

# Analysis of structural properties for AlSi11 alloy with use of thermal derivative gradient analysis TDGA

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## Abstract

In this paper a basis of thermal derivative gradient analysis was shown. Authors presented methodology of the studies, results and analysis. Studies of crystallization kinetics were conducted on non-modified AlSi11 eutectic alloy. Analyzing the results authors proposed some parameters for description of crystallization kinetics and their relation to microstructure and mechanical properties.

**Keywords:** Microstructure, AlSi alloy

## 1. Introduction

Theoretical fundamentals of the method have its roots in TDA method [1]. There is a wide knowledge on relations between kinetics of solidification, alloy structure and its mechanical properties [2-27]. Practical methods of alloys diagnostics are based on chemical and thermal analyses in conditions similar to those during alloy solidification in a casting mould. Such methodology together with analysis of mechanical properties of cast-on test bars is connected with assumed approximation to real conditions. Theoretical knowledge as well as the numerical techniques are much more developed than the experimental procedures.

This work is one of the approach presented by the authors [28-32], to improve the technological aspects of TDA method. The studies have experimental character and are aided by numerical simulation, using universal methods of measurement, registration and conversion of analogue thermal signal to numerical electrical signal [33]. The idea of the studies is based on multi-point measurement of temperature and analysis of its both derivatives showing crystallization process kinetics: temperature derivative over time and direction in range of time

and temperature of crystallization. Three variables, together with the primal- temperature, are considered in function of time and direction of solidification. The purpose of these studies is the absence of similar solutions applied in casting practice. Potential application of the studies results are foreseen for alloys with structural components which differs in range of thermal properties, especially for cast metal matrix composites. [28].

## 2. Methodology and results

The concept of this research [28, 31] is based on three assumptions:

1. Evaluation of crystallization kinetics for test casting taking into account diverse thickness of casting wall and the their interaction together with the feeder.
2. Temperature measurement localized half-way between casting wall and its thermal axis.
3. Detailed analysis of pouring temperature by casting two test castings with minimal interval, using the same metal form one cast and ladle.

First two assumptions resulted from aspiration to enclose in the studies the thermal interactions between parts of casting characterized by different solidification module. During one-point measurement the thermocouple is usually placed on thermal axis of the casting, in its thermal center, which moves during solidification in function of time. In such cases, the thermocouple position is assumed with some approximation. The small volume of the test casting with compact geometry suits the solidification module of a real casting. Assumed position of the thermocouple exposes it to measuring errors connected to physical and chemical phenomena as well as the discontinuities caused by shrinkage. Assuming changes in thermal center position in range of crystallization, the temperature measurement is realized only with some approximation of real thermal center position. Thus, it is assumed that analysis of crystallization kinetics should consider not the specific case of thermal center but the region between the mould wall and thermal axis of a casting solidifying directionally. The third assumption is mainly technological – its aim is to evaluate the differences in solidification kinetics caused by different pouring temperature. This parameter is the main technological factor, possible to regulate – especially important for liquid composite dispersions. Experimental studies and numerical simulation were conducted for non-modified AlSi11 eutectic alloy cast to sand moulds. The crystallization rate was assumed in range of:  $0,12 \div 1,2$  [K/s], what corresponded to casting wall thickness in range of:  $1,5 \div 45$  [mm]. The aim of numerical simulation was the verification of thermal and geometrical assumptions, real conditions and analysis of liquidus and solidus surfaces movement inside the solidifying casting. For experimental castings some structural analyses were conducted. Characteristic microstructure is shown in fig. 1.

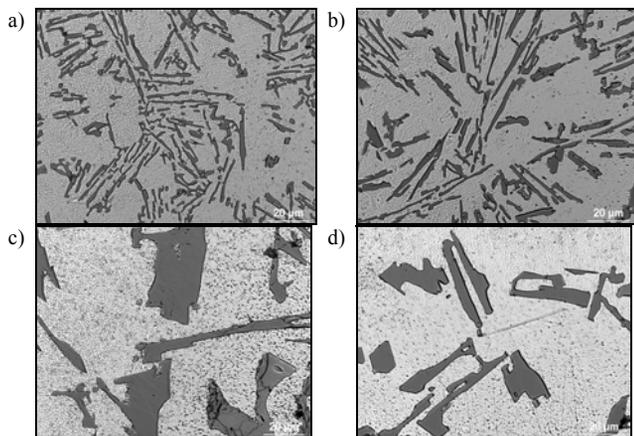


Fig. 1. Microstructure of studied non-modified AlSi11 alloy obtained at different cooling rates: a) and b)  $v_{chl}=1,1$ ; c) and d)  $v_{chl}=0,2$  K/s and for different pouring temperature  $\Delta T_{zal}=4^{\circ}\text{C}$ : a) and c)  $T_{zal}=718^{\circ}\text{C}$ , b) and d)  $T_{zal}=714^{\circ}\text{C}$ . In  $\alpha$  matrix non-modified crystals of eutectic silicon. Visible diversification of structural components dispersion

As expected, typical diversification of structure occurred caused by different cooling rates and different pouring temperature ( $\Delta T_{zal}=4^{\circ}\text{C}$ ).

In table 1, minimum, maximum and average values of dendrite arms spacing (DAS) are shown together with standard deviation values.

Table 1. Results of DAS measurements

Cooling rate [K/s]	Dendrite arm spacing (DAS) [ $\mu\text{m}$ ]				
	min.	max.	ave.	standard deviation	
				[ $\mu\text{m}$ ]	[%]
1,1	34,31	123,94	<b>82,97</b>	16,93	20,0
0,74	86,06	196,24	<b>133,60</b>	25,54	19,0
0,397	118,24	246,60	<b>180,73</b>	21,19	11,7
0,297	120,87	347,58	<b>203,52</b>	42,09	20,7
0,232	112,97	374,77	<b>264,40</b>	59,43	22,5
0,170	155,13	402,73	<b>277,76</b>	55,41	20,0
$T_{zal}=718^{\circ}\text{C}$					
1,1	43,72	133,70	<b>94,55</b>	18,97	20,1
0,828	35,21	175,21	<b>121,33</b>	26,77	22,1
0,431	101,23	261,47	<b>171,58</b>	27,34	15,9
0,318	108,48	283,64	<b>216,54</b>	45,66	21,1
0,215	131,87	361,06	<b>249,31</b>	48,85	19,6
0,153	174,05	564,52	<b>324,38</b>	71,60	22,1
$T_{zal}=714^{\circ}\text{C}$					

In fig. 2 examples of approximate relation between objects quantity ( $y=F(x)$ ) and DAS logarithm ( $\log x$ ) for different cooling rates and pouring temperature. This relation has the general form of (1):

$$F(x) = U Z \exp(Z (W - \log(x))) \cdot (1 + \exp(Z(W - \log(x))))^{-2} \quad (1)$$

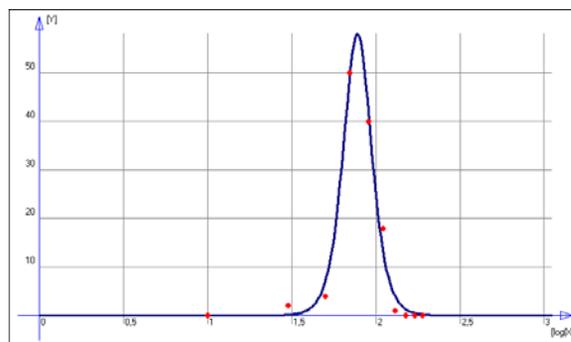
where:

U, W, Z – represents constants of approximation

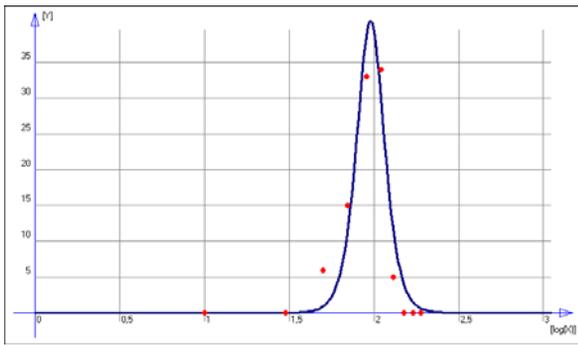
To compare measurements of DAS with standard deviation (table 1), the technological parameters and approximation parameters are shown below (the diagrams were neglected):

- $T_{zal}=718^{\circ}\text{C}$ ;  $v_{chl}=0,397$  K/s  
stand. dev.= 0,298; correlation= 0,9999; Fisher's test = 5245
- $T_{zal}=714^{\circ}\text{C}$ ;  $v_{chl}=0,431$  K/s  
stand. dev.= 0,269; correlation= 0,9999; Fisher's test = 4691

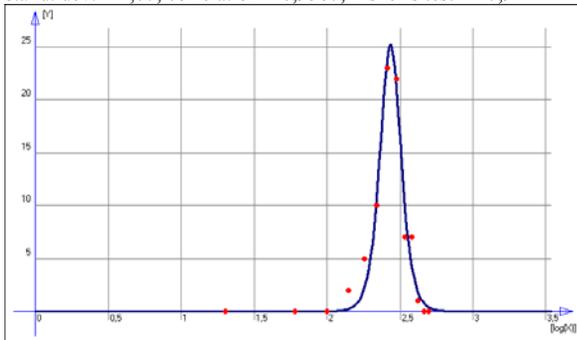
Analogy is based on the most accurate measurement for cooling rate of 0,4 K/s.



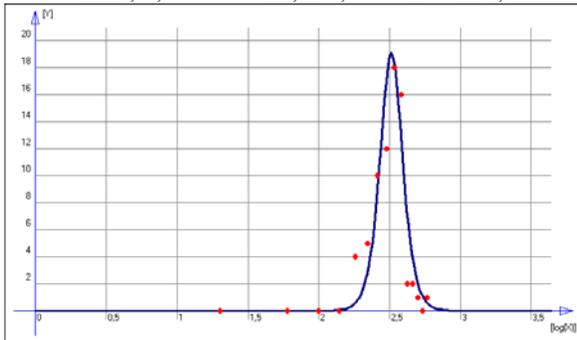
a)  $T_{zal}=718^{\circ}\text{C}$ ;  $v_{chl}=1,1$  K/s;  
U = 12,913; W = 1,8901; Z = 18,012;  
stand. dev. = 2,36; correlation = 0,9922; Fisher's test = 89,8



b)  $T_{zal}=714^{\circ}\text{C}$ ;  $v_{chl}=1,1$  K/s;  
 $U = 8,8186$ ;  $W = 1,9795$ ;  $Z = 18,509$ ;  
 stand. dev. = 4,07; correlation = 0,9567; Fisher's test = 17,9



c)  $T_{zal}=718^{\circ}\text{C}$ ;  $v_{chl}=0,17$  K/s;  
 $U = 4,7899$ ;  $W = 2,4352$ ;  $Z = 21,097$ ;  
 stand. dev. = 1,69; correlation = 0,9791; Fisher's test = 37,1



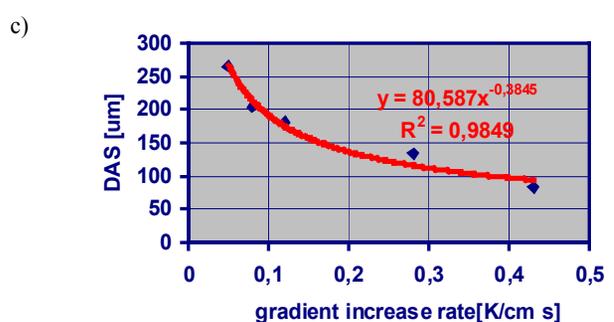
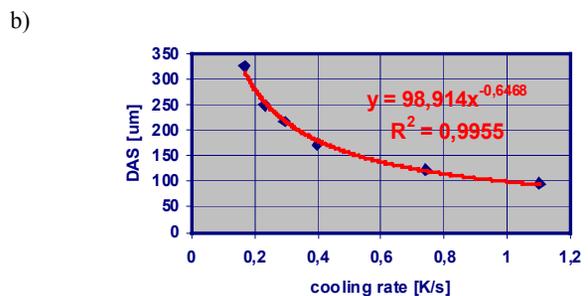
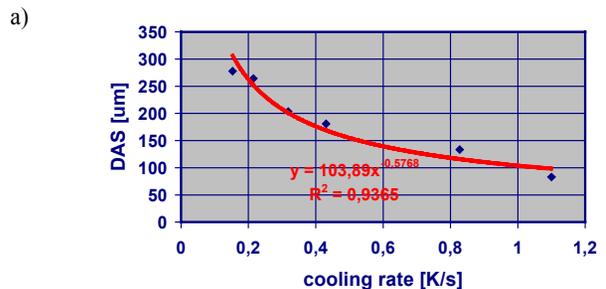
d)  $T_{zal}=714^{\circ}\text{C}$ ;  $v_{chl}=0,1531$  K/s;  
 $U = 4,0066$ ;  $W = 2,5121$ ;  $Z = 19,061$ ;  
 stand. dev. = 2,51; correlation = 0,9234; Fisher's test = 11,0

Fig. 2. Functional, approximate size of objects [1/1] related to logarithm of measured DAS [ $\mu\text{m}$ ]. Under each diagram technological and approximation parameters were set-up

Next, for each value of DAS, which characterize the microstructure, some relations describing the crystallization kinetics were assigned (fig. 3 and 4). Placing the thermocouples symmetrically between the thermal axis and mould cavity wall enabled determination of thermal gradient components. At this stage of the studies, according to literature, the vertical component was considered in relation to temperature derivative after time. Temperature and vertical component increases ( $\Delta T$  and  $\Delta G_v$ ) were analyzed in time range ( $\Delta t$ ), corresponding to extreme thermal effects in crystallization range. Such approach enables avoiding the measuring error influence. Analysis of tangent of

inclination angle measured on several points causes smaller error than building the relation on individual extreme values.

In fig. 3 the relation of dendrite arms spacing (DAS) in [ $\mu\text{m}$ ] and cooling rate ( $v_{chl}$ ) in [K/s], rate of vertical component of thermal gradient increase ( $v_{Gv}$ ) in [K/(cm s)] and  $v_{chl}/v_{Gv}$  ratio were shown for assumed different pouring temperature values. Shown relations in compare to others, skipped in this work, illustrated the crystallization kinetics in most clear way. As a criterion for evaluation, clear display of differences in crystallization kinetics was assumed, what facilitated automation of the analysis and interpretation. It is connected with the need of eventual application. It must be pointed out, that during the experiments some thermocouples were malfunctioning. Thus, the relations shown in fig. 3 are a bit poorer than expected. One of the measurements considering the lower pouring temperature and the slowest cooling was classified as a gross error and was neglected. Thus, the number of points on which presented relations are based differs. According to the concept of TDGA danger of loosing some data was one of the issue for determination of number of points in which the temperature will be measured ( $n=6$ ) within one test casting.



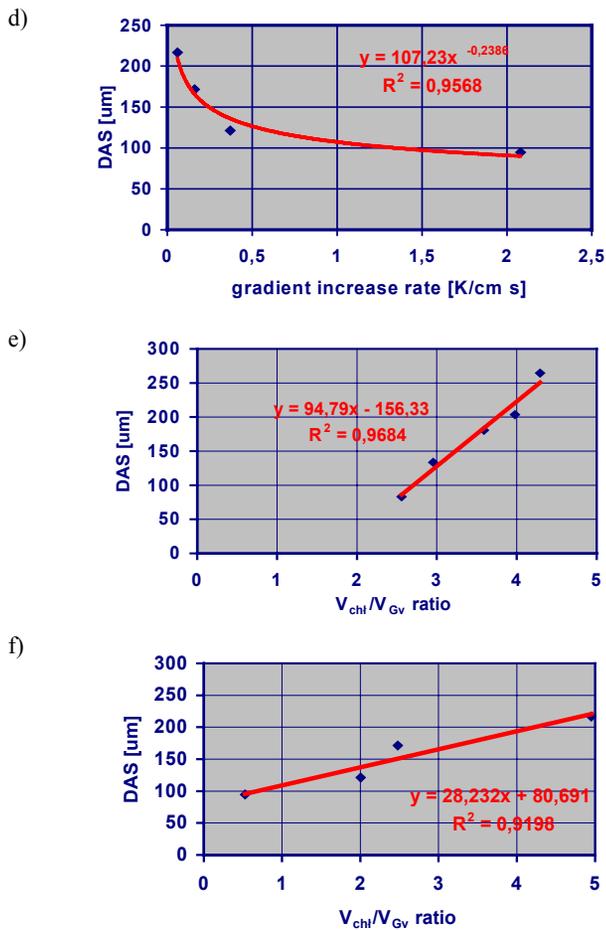


Fig. 3. Example diagrams for relations of DAS and kinetic parameters of crystallization at different pouring temperature  $\Delta T_{zai}=4\text{ }^{\circ}\text{C}$ ;  
 a) and b) in function of cooling rate  $v_{chl}=\Delta T/\Delta t$  [K/s]  
 c) and d) in function of gradient increase rate  $v_{Gv}=G_v/\Delta t=dT/(dl\cdot\Delta t)$  [K/(cm s)]  
 e) and f) in function of  $v_{Gv}/v_{chl}$  ratio;  
 Relation shown in pairs: fig. a), c), e) for  $T_{zai}=718\text{ }^{\circ}\text{C}$ ,  
 fig. b), d), f) for  $T_{zai}=714\text{ }^{\circ}\text{C}$

### 3. Results analysis and summary

Results analysis enclosed quantitative influence of variable pouring temperature and cooling rate during primal crystallization on casting microstructure. Fig. 1 shows the diverse refinement of structural components – the eutectic silicon crystals and  $\alpha$  phase dendrites. At each cooling rate the influence of pouring temperature is visible ( $\Delta T_{zai}=4\text{ }^{\circ}\text{C}$ ). Maximal difference in pouring temperature registered is equal to  $\Delta T=6\text{ }^{\circ}\text{C}$ , with delay of  $\Delta t=4\text{ s}$ .

Fig. 2 a) and b) indicate, that dendrite number increases of 50% with decreased DAS of 33% influenced by higher pouring temperature. Similar effects are observed for minimal cooling

rates fig. 2 c) and d). At higher pouring temperature dendrite number increased of 37%, with DAS decreased of 17%. Results of studies shown in table 1 was measured on one surface of both castings.

Standard deviation value for DAS is similar for both castings and equal to about 20%; the lowest value is observed for cooling rate of 0,4 K/s, what indicated the strongest orientation of the structure. In such conditions the histograms are represented in the most accurate way. Moreover, in this case the influence of pouring temperature observed is the weakest – such cooling rate of about 0,4 K/s represents the casting wall of 18 mm thickness.

The strongest influence of pouring temperature on structure diversification is observed at maximal cooling rates of about 1,1 K/s.

From setting-up the kinetic parameters of solidification concluded, that evaluation of microstructural features was possible with use of different mathematical parameters which displayed the heat flow in solidifying casting. Nevertheless, most of the cited works is based on two variables occurring in relations bonding the structural and mechanical properties. These are the crystallization rate calculated as a ratio of temperature derivative after time and direction of solidification and thermal gradient.

In fig. 4 an example of DAS relation from  $v_{Gv}/v_{chl}$  ratio was shown.

All presented relations fully describe connection between microstructure and crystallization kinetics parameters. Influence of pouring temperature on crystallization of non-modified eutectic alloy is readily observed in fig. 4 described with equations (2) and (3):

$$DAS = 94,79 \left( \frac{v_{chl}}{v_{Gv}} \right) - 156,33 \quad [\mu\text{m}] \quad (2)$$

$$DAS = 28,23 \left( \frac{v_{chl}}{v_{Gv}} \right) + 80,69 \quad [\mu\text{m}] \quad (3)$$

The main advantage of  $DAS=f(v_{Gv}/v_{chl})$  relation is its sensitivity to pouring temperature. In compare to other linear relations are easier for interpretation. From compared analyses and results the main factor influencing the crystallization process is the vertical component of thermal gradient and its rate of increase. Results of DAS evaluation based on cooling rate parameter are not satisfactory for observing the differences in crystallization kinetics. Relations (2) and (3) shown in fig. 3 e and f suggest some analogies to DAS evaluation based on solidification module. Such relation is shown in fig. 4 for comparison. Significant similarity of both relations at different pouring temperatures is caused by lack of possibility of noticing the thermal interactions between parts of casting characterized by different solidification module.

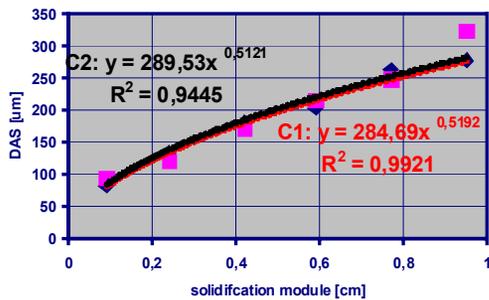


Fig. 4. Relation of DAS (DAS) [ $\mu\text{m}$ ] and solidification module ( $M_k$ ) [cm]

Determination of significant influence of pouring temperature on microstructure with use of TDGA shows a need for physical and mathematical model of this phenomena.

## Conclusions

1. Thermal derivative gradient analysis conducted according to presented methodology enables precise determination of casting microstructure with divers wall thickness.
2. Pouring temperature influences the casting microstructure for eutectic alloys, which in its nature have refined structure. For alloys with wide range of solidification temperatures the influence is expected greater.
3. Proposed methodology showed statistically important results. Presented method can be an alternative for other methods of alloy diagnostics.

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