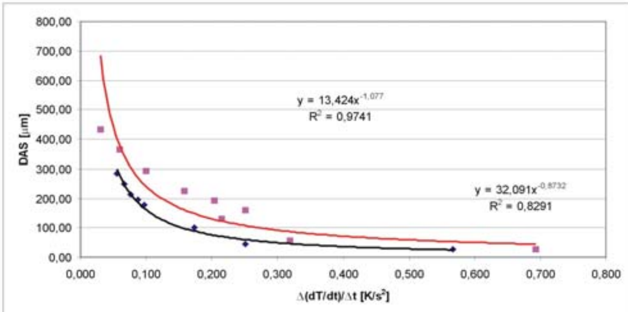




AlSi11 - The DAS = f ($v_{chł}$); ΔT_{zal} = 14 K

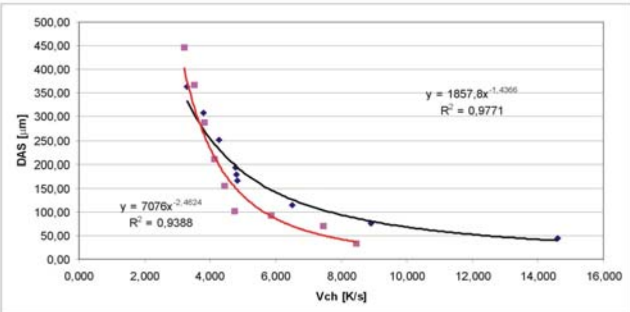
CuSn8 - The DAS = f (G_y); ΔT_{zal} = 25 K

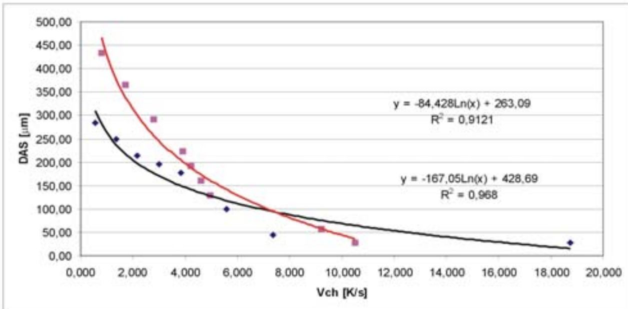




CuSn8 - DAS dependence = f (Δ(dT/dt)/dt); ΔT_{zal} = 25 K

GJL 200 - DAS dependence = f (Δ(dT/dt)/dt); ΔT_{zal} = 20 K





CuSn8 - DAS dependence = f (v_{ch}); ΔT_{zal} = 25 K

GJL 200 - DAS dependence = f ($v_{chł}$); ΔT_{zal} = 20 K

Fig. 2. Examples of structural relationships represented by the DAS as a function of selected parameters describing the crystallization kinetics

where: $v_{chł}$ - means the rate of cooling in the liquid alloy to T_{likw}; Δ(dT/dt)/ΔT - means the of temperature derivative after time increase before the first crystallization effect; G_y - means the maximum value of vertical component of thermal gradient before the first crystallization effect

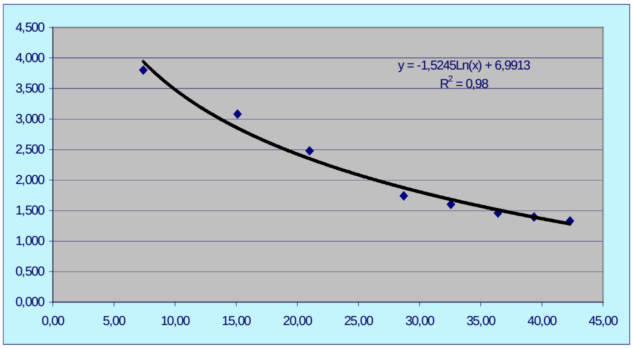
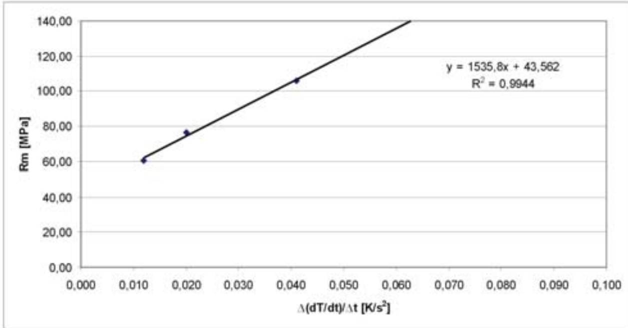


Fig. 3. Dependence of cooling rate and the wall thickness in AlS11 casting, T_{zal} = 668°C

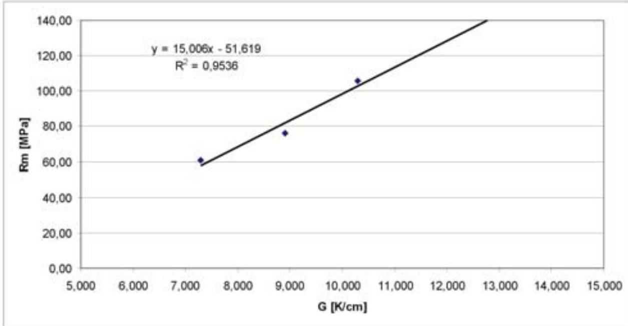
Fig. 4 gives examples of dependencies for hardness and tensile strength. It should be noted that the sample was obtained according to experimental measurements. There is also the possibility of determining the basic mechanical properties from the known relations, among others cited in the proposal for a project as well as in the first annual report. Dependencies are widely used in the simulation and are based on the DAS, rate of cooling and temperature gradient before the crystallization front. Due to the arithmetic conversion they were omitted in the presented work. However, their use to estimate the strength and hardness based on the results TDGA is fully justified. Experimental verification of the mechanical properties requires one comment. Strength tests were carried out on samples of dimensions (ø6x30) mm. Despite the small size, the number of samples obtained from cast sample is small, so the possible effect of casting defects and other inaccuracies of the measurement result need to be taken into account and the obtained results are in many cases inconclusive. This requires further detailed studies. This is due to the necessity of creating a database for the selected alloy casting of particular importance for industrial practice. This is undoubtedly a large and important research problem.

In addition to the schedule TDGA method was used to determine the kinetics of crystallization for skeleton castings. It is an example of the method application for experimental studies, in which the temperature measurement using thermocouples is difficult because of the minimum size of the wall and there is no access to interior parts of casting. The characteristics of the skeleton geometry are based on repetitive macrostructure with cells created from rectilinear elements of circular cross-section through the three perpendicular axes.

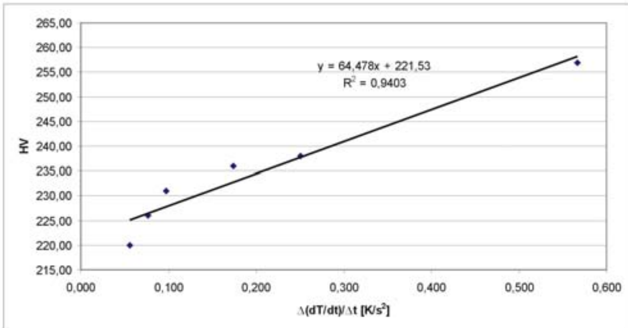
Elements create the skeleton of the rectangular cells, the axis of symmetry of the elements are the edges of the rectangular frame. The radius of the components of the frame falls within the range of up to 3 mm. An example of closed skeleton casting was shown in fig. 5.



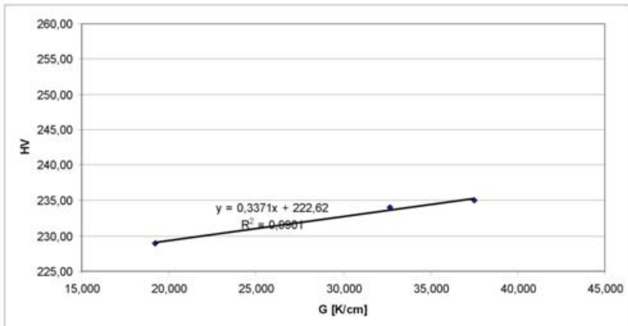
AlSi11 - dependence of Rm = f ((Δ(dT/dt)/Δt); T_{zal} = 668 ° C



AlSi11 - dependence on Rm = f (Gy); T_{zal} = 668 ° C



GJL 200 - dependence of HV = f ((Δ(dT/dt)/Δt) T_{zal} = 1355 ° C



GJL 200 - dependence of HV = f (G_y) T_{zal} = 1335 ° C

Fig. 4. Hardness and strength as a function of crystallization kinetics parameters for selected alloys tested

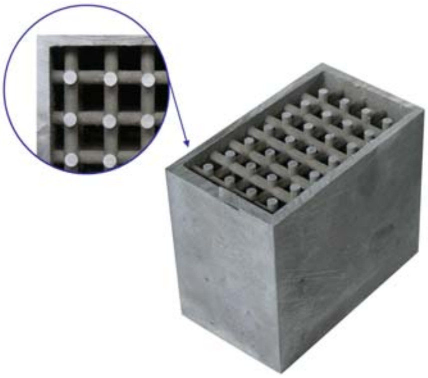


Fig.5. Example of closed skeleton AlSi11 casting, 125x70x125 mm T_{zal} 983 K

The need of minimizing the cell and connectors dimensions raises questions about the nature of crystallization kinetics. Skeleton walls crystallize at high cooling rate not seen in classic castings. TDGA method was used to assess the rate of cooling, but also other parameters describing the crystallization kinetics. Using the regression relation (1 ÷ 3) the parameters describing the crystallization kinetics were estimated, based on an analysis in temperature range T_{zal} to T_{lik}. Accepted range, as confirmed by preliminary studies is responsible for producing cast microstructure with the degree of dispersion, defined by a distance of secondary DAS of α solution. As shown by other studies stereological quantities characterizing microstructure are correlated to the value of DAS. Analyzed, quantitative results of the morphology of microstructure are presented in tab. 2.

Table 2.
Summary results of microstructural studies

Numer odlewu próbnego szkieletowego	$\frac{B}{L}\left[\frac{1}{1}\right]$	$\frac{P}{A}\left[\frac{1}{\mu m}\right]$	DAS [µm]
1	0,42 – 0,51	0,47 – 0,60	47,6 – 51,6
2	0,44 – 0,48	0,44 – 0,54	54,1 – 69,7
3	0,41 – 0,46	0,81 – 9,33	116,2 - 188
4	0,31 – 0,45	0,83 – 0,99	96,6 - 120
5	0,45 – 0,52	0,47 – 0,54	49,1 – 50,2
6	0,39 – 0,44	0,48 – 0,56	44,1 – 59,7
7	0,40 – 0,52	1,07 – 12,27	117 - 164
8	0,45 – 0,51	0,48 – 0,56	52,2 – 52,6
9	0,39 – 0,43	0,47 – 0,56	49 – 53,3
10	0,44 – 0,46	0,47 – 0,55	39 - 49
11	0,40 – 0,48	0,50 – 0,55	48 – 51,3

where:
B/L, P/A, DAS - are, respectively: the width to length ratio, silicon surface area to perimeter ratio, dendrite arm spacing of α solution.

The smallest average total surface area of silicon crystals was obtained for sample 5 ($A_{śr}$=27,27 [µm^2]). The B/L coefficient also attains the highest value for sample 5, where the coefficient varies in the range 0.45 ÷ 0.52 for extreme regions of casting. The value shows the least extended crystals of silicon in the sample. The coefficient of Q/A shows the smallest range of variation for the extreme regions of sample 5, the coefficient varies in range 0.47 - 0.54. The smallest value of DAS was observed for samples 1, 5, 6, 9, 10. At the same time sample 5 was identified as a positive, in which DAS changed in a limited extent, as well as the extreme regions of casting achieves the smallest possible value. On the basis of preliminary studies, it can be concluded that the sample 5 (AlSi11, T_{zal} 1013K, T_{mould} 333K, h – 265 mm) shows the best structural properties. Potentially, sample casting manufactured under such conditions would have beneficial operating properties.

Taking into account the over-represented in trial castings made at a pouring temperature of 983K these castings crystallization kinetics parameters were analyzed. These are samples marked with numbers 7 ÷ 11.
Based on the relations (3 ÷ 5) following parameters were evaluated: range of changes for cooling rates (v_{chl}) in [K/s], increase of the vertical component of thermal gradient in time (v_{Gz}) in [K/(cm s)] and the ratio of both rates - decrease of temperature ($p_T = v_{chl}/v_{Gz}$) in [cm] for the selected conditions of castings manufacturing 7 ÷ 11. Common feature of sample castings was the same pouring temperature T_{zal} = 983 K.

$$DAS = 103{,}89 \cdot v_c^{-0{,}5768} \ [\mu m]; \quad v_c = \frac{\Delta T}{\Delta t} \ [K/s] \tag{3}$$

$$DAS = 80{,}587 \cdot v_G^{-0{,}3845} \ [\mu m]; \quad v_G = \frac{dT}{dz \cdot \Delta t} \ [K/cm{\cdot}s] \tag{4}$$

$$DAS = 94{,}79 \cdot p_T - 156{,}33 \ [\mu m]; \quad p_T = \frac{v_G}{v_c} \ [cm] \tag{5}$$

After transformations of (3 ÷ 5) we get:

$$v_c = 0{,}001 DAS^{-1{,}734} \ [K/s] \tag{6}$$

$$v_G = 0{,}0124 \cdot DAS^{-2{,}6} \ [K/cm{\cdot}s] \tag{7}$$

$$p_T = 0{,}0106 \cdot (DAS + 156{,}33) \ [cm] \tag{8}$$

In table 3 comparison of the parameters describing the crystallization kinetics in relation to DAS was presented. The resulting diversity of structure was caused by different initial temperature of the moulds. A similar effect can be obtained using moulding materials carefully selected with respect to thermal properties. This is possible when the crystallization kinetics parameters are determined.
It should be noted that the TDGA method could be a powerful tool when designing casting technology of cast skeletons and shaping their microstructure. Relationship (5), so-called relative rate of crystallization, as a linear dependence of the thermal derivative after time and direction due to its linear character is particularly worthy parameter in practice production.

Assuming the relationship of DAS and mechanical properties in skeleton castings, there is a real possibility of obtaining attractive mechanical properties by precise selection of only two technical factors related to the production and crystallization such as molding materials and the temperature of pouring. This verifies presented, forward-looking TDGA method application.

Table 3.
Cooling rates (v_c), increase of the vertical component of temperature gradient rates (v_G) and the relative speed of crystallization (p_T) related to DAS measured for skeleton castings

Numer odlewu próbnego	DAS [µm]	v_c [K/s]	v_G [K/cm·s]	p_T [cm]
7	117 - 164	0, 5 ÷ 0,8	0,2 ÷ 0,4	2,88 ÷ 3,38
8	52,2 – 52,6	3,3	3,1	2,20
9	49 – 53,3	3,2 ÷ 3,7	2,9 ÷ 3,7	2,17 ÷ 2,21
10	39 - 49	3,6 ÷ 5,5	3,6 ÷ 6,7	2,06 ÷ 2,17
11	48 – 51,3	3,4 ÷ 3,8	3,2 ÷ 3,8	2,16 ÷ 2,19

3. Summary

From presented statement of crystallization kinetics parameters it can be seen that microstructural properties can be evaluated based on application of different mathematical operators representing the kinetics and dynamics of heat movement in solidifying casting. However, most studies cited are based on two variables of the relationship describing the structural and mechanical properties, these are the rate of crystallization determined from temperature derivatives ratio after time and direction and the thermal gradient.

Presented relations compared to other, which were omitted in this work, illustrate best the kinetics of crystallization. As the evaluation criterion strong exposure of differences in crystallization kinetics related to pouring temperature, which facilitated their automatic analysis and interpretation.

Conclusion may be limited to the statement that the TDGA method can be recommended for technologies of castings with a very great diversity of crystallization kinetics. However, the essence is to identify the advantages of measuring the thermal gradient, the quantity most sensitive to changes in the transport of heat in the process of crystallization.

4. Conclusions

1. Thermal derivative gradient analysis conducted by the methodology enables determination of microstructure for different experimental castings with diverse wall thickness.
2. The method offers possibilities to develop a wide set of mathematical results.
3. Studies showed clearly the influence of pouring temperature on microstructure.
4. Analysis methodology yields results statistically important.
5. TDGA method can be recommended for use in research and experimental and industrial conditions. In both cases, intentionally, though not necessarily is to develop a database taking into account the metallurgical processes and charge materials.

Acknowledgements

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