DETERMINATION OF FITTING PARAMETERS IN COMPOSITE NUCLEATION MODEL: AZ91/SiC

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Abstract: The aim of this paper was to found the fitting parameters in micro-macro model for primary phase AZ91 based composite nucleation. Authors describe the theoretical basis of nucleation model. To find fitting parameters in this model the series of castings were performed. Data from those casting was used for statistical analysis. At the end mathematical formula that describe reinforcement particles size and undercooling effect on nucleation rate was derived.

1. INTRODUCTION:

Grain size is one of the most important structural characteristic that determining mechanical properties. Knowing element properties, the proper application regions for it can be chosen to achieve best mechanical properties and performance[1-3]. Nowadays simulation software can be use to predict the element microstructure. those programs base on micro-macro model of crystallization. the model consists of partial differential equations (PDEs) that describe the nucleation rate, diffusion in the casting, casting cooling rate and every single grain growth rate. often it is hard to find the theoretical value of the parameters that appear in those PDEs. it is possible to find them from experiment. the experimental data after applying statistical methods let us find approximated values of the so-called “fitting parameters” in the mentioned models.

2. MODEL DESCRIPTION

In the mathematical model it is assumed that heat transfer is governed by Fourier – Kirchhoff (FK) equation:

$$\frac{\partial T}{\partial \tau} = \frac{1}{c_p \rho} div(-\lambda gradT) + \frac{q}{c_p \rho}, \qquad (1)$$

where: T [K] – is temperature; τ [s] – time; c_p [J kg^{-1} K^{-1}] – specific heat; ρ [kg m^{-3}] – density; λ [W m^{-1} K^{-1}] –thermal conductivity; $q = L\ (df_s/d\tau)$ [W kg^{-1}] – heat of crystallization.

q **-** is a parameter determining the amount of heat evolved in the crystallization process, and is a function of f_s depend on the number of grains appearing over time and the radius growth rate of grains. After nucleation period existing grains still grow but there are no new grain initiation. Nucleation ends as recalescence appears [4].

Tab. 1. Chemical composition of AZ91 alloy, wt. % determined using ICP-OES VARIAN Vista MPX

Al	Zn	Mn	Fe	Be	Si	Cu	Ni
8.5	0.64	0.23	<0.002	10 ppm	0.03	0.003	0.001

3. NUCLEATION RATE

Nucleation rate can be described on the basis of models of nucleation of grains such as:

- Oldfield nucleation model [4,5] :

$$N_V(T) = \psi(T_N - T)^m, \left[m^{-3}\right] \tag{2}$$

- Fras nucleation model [6]:

$$N_V(T, d_{SiC}) = \lambda(d_{SiC})\exp\left(-\frac{b(d_{SiC})}{(T_N - T)}\right) \tag{3}$$

where: T [K] is current temperature; d_{SiC} [m] is the particles mean diameter; λ [m^{-3}], b [K] denotes model adjustment parameters; T_N – is nucleation temperature, ψ, m – ucleation parameters determined experimentally.

4. EXPERIMENTAL PROCEDURE - COMPOSITE CASTING:

The composite with AZ91 (**Tab. 1**) metal matrix and SiC reinforcement particles was prepared. Three castings were prepared for different SiC particles size, the sizes were: A – 10 μm, B – 40 μm, C – 76 μm. During each casting 6000g of the AZ91 alloy was used. Different weight percentage of SiC particles: 0, 0.1, 0.5, 1, 2, 3.5 wt.% were used to prepare different composites. The samples were casted into standard thermoanalysis croning sand cup with thermocouple K type. The specimens were taken from the region near to the thermocouple. Thermoanalysis data was used for cooling speed and undercooling determination for each sample.

Tab. 2. Conditions comparison for castings made in Chair of Casting Research, Leoben

CASTING CONDITIONS			
Casting symbol	A	B	C
Particles size, [μm]	10	40	76
AZ91 mass, [g]	5960	6250	5800
Ambient temperature, [°C]	22	22	24
Furnace temperature, [°C]	750	750	750
Particles temperature, [°C]	320	320	320
In-mould temperature, [°C]	100	100	100
Stirring time, [s]	240	180	180

5. ETCHING

The specimens were then cut, grinded and polished with the grinding machine. Etching of the specimens was performed according to the procedure described by the Maltais [7,8] and improved by authors[9]. The chemical composition of etchant was: 50 ml Distilled Water, 150 ml Ethanol, 1 ml

Acetic Acid. The etched specimens were examined using a light optical microscope Carl Zeiss AXIO Imager.A1 with cross polarized light and λ filter. The images on computer display reveals arms of different dendritic grains as areas with different colours.

<u>Optical micrographs of the etched samples viewed under cross polarized light using λ filter for different AZ91/SiC composites (Examples of the etching effect):</u>

a) 0% of SiC;

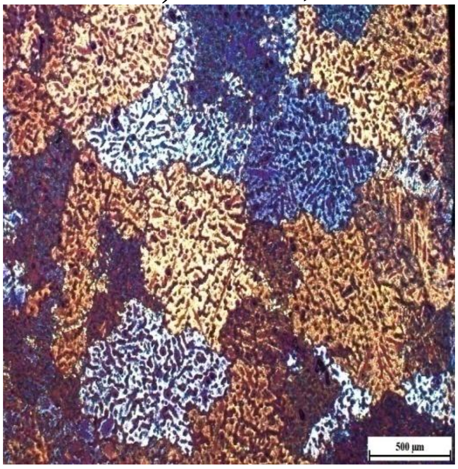

Fig. 1. 0% of SiC;

b) 2% of SiC, particles mean diameter 40 μm;

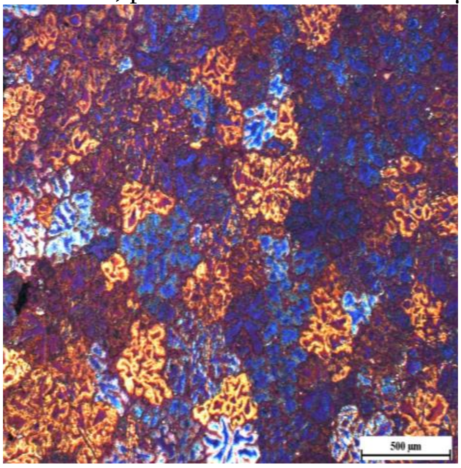

Fig. 2. 2% of SiC, particles mean diameter 40 μm;

c) 0.1% of SiC, 10 µm;

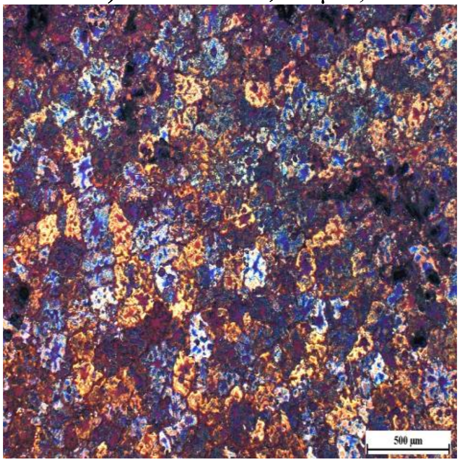

Fig. 3. 0.1% of SiC, 10 µm;

d) 3.5% of SiC, 76 µm.

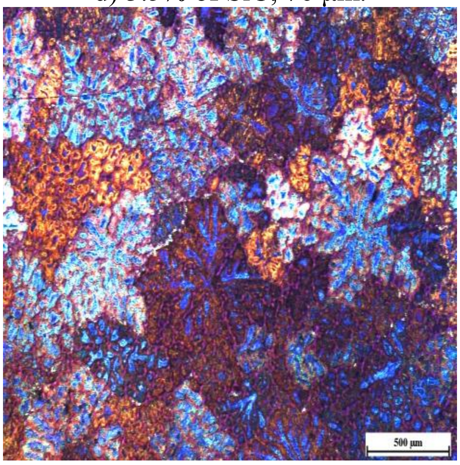

Fig. 4. 3.5% of SiC, 76 µm.

6. GRAIN DENSITY MEASUREMENT

Data gathered from optical micrographic analyse was then used to calculate the grain density N_V. To find this value from optical analysis data, the Saltykov equation can be used [10]:

$$N_V = \frac{2}{\pi} N_a \left(\frac{1}{d}\right)_{mean} \tag{4}$$

where: N_V [m^{-3}] is mean volumetric grain density, N_a [m^{-2}] is mean grain density on the surface, and $(1/d)_{mean}$ [m^{-1}] denotes average value of $(1/d)$ [m^{-1}] for all grains found on the polished section.

7. RESULTS OF THE ANALYSIS:

Statistical analysis was done In Statistica 8.0 software,

N_V on undercooling dependence for <u>10 µm</u> particles:

$$N_V(T) = 1.7 \cdot 10^{14} \exp\left(-\frac{43.21}{T_N - T}\right), \qquad R^2=0.993,$$

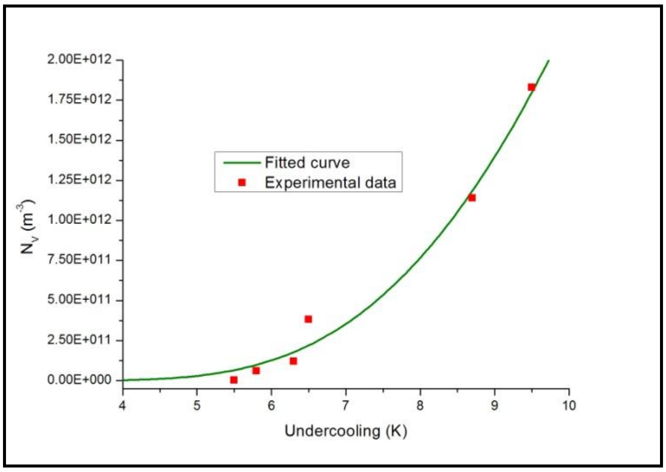

Fig. 5. N_V on undercooling dependence for <u>10 µm</u> particles.

N_V on undercooling dependence for <u>40 µm</u> particles:

$$N_V(T) = 3.9 \cdot 10^{15} \exp\left(-\frac{93.83}{T_N - T}\right), \qquad R^2=0.984,$$

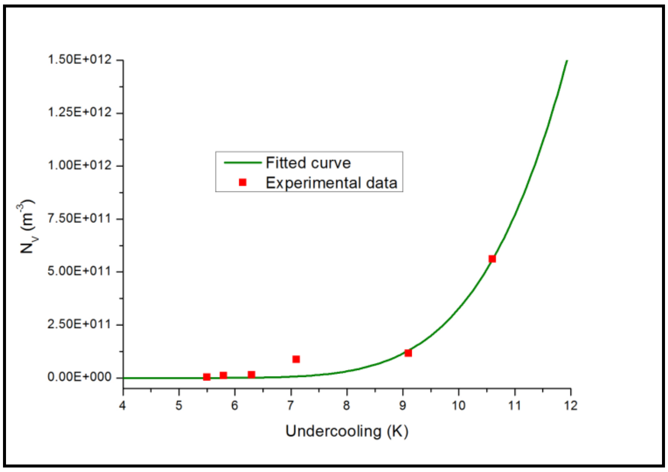

Fig. 6. N_V on undercooling dependence for <u>40 µm</u> particles.

N_V on undercooling dependence for 76 µm particles:

$$N_V(T) = 9.4 \cdot 10^{16} \exp\left(-\frac{135.09}{T_N - T}\right), \qquad R^2=0.999.$$

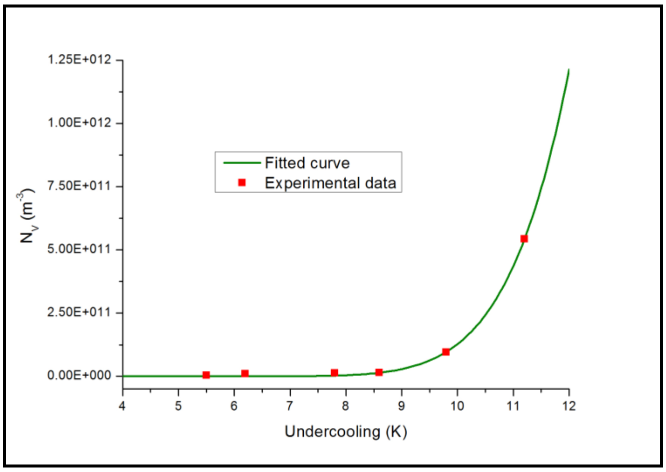

Fig. 7. N_V on undercooling dependence for 76 µm particles.

After more complicated statistical analysis that takes into account undercooling and mean particle diameter the global equalition can be derived:

$$N_V(T, d_{SiC}) = 9.1 \cdot 10^{14} \exp\left(3.1 \cdot 10^4 - \frac{32.6 + 1.4 \cdot 10^6 d_{SiC}}{T_N - T}\right), \qquad R^2=0.932.$$

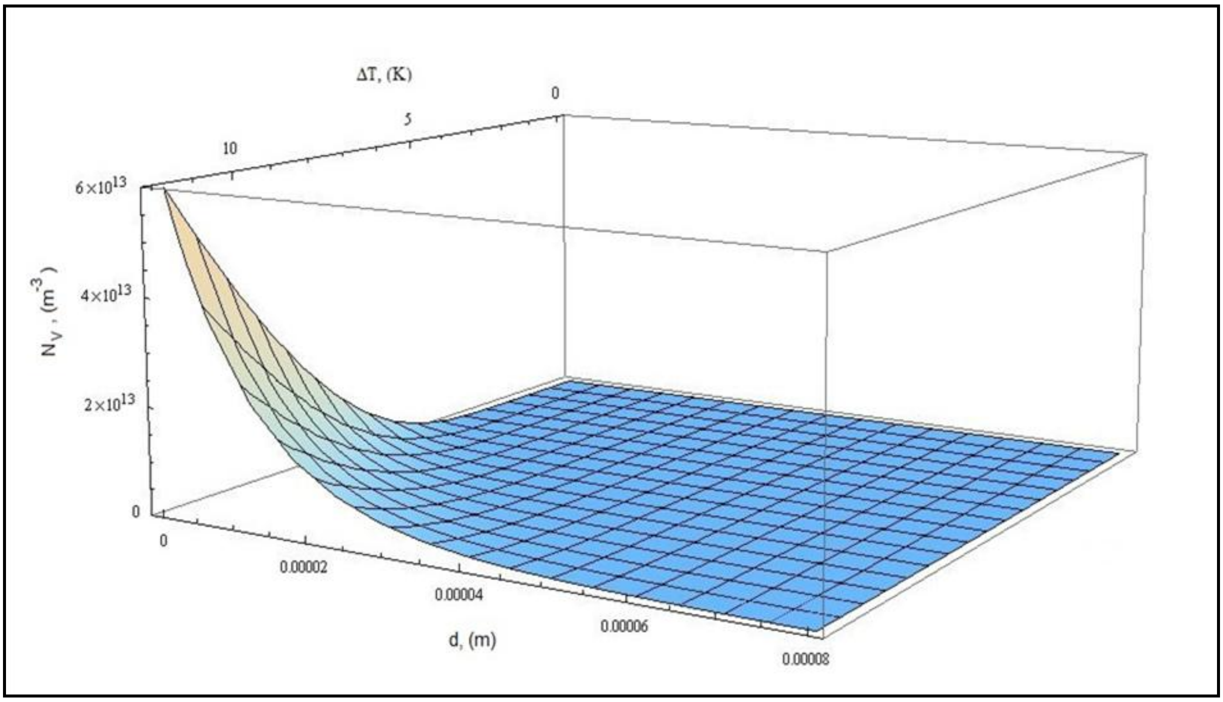

Fig. 8. N_v dependence on undercooling and mean grain diameter.

8. CONCLUSIONS

The experimental data can be used to prepare micro-macro composite crystallization model. SiC particles have refinement effect on the composite microstructure. The smaller particles give finer microstructure. The biggest change of the grain density can be observed in composite versus pure AZ91 alloy, even if the amount of the particles is not so large. Ethanol and Acetic Acid based solute is suitable for colour etching of AZ91/SiC composite. The particles content in composite has positive impact on composite microstructure.

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9. REFERENCES

[1] Asthana R.: Solidification processing of reinforced metals. Key Engineering materials 151-152 (1998).
[2] Luo A.: Heterogeneous nucleation and grain refinenent in cast Mg(AZ91)/SiC metal matrix composites. Canadian Metallurgical Quaretly 35 (1996), 375-383
[3] Lelito J., Zak P., Suchy J.S.: The grain nucleation rate of AZ91/SiC composite based on Maxwell-Hellawell model. Archives of Metallurgy and Materials 54 (2009), 347-350
[4] Kurz W., Fisher D.J., Fundamentals of Solidification, Zurich.Trans Tech Publications, 1992.
[5] Gorny M., Badania modelowe zarodkowania ziaren eutektycznych w żeliwie i ich weryfikacja doświadczalna. PhD thesis, Krakow 2001 [in polish]
[6] Fras E., Wiencek K., Gorny M., Lopez H.: Theoretical model for heterogeneous nucleation of grains during nucleation. Material Science and Technology 19 (2003), 1653-59.
[7] Maltais A., Dube D., Fiset M., Laroche G., Tugeon S.: Improvements in the metallography of as-cast AZ91 alloy. Materials Characterization 52 (2004), 103-119.
[8] Maltais A., Dube D., Roy F., Fiset M.: Optical anisotropy of a color-etched AZ91 magnesium alloy. Materials Characterisation 54 (2005), 315-326.
[9] Zak P., Lelito J., Shumcher P., Haberl K., Krajewski W., Suchy J.S.: Color etching of AZ91/SiC composite. XXXIII Konferencja naukowa z okazji Święta Odlewnika, Kraków, Poland 11 grudnia 2009. http://www.odlew.agh.edu.pl/inne/konferencje/dzien_odlewnika_2009.html.
[10] Cybo J., Jura S.: Funkcyjny opis struktur izometrycznych w metalografii ilościowej [in Polish]. Wydawnictwo Politechniki Śląskiej, Gliwice,Poland 1995.