Homogeneity of particle size in the space of composite suspension casting

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Abstract
The presented analysis of the reinforcement particle size homogeneity in the space of composite casting is made by means of descriptive statistics methods and the analysis of variance. The reinforcement phase consisted of SiC particles with 15% content, while the matrix was an AlSi11 alloy. The composites were made by the mechanical stir casting method.

Key words: composite casting, homogeneity, analysis of variance.

1. Introduction

The reinforcement structure in composite materials is characterized by non-homogenous distribution in the casting volume as well as non-homogenous shape and size of the particles [1–5].

Therefore, this author decided to define a new characteristic of composite castings only, i.e. structure homogeneity. The concept of structure inhomogeneity (there are different versions defining it in the literature) includes the following features [3–4, 6–7]:

- deviation of some of its geometrical features from the structure conventionally adopted as homogenous;
- local disturbance of the structure, the intensity of which occurs with varying probability;
- variety of geometrical features of the measured elements resulting from their orientation (anisotropy) or position (gradient) in the examined specimen.

This author deals with the determination and description of quality parameters and defects (deviations from these parameters) of composite castings. In their case, it is right to use the concept of structure homogeneity, so that deviations from this feature, or defects, such as structure inhomogeneity, will refer to the quantity, distribution or size of reinforcement particles. This work introduces a statistical method of describing the homogeneity of the reinforcement particle size [8–9].

2. Research

In order to carry out an analysis of reinforcement particle size homogeneity in the casting volume, the area surfaces as shown in Figure 1 were examined. Four areas were prepared – hereinafter called samples – one model area denoted as M in diagrams and tables and three sample areas marked 1, 2, 3 (points of sampling for the analysis as per Figure 2). All the variables describing the reinforcement phase in the areas examined (Fig. 1) were calculated by means of the computer program Metilo [10] for image analysis, while the description and relationships between these variables are presented using STATISTICA PL software [11].
Fig. 1. Areas for an analysis of reinforcement structure homogeneity in the casting space. M – model area, 1,2,3 – areas of sampling for the analysis as per Fig. 2. Composite suspension casting: reinforcement – SiC particles, matrix: silumin, surface area ×600 (SEM)

Fig. 2. Areas of sampling for the analysis of the reinforcement structure homogeneity in the casting space

The assessment of particle size homogeneity made use of descriptive statistics methods and the ANOVA analysis for two variables describing the particle size, that is:

variable X – surface area of a particle in square micrometres [µm²]; and

variable Y – particle diameter in micrometres [µm].

With a variety of descriptive statistics to choose from, it was difficult to select the most reliable parameter that would allow to assess the sample homogeneity. By grouping these parameters in terms of the of measures and dispersion, we can assess the homogeneity in these two categories [3, 12–17].

Table 1 below includes basic parameters of descriptive statistics and confidence levels for the arithmetic mean and standard deviation at confidence coefficient 95% for the variables: particle surface area [µm²] and particle diameter [µm].

<table>
<thead>
<tr>
<th>Variable</th>
<th>Sample M</th>
<th>Sample 1</th>
<th>Sample 2</th>
<th>Sample 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>X – particle surface area in square micrometres</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample size</td>
<td>69</td>
<td>98</td>
<td>82</td>
<td>66</td>
</tr>
<tr>
<td>Mean surface area of particle</td>
<td>58.2</td>
<td>49.2</td>
<td>69.7</td>
<td>76.7</td>
</tr>
<tr>
<td>Lower bound of 95% confidence interval for the mean</td>
<td>55.4</td>
<td>39.9</td>
<td>57.5</td>
<td>60.8</td>
</tr>
<tr>
<td>Upper bound of 95% confidence interval for the mean</td>
<td>61.0</td>
<td>58.5</td>
<td>81.9</td>
<td>92.6</td>
</tr>
<tr>
<td>Median</td>
<td>57</td>
<td>35.5</td>
<td>53</td>
<td>58.5</td>
</tr>
<tr>
<td>Mode</td>
<td>54</td>
<td>6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mode size</td>
<td>7</td>
<td>6</td>
<td>3</td>
<td>4</td>
</tr>
<tr>
<td>Total</td>
<td>4015.5</td>
<td>4821.6</td>
<td>5716.5</td>
<td>5063.5</td>
</tr>
<tr>
<td>Minimum</td>
<td>30</td>
<td>2.5</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>Maximum</td>
<td>78</td>
<td>243</td>
<td>268</td>
<td>324</td>
</tr>
<tr>
<td>Lower quartile</td>
<td>51</td>
<td>18</td>
<td>31</td>
<td>29</td>
</tr>
<tr>
<td>Upper quartile</td>
<td>68.5</td>
<td>64</td>
<td>86</td>
<td>102</td>
</tr>
<tr>
<td>Standard deviation</td>
<td>11.50</td>
<td>46.40</td>
<td>55.62</td>
<td>64.63</td>
</tr>
<tr>
<td>Lower bound of 95% confidence interval for the standard deviation</td>
<td>9.85</td>
<td>40.69</td>
<td>48.22</td>
<td>55.18</td>
</tr>
<tr>
<td>Upper bound of 95% confidence interval for the standard deviation</td>
<td>13.82</td>
<td>53.99</td>
<td>65.73</td>
<td>78.02</td>
</tr>
<tr>
<td>Coefficient of variation</td>
<td>20%</td>
<td>94%</td>
<td>80%</td>
<td>84%</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Y – particle diameter in micrometres</th>
<th>Sample M</th>
<th>Sample 1</th>
<th>Sample 2</th>
<th>Sample 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample size</td>
<td>69</td>
<td>98</td>
<td>82</td>
<td>66</td>
</tr>
<tr>
<td>Mean</td>
<td>8.6</td>
<td>9.6</td>
<td>11.0</td>
<td>10.6</td>
</tr>
<tr>
<td>Lower bound of 95% confidence interval for the mean</td>
<td>8.3</td>
<td>8.6</td>
<td>9.9</td>
<td>9.4</td>
</tr>
<tr>
<td>Upper bound of 95% confidence interval for the mean</td>
<td>9.0</td>
<td>10.7</td>
<td>12.2</td>
<td>11.8</td>
</tr>
<tr>
<td>Median</td>
<td>9.0</td>
<td>8.2</td>
<td>10.0</td>
<td>10.0</td>
</tr>
<tr>
<td>Mode</td>
<td>9</td>
<td>8</td>
<td>6</td>
<td>11</td>
</tr>
<tr>
<td>Mode size</td>
<td>19</td>
<td>12</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Total</td>
<td>595.0</td>
<td>944.1</td>
<td>904.5</td>
<td>697.0</td>
</tr>
<tr>
<td>Minimum</td>
<td>6</td>
<td>1.5</td>
<td>3</td>
<td>4</td>
</tr>
<tr>
<td>Maximum</td>
<td>11</td>
<td>35</td>
<td>26</td>
<td>25</td>
</tr>
<tr>
<td>Lower quartile</td>
<td>8</td>
<td>6</td>
<td>7</td>
<td>7</td>
</tr>
<tr>
<td>Upper quartile</td>
<td>9.5</td>
<td>13</td>
<td>13</td>
<td>12</td>
</tr>
<tr>
<td>Standard deviation</td>
<td>1.37</td>
<td>5.31</td>
<td>5.18</td>
<td>4.92</td>
</tr>
<tr>
<td>Lower bound of 95% confidence interval for the standard deviation</td>
<td>1.17</td>
<td>4.66</td>
<td>4.49</td>
<td>4.20</td>
</tr>
<tr>
<td>Upper bound of 95% confidence interval for the standard deviation</td>
<td>1.65</td>
<td>6.18</td>
<td>6.12</td>
<td>5.94</td>
</tr>
<tr>
<td>Coefficient of variation</td>
<td>15.9%</td>
<td>55.2%</td>
<td>47.0%</td>
<td>46.6%</td>
</tr>
</tbody>
</table>

* multiple mode – means that the most frequent value occurs several times.
To visualize the parameters of descriptive statistics relating to the range of measures of the variable position we use graphs called ‘box plots’, where the box represents the range from \( Q_1 \) – first quartile to \( Q_3 \) – third quartile, that is from 25% to 75% of the feature variation, while the whiskers indicate the range of non-outlying values of the feature. The outlying values are the feature values not belonging to the interval \([Q_1-1.5 \times H, Q_3+1.5 \times H]\), where \( H = Q_3 - Q_1 \), while extreme values are regarded to be those values lying outside the interval \([Q_1-3 \times H, Q_3+3 \times H]\).

Thanks to the box-and-whisker diagrams you can compare the position of variables \( X \) – particle surface area [\( \mu \text{m}^2 \)] and \( Y \) – particle diameter [\( \mu \text{m} \)] in each sample with the model sample.

![Fig. 3. Visual representation of position measures for the variable ‘particle surface area’ based on Table 1, box plot [8, 12, 17], made by means of [11]](image)

From an analysis of the diagram in Figure 3 we can see that each sample contains one extreme value, sample 3 has three outlying values, while samples 1 and 2 include five outlying values each, that is in terms of the variable ‘particle surface area’ the sample 3 is characterized by least inhomogeneity.

![Fig. 4. Visual representation of position measures for the variable ‘particle diameter’ based on Table 1, box plot [8, 12, 17], made by means of [11]](image)

The diagram 4 above relating to the variable ‘particle diameter’ leads to a conclusion that only sample 1 contains an extreme value, and samples 2 and 3 contain three outlying values each, i.e. for this variable the sample 3 again has the least non-homogeneity 3.

The classical coefficient of variation is a reliable parameter for the assessment of variable dispersion. Its mathematical formula is this [8, 12]:

\[
V(x) = \frac{s(x)}{\bar{x}} \times 100\%
\]

where \( s(x) \) – standard deviation of the variable \( x \), \( \bar{x} \) – arithmetical mean of the variable \( x \).

The coefficient of variation [8, 15] of particle size was determined from Table 1 and is shown in Table 2.

<table>
<thead>
<tr>
<th>Classical coefficient of variation</th>
<th>Sample M</th>
<th>Sample 1</th>
<th>Sample 2</th>
<th>Sample 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Variable ( X ) – particle surface area in square micrometres</td>
<td>19.8%</td>
<td>94.3%</td>
<td>79.8%</td>
<td>84.2%</td>
</tr>
<tr>
<td>Variable ( Y ) – particle diameter in micrometres</td>
<td>15.9%</td>
<td>55.2%</td>
<td>47.0%</td>
<td>46.6%</td>
</tr>
</tbody>
</table>

If we examine the coefficients of variation for each sample, the conclusion follows that in terms of particle size the sample 1 varies the most from the model sample.

In order to test the sample homogeneity we used a one-way ANOVA analysis, that allows to determine the differentiation of a given variable from the grouping factor.

The ANOVA analysis was performed for the two variables: variable \( X \) – particle surface area [\( \mu \text{m}^2 \)] and variable \( Y \) – particle diameter [\( \mu \text{m} \)], while the grouping factor was the type of sample denoted as 1, 2, 3 and \( M \).

In the ANOVA analysis we verify the null hypothesis that the grouping factor does not differentiate the mean value of a given variable, i.e.:

\[
H_0 : m_{X_1} = m_{X_2} = \ldots = m_{X_M}
\]

against an alternative hypothesis \( H_1 \): not all means are equal.

The test statistic \( F \) is calculated. If the grouping factor does not differentiate the variable, then the value \( F \) is close to 1; if \( F \) is larger than 1, the null hypothesis is rejected and the alternative hypothesis is accepted at the set level of test significance \( \alpha \). The test significance level \( \alpha \) is a probability that the true hypothesis will be rejected.

The program STATISTICA PL was used to calculate the test statistics \( F \) and critical confidence levels of tests for each variable.

The test for the variable \( X \) – particle surface area in square micrometres.

\[
H_0 : m_{X_1} = m_{X_2} = m_{X_3} = m_{X_M}
\]

\( H_1 \): not all means are equal.
Another problem is the assessment of the degree of inhomogeneity of each sample. To this end, the ANOVA analysis was used for comparing the mean values of variables of individual samples with the mean values of these variables of the model sample.

Tests for the sample 1:

$H_0 : m_{X_i} = m_{XM}$

$H_1 : m_{X_i} \neq m_{XM}$

The value of test statistic $F$ and the critical level of probability $p$ are given in the diagram. (Fig. 7)

At the significance level $\alpha = 0.05$ the null hypothesis on equal mean values should be rejected in favour of the alternative hypothesis.

$H_0 : m_{X_i} = m_{XM}$

$H_1 : m_{X_i} \neq m_{XM}$

The value of test statistic $F$ and the critical level of probability $p$ are given in the diagram. (Fig. 8)

The analysis of variance has shown that examining the particle surface area and diameter we can observe that the samples 1, 2 and 3 have inhomogeneous particles size and differ from the model sample.
At the significance level \( \alpha = 0.05 \) the null hypothesis on equal mean values should be rejected in favour of the alternative hypothesis.

Test for the sample 2:

\[ H_0 : m_{X_2} = m_{X_M} \]
\[ H_1 : m_{X_2} \neq m_{X_M} \]

The value of test statistic F and the critical level of probability \( p \) are given in the diagram (Fig. 9).

At the significance level \( \alpha = 0.05 \) the null hypothesis on equal mean values should be rejected in favour of the alternative hypothesis. This means that the sample 2 differs statistically from the model sample in terms of particle diameter.

Test for sample 3:

\[ H_0 : m_{Y_2} = m_{Y_M} \]
\[ H_1 : m_{Y_2} \neq m_{Y_M} \]

The value of test statistic F and the critical level of probability \( p \) are given in the diagram (Fig. 10).

At the significance level \( \alpha = 0.05 \) the null hypothesis on equal mean values should be rejected in favour of the alternative hypothesis. This means that the sample 2 differs statistically from the model sample in terms of particle diameter.

Test for sample 3:

\[ H_0 : m_{Y_3} = m_{Y_M} \]
\[ H_1 : m_{Y_3} \neq m_{Y_M} \]

The value of test statistic F and the critical level of probability \( p \) are given in the diagram (Fig. 11).

At the significance level \( \alpha = 0.05 \) the null hypothesis on equal mean values should be rejected in favour of the alternative hypothesis. This means that the sample 3 differs statistically from the model sample in terms of particle diameter.

Test for sample 3:

\[ H_0 : m_{Y_3} = m_{Y_M} \]
\[ H_1 : m_{Y_3} \neq m_{Y_M} \]

The value of test statistic F and the critical level of probability \( p \) are given in the diagram (Fig. 12).
At the significance level $\alpha = 0.05$ the null hypothesis on equal mean values should be rejected in favour of the alternative hypothesis.

Using the ANOVA analysis for the assessment of particle size inhomogeneity in samples we can adopt the value of test statistic $F$ as a coefficient determining the degree of inhomogeneity. The relevant data are given in Table 3.

Table 3.
The value of test statistic $F$ – coefficient $F$, defining the degree of particle size inhomogeneity in the examined samples compared to the model sample

<table>
<thead>
<tr>
<th>Coefficient $F$ – test statistic $F$</th>
<th>Sample 1</th>
<th>Sample 2</th>
<th>Sample 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Variable $X$ – particle surface area in square micrometres</td>
<td>21.09</td>
<td>2.85</td>
<td>5.49</td>
</tr>
<tr>
<td>Variable $Y$ – particle diameter in micrometres</td>
<td>30.18</td>
<td>40.38</td>
<td>24.36</td>
</tr>
</tbody>
</table>

The greater is the value of coefficient $F$, the more inhomogeneous the sample is. Considering the particle surface area, the sample 2 is least inhomogeneous, while in terms of variable particle diameter, the sample 3 is least inhomogeneous.

With both criteria taken into account, the sample 1 turns out to be the most inhomogeneous.

3. Conclusions

The foregoing analysis of the homogeneity of reinforcement particle size in the space of composite suspension casting carried out by means of descriptive statistics methods and the analysis of variance introduces a description of this composite component, which will significantly contribute to the quality improvement of examined materials.

Literature