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:

\[
\frac{\partial T}{\partial t} = \frac{1}{\varrho c_p} \text{div}(-\lambda \text{grad} T) + \frac{q}{\varrho c_p},
\]

where: \( T [K] \) – is temperature; \( t [s] \) – time; \( c_p [J \text{ kg}^{-1} \text{ K}^{-1}] \) – specific heat; \( \varrho [\text{kg m}^{-3}] \) – density; \( \lambda [\text{W m}^{-1} \text{ K}^{-1}] \) – thermal conductivity; \( q = L (df/dt) [\text{W kg}^{-1}] \) – heat of crystallization.
$q$ - is a parameter determining the amount of heat evolved in the crystallization process, and is a function of $f$, 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

<table>
<thead>
<tr>
<th></th>
<th>Al</th>
<th>Zn</th>
<th>Mn</th>
<th>Fe</th>
<th>Be</th>
<th>Si</th>
<th>Cu</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>8.5</td>
<td>0.64</td>
<td>0.23</td>
<td>&lt;0.002</td>
<td>10 ppm</td>
<td>0.03</td>
<td>0.003</td>
<td>0.001</td>
</tr>
</tbody>
</table>

3. NUCLEATION RATE

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

- Oldfield nucleation model [4,5]:
  $$N_v(T) = \psi(T_N - T)^m \left[ m^{-3} \right]$$  (2)

- Fras nucleation model [6]:
  $$N_v(T, d_{SiC}) = \lambda(d_{SiC}) \exp\left(\frac{\lambda d_{SiC}}{T_N - T}\right)$$  (3)

where: $T$ [K] is current temperature; $d_{SiC}$ [m] is the particles mean diameter; $\lambda$ [m$^{-3}$], $b$ [K] denotes model adjustment parameters; $T_N$ – is nucleation temperature, $\psi$, $m$ – nucleation 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

<table>
<thead>
<tr>
<th>CASTING CONDITIONS</th>
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<tbody>
<tr>
<td>Casting symbol</td>
<td>A</td>
<td>B</td>
</tr>
<tr>
<td>Particles size, [μm]</td>
<td>10</td>
<td>40</td>
</tr>
<tr>
<td>AZ91 mass, [g]</td>
<td>5960</td>
<td>6250</td>
</tr>
<tr>
<td>Ambient temperature, [°C]</td>
<td>22</td>
<td>22</td>
</tr>
<tr>
<td>Furnace temperature, [°C]</td>
<td>750</td>
<td>750</td>
</tr>
<tr>
<td>Particles temperature, [°C]</td>
<td>320</td>
<td>320</td>
</tr>
<tr>
<td>In-mould temperature, [°C]</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>Stirring time, [s]</td>
<td>240</td>
<td>180</td>
</tr>
</tbody>
</table>

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 Malais [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.

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

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;
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]:

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

where: $N_v$ [m$^{-3}$] is mean volumetric grain density, $N_a$ [m$^2$] is mean grain density on the surface, and $(1/d)_{\text{mean}}$ [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\textsubscript{V} on undercooling dependence for 10 \textmu m particles:**

\[ N\textsubscript{V} (T) = 1.7 \cdot 10^{14} \exp \left( \frac{43.21}{T - T_{N}} \right), \quad R^2 = 0.993, \]

![Graph of N\textsubscript{V} on undercooling dependence for 10 \textmu m particles.](image)

**N\textsubscript{V} on undercooling dependence for 40 \textmu m particles:**

\[ N\textsubscript{V} (T) = 3.9 \cdot 10^{15} \exp \left( \frac{93.53}{T - T_{N}} \right), \quad R^2 = 0.984, \]

![Graph of N\textsubscript{V} on undercooling dependence for 40 \textmu m particles.](image)
$N_v$ on undercooling dependence for 76 $\mu$m particles:

$$N_v(T) = 9.4 \cdot 10^{16} \exp\left(\frac{13509}{T_v - T}\right), \quad R^2 = 0.999.$$  

Fig. 7. $N_v$ on undercooling dependence for 76 $\mu$m particles.

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

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

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.

Acknowledgements

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9. REFERENCES