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Structure homogeneity as a parameter for evaluation of composite casting quality

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Abstract

The structure of composite materials is be usually described as a compound of two structural components called matrix and reinforcement, respectively. A classic, commonly known example is polyester resin reinforced with glass fibres. Composite materials obtained through casting techniques are frequently characterised by irregular distribution and content of reinforcement in the casting volume as well as by different shape and size of this structural element [1–5]. It clearly results from the fundamentals of materials engineering that this type of structural diversity has a crucial effect on its broadly understood properties. Therefore, a need arises to define in a simple but precise way what we understand as homogeneity or non-homogeneity of the material, as well as for introduction of measures for this feature. The present study is limited to cast metal-matrix composite materials that, due to their manufacture technology, are particularly susceptible to the occurrence of non-homogeneity. However, the proposed solution may be also applied in characterisation of other materials.
Till now, the concept of non-homogeneity has no commonly accepted definition [6-10]. Among others, it is defined as:

- deviation of certain geometric features from the structure accepted conventionally as homogenous;
- local structure disorder, the intensity of which is accomplished with different probability;
- derivative of the diversity of geometric features of measured elements which results from their orientation (anisotropy) or position (gradient) in a tested object.

In the case of composite castings when the concept of defect as deviation from the desired features is being used as a rule in describing the quality parameters of these materials, it seems to be advisable to introduce the concept of material homogeneity. Deviation from this feature, i.e. a defect, will be the non-homogeneity of, for instance, structure porosity or amount, spatial distribution, size or shape of reinforcing phase precipitations. This paper presents a proposal for complex determination of reinforcement structure homogeneity along with its practical application.

Keywords: homogeneity, reinforcement, casting, metal-matrix composites

1. Introduction

For the needs of developing the estimators of basic stereological parameters, it is being assumed that analysed structure of material is non-oriented, homogeneous and random. This happens when each optional cut section of this structure shows the same values for all parameters of its quantitative description. Such an implicit definition of broadly understood homogeneity (here, the concept of homogeneity is one of three elements characterising the structure) is good from the point of view of philosophy but not much useful for the practice what will be presented in the further part of this paper on the example of reinforcement structure homogeneity of a casting.

For the needs of further considerations, it has been assumed that the structure shows features of randomness and lack of orientation, with search for strict mathematical evidence of the fulfilment of these features being omitted.

The following definitions of homogeneity and its measures are being proposed:

Homogeneity as a feature is the property of material structure being characterised by showing no statistically significant differences in different places of the examined structure. It is assumed that homogeneous materials usually have better performance properties than non-homogeneous ones [2-5,7].

Homogeneity scale is the size of tested area used for examining it. Homogeneity scale should be adjusted to analysed application of a material. For example, in the case of a mono-crystal designed for production of integrated circuits the homogeneity scale may be the volume of crystal cut for the needs of producing such one circuit.

Homogeneity feature (measure) is a structural parameter used for examining it. Homogeneity feature may be the quantity (volume fraction), size, shape or orientation of phase precipitations and pores, or other structural components.

Local homogeneity (or: microscopic scale homogeneity) is the homogeneity analysed within one area corresponding with its size to the homogeneity scale.

Global homogeneity (or: macroscopic scale homogeneity) is the homogeneity analysed within an area being extensively larger than the homogeneity scale. Global homogeneity can be both similar and clearly different from local homogeneity. For example, porous microfilters cut from a large block may contain highly size-variable pores. Therefore locally, on a scale of one microfilter, its material will not be homogeneous with respect of the pore size. However, if all microfilters cut from the same block of material show the same distribution of pore size, the material will be globally homogeneous.

It is worthy noticing that a feature opposite to homogeneity, i.e. non-homogeneity, is very easy to define and characterise in the light of definitions mentioned above. A material, the structure of which is not homogeneous from the point of view of certain criteria, can be considered as non-homogeneous for these criteria. Sample results of the examinations aimed at evaluation of the homogeneity of cast metal-matrix composite materials are presented in the further part of this paper.

2. Examples of homogeneity analysis

Let us take into account a two-phase material – it may be, for instance, a solid solution with pores, a single-phase matrix with reinforcement, a mixture of two phases, etc. Irrespectively of the degree of fineness of the phases composing this material, we can come – at least theoretically, but also practically when using appropriate tools – to such a magnification when observing the structure of this material that the structure image will present only one of the analysed phases. Therefore, we will have an impression of observing a single-phase material although in fact it is a two-phase one. This way, a fundamental conclusion is being arrived at that **any material becomes non-homogeneous if its structure is analysed at an adequately large magnification**.

There are no fixed and simple criteria for selection of the size of test area for evaluation of homogeneity or non-homogeneity. In the case of analysis of carbon steel bar soft skin, a change in ferrite linear fraction can be examined along the family of lines parallel to the bar surface [5, 10]. In such a case, the size of analysed area will in fact correspond to the real distance between adjacent test lines and can be changed almost freely. Classic evaluation of the homogeneity of ingots was reduced to the analysis of three locations: lower, middle and upper parts of the ingot [7, 9–10]. This was completely enough for practical needs. Increase in the number of analysed areas and corresponding reduction of their volume (or surface in 2-D analysis) causes significantly larger scatters of measurement results. It should be kept in mind that in the statistical analysis two results can be considered as significantly different from each other if the difference between arithmetical means is at least equal to doubled standard deviation [9] (this is of course a rough, practically qualitative evaluation but it shows the problem's essence). Excessive reduction of the area of analysis may lead to completely worthless results, which is presented in Table 1. It contains results of composite structure examination using systematic scanning method [8, 11–12]. For measurement frames from 2×2 in pixels to 9×9 (and for direction Y 12×12), the values of F-statistics (bold) exceed the critical value $F_{0.05}$. This means that with so small measurement frames the hypothesis about non-significant effect of rows and columns, so the hypothesis about uniform distribution of SiC particles should be rejected [7–8, 13–15]. Starting with the measurement frame size of 14×14, there are no grounds to reject the hypothesis about significant effect of rows and columns, i.e. about the occurrence of anisotropy (irregular distribution of SiC particles) from the point of view of two mutually perpendicular directions of the analysis.

The rejection of hypothesis in the case of "small" measurement frames is a consequence of excessive variation of the surface fraction A_A of SiC particles between successive fields of the analysis (frames). The SiC particles are not found in part of "small" frames, so the surface fraction amounted in them to zero. To avoid this effect, it is recommended to use measurement frames starting with 14×14 ones. This analysis refers to selection of measurement frame size but not magnification. The values presented in Table 1 were recorded at 200× magnification. With 500× magnification and when preserving similar "small" frames, the effect is even worse [16]. Part of the frames is being totally filled by the examined particles, so their surface fraction is 1.0 (i.e. 100%). This induces even larger variation between frames and the occurrence of strong anisotropy (even with frames larger than 14×14).

Generally, as large analysis areas as possible should be selected and a common sense should be absolutely used (which is a very imprecise criterion, difficult to be verified scientifically, especially by engineers). The upper limit of size is always determined by the study objective; after exceeding it, the results become useless. The lower limit is the scatter of results discussed above. In practice, our attention is primarily focused on the upper limit, with checking only if its size is not below the lower limit, that would undermine the reliability of study results.

Table 1.
Results of the evaluation of SiC/AlSi9 composite segregation with systematic scanning at 200× magnification

Frame size in pixels	A_A	$CV(A_A)$	$\hat{s}_X^2$	$\hat{s}_Y^2$	$\hat{s}_R^2$	$F_X = \hat{s}_X^2 / \hat{s}_R^2$	$F_Y = \hat{s}_Y^2 / \hat{s}_R^2$	$\eta = F_X / F_Y$	$F_{0,05}$
2×2	**0.2456**	**1.6504**	**0.9413**	**1.0971**	**0.1576**	**5.9727**	**6.9614**	**1.1655**	**1.231**
3×3	**0.2459**	**1.5779**	**0.6170**	**0.7098**	**0.1444**	**4.2726**	**4.9152**	**1.1504**	**1.291**
4×4	0.2456	1.5175	0.4507	0.5310	0.1334	**3.3796**	**3.9816**	1.1781	1.344
6×6	0.2459	1.3992	0.2829	0.3285	0.1139	**2.4844**	**2.8851**	1.1613	1.438
7×7	0.2457	1.3555	0.2420	0.2767	0.1068	**2.2651**	**2.5907**	1.1438	1.482
9×9	0.2472	1.2569	0.1842	0.1971	0.0931	**1.9776**	**2.1161**	1.0700	1.564
12×12	0.2472	1.1355	0.1226	0.1364	0.0763	1.6067	**1.7869**	1.1122	1.682
14×14	0.2472	1.0673	0.1066	0.1166	0.0672	1.5854	1.7340	1.0938	1.757
18×18	0.2472	0.9588	0.0782	0.0849	0.0543	1.4399	1.5624	1.0851	1.905
21×21	0.2472	0.8787	0.0532	0.0657	0.0461	1.1527	1.4237	1.2352	2.014
28×28	0.2472	0.7461	0.0373	0.0456	0.0332	1.1254	1.3756	1.2223	2.272
36×36	0.2472	0.6597	0.0289	0.0336	0.0259	1.1162	1.2995	1.1642	2.577

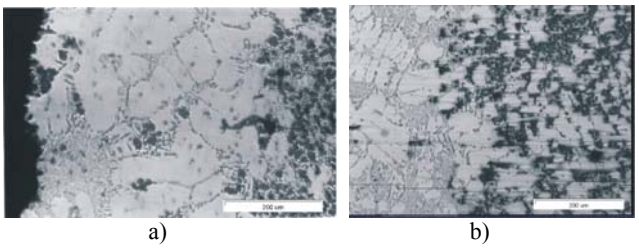
where:
A_A – surface fraction,
$CV(A_A)$ – coefficient of variation of the surface fraction,
$\hat{s}_X^2$ – variance of the surface fraction (variation between rows – direction X),
$\hat{s}_Y^2$ – variance of the surface fraction (variation between columns – direction Y),
$\hat{s}_R^2$ – residual variance,
F_X i F_Y – values of F-statistics in directions X and Y,
η – anisotropy index,
$F_{0,05}$ – critical value at a significance level of 0.05.

However, the size of elementary space is variable and depends first of all on the potential application of material under analysis. For example, abrasive paper for rough surface processing can be considered as homogeneous even though there are abrasive grains in it with a diameter of the order of one millimetre. On the other hand, such large objects are completely unacceptable in the abrasive paper designed for the last grinding of kerbs preceding the polishing.

Importance of the problem of selecting the size of analysis area has found its illustration in the presented concept of homogeneity definition in the form of homogeneity scale parameter.

The necessity of determining the size of area, in which homogeneity is being analysed, is not the only problem when evaluating this feature of the structure. It is being assumed that for the needs of material evaluation from the point of view of stereology [16] it is necessary and enough to determine four features for each analysed phase, i.e. quantity (understood rather as total volume than number), size, shape and distribution of separations or other structural components. Each of these features may show homogeneity or non-homogeneity. Thus, we have as follows:

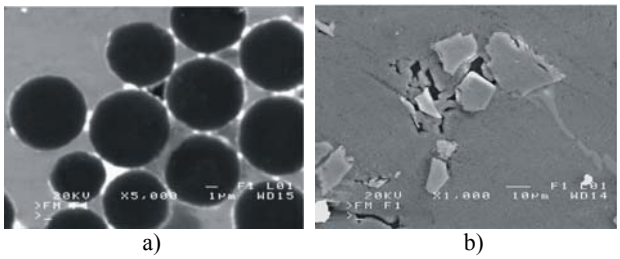
- **Quantity homogeneity** – an example of deviation from it can be a well-known tendency of impurities to group in these casting areas that are solidified as the latest ones or the non-homogeneous quantity of reinforcement phase in the casting space (Fig. 1).



a) b)

Fig. 1. Quantity non-homogeneity of reinforcement phased in the composite casting space. Suspension composite SiC/ AlSi7Mg: a) sedimentation of particles; light microscopy, b) dendritic segregation; light microscopy

- **Size homogeneity** – the great majority of precipitations in materials have a more or less diverse size (carbides in tool materials, non-metallic inclusions, single cells in foamed polystyrene, or size non-homogeneity of reinforcement phase on a given area (Fig. 2) or in the casting space, etc.), so these materials are usually non-homogeneous, at least on a micro scale.



a) b)

Fig. 2. Lack of the homogeneity of reinforcement phase in microareas, a) composite with saturated reinforcement, long boron fibre, titanium matrix; SEM, b) suspension composite (SiC reinforcement, AlSi9 matrix); SEM

If we assume the area of plane section as a measure of the size of reinforcement phase particles [6, 9-10, 12], it will be easy to determine the size homogeneity of this phase on a local scale by means of statistical methods [13–15]. Figure 3 presents the result of comparison of the plane section of reinforcement phase (from

ten analysis fields) evaluated with the Kruskal-Wallis test at a defined value $p > 0.05$ (axes X and Y – analysis fields of the plane section area of SiC reinforcement phase particles, axis Z – median). Table 2 comprises the result of Kolmogorov's test, where the normal distribution of analysed fields was verified.

The value $p > 0.05$ in the Kolmogorov's test is evidence of the lack o grounds to reject the null hypothesis, while no statistically significant differences in the median value suggest that the sizes of reinforcement phase separations within the examined area are homogeneous.

Table 2.
Quantitative description of the surface of place section of SiC/AlSi11 reinforcement phase particles

No. of analysis field	Meana μm^2	SD μm^2	Cv %	Min μm^2	Quartileyl_1 μm^2	Median μm^2	Quartile_3 μm^2	Max μm^2	Kolmogorov's test result
1	75.30	83.12	110.4%	1.15	16.69	45.42	103.38	426.92	**<0.01**
2	65.72	69.59	105.9%	1.32	20.49	47.98	85.98	480.85	**<0.01**
3	81.31	81.45	100.2%	1.05	24.03	64.20	111.14	488.04	**<0.01**
4	85.25	85.22	100.0%	1.55	21.61	58.02	114.97	441.99	**<0.05**
5	72.93	74.28	101.9%	1.01	24.58	45.50	96.66	318.62	**<0.01**
6	76.39	76.03	99.5%	1.08	18.31	51.90	108.72	404.41	**<0.02**
7	71.63	77.01	107.5%	1.35	19.53	43.32	101.04	429.92	**<0.01**
8	78.71	82.18	104.4%	1.05	19.46	42.68	106.04	405.43	**<0.01**
9	76.72	70.21	91.5%	1.01	24.63	57.14	105.61	304.22	**<0.05**
10	59.13	75.47	127.6%	1.15	10.36	37.90	77.26	514.81	**<0.01**

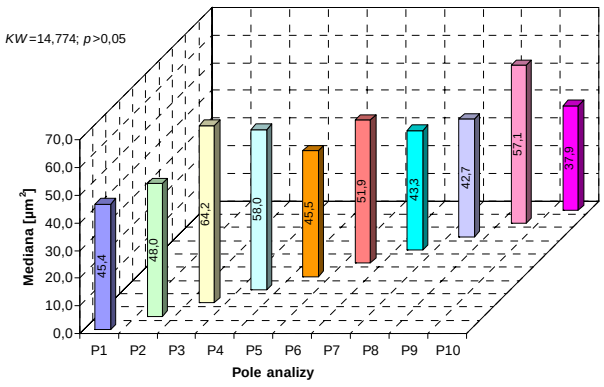


Fig. 3. The median values for the plane section area of reinforcement phase particles and the result of Kruskal-Wallis test (SiC/AlSi9 composite)

- **Shape homogeneity** – an example of deviation from it can be the incidence of degenerated nodular graphite in these parts of casting which remained too long in the liquid state, or the shape non-homogeneity of reinforcement phase on a given area (Fig. 4).

a) b)

Fig. 4. Shape non-homogeneity of reinforcement phase; a) suspension composite (SiC reinforcement, AlSi11 matrix); SEM, b) Al3Mg+N2 in-situ composite; light microscopy [17]

The shape of reinforcement phase precipitations can be described by means of dimensionless shape factor F_κ which is sensitive to a deviation from the circularity consisting in the folding of separation edge without elongation in one direction determined according to [6, 10, 14–15]:

$$F_\kappa = \frac{4 \cdot \pi \cdot A}{L^2} \tag{1}$$

where: A – area, and L – length of the plane section circumference of a measured object.

Results of the quantitative evaluation of reinforcement phase particles [12] in the composite casting space allowing for the shape factor (F_k) are presented in Table 3. Samples for the analysis were collected from the casting in three places: at the top (sample No. 1), in the middle (sample No. 2) and at the bottom (sample No. 3).

Table 3.
Results of the quantitative evaluation of reinforcement phase particles within the casting space

Sample No.	A_A [%]	$DNI(A_A)$ [%]	$\overline{A}$ [μm^2]	$DNI(A)$ [%]	N_A [mm^{-2}]	$DNI(N_A)$ [%]	F_κ	$DNI(F)$ [%]
1	20,0	12,0	177,3	91,7	1120	4,0	0,90	23,1
2	10,0	18,8	187,3	91,3	527	18,1	0,90	22,0
3	16,9	10,6	191,0	88,3	879	9,8	0,89	23,6

- **Distribution homogeneity** – an example of deviation from this feature can be diversity of the arrangement of fibres in composite materials, where largely parallel orientation of fibres to the direction of greatest loads is being aimed at.

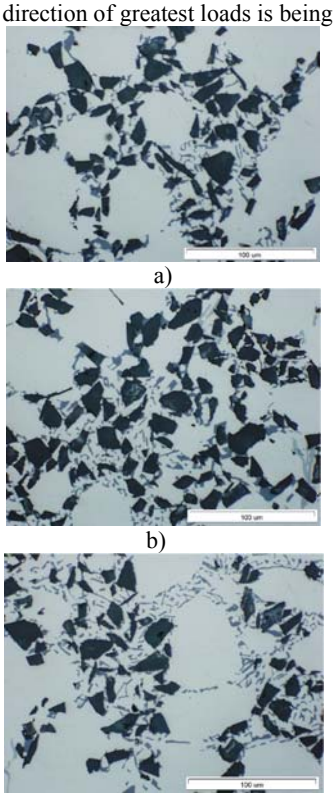


Fig. 5. Distribution non-homogeneity of reinforcement phase; a) sample No 1(table 3), b) sample No 2 (table 3), c) sample No 3 (table 3); suspension composite (SiC reinforcement, AlSi9 matrix)

Diversity of the distribution of particles (Fig. 5) on a given area is described by coefficient of variation, also called the distribution non-homogeneity index (DNI) [9, 12]. By definition, it is a classic measure of diversity of the distribution of features [14]. It is a relative measure dependent on the size of arithmetic mean [7, 9–10, 12, 14] and is being described by the following formula:

$$\mathrm{DNI} = \frac{\text{sample standard deviation}}{\text{sample arithmetic mean}} \cdot 100\,[\%]$$

The values of this index are presented in Table 3, where areas from different places of the casting were compared in order to describe the homogeneity of structure. Diverse distribution of the reinforcement phase in the casting space was found (the evidence of which is the DNI value; see the last column of Table 3).

Conclusions

1. The proposed definitions of the homogeneity concept as well as its measures may be successfully used for characterisation of cast metal-matrix composite materials.
2. Introduction of the concept of homogeneity scale does not present larger measuring difficulties but allows at the same time the adjustment of homogeneity evaluation procedures to particular application of the material under analysis.
3. Introduction of the concept of homogeneity measure (feature) corresponds to its complex character but allows at the same time its relatively easy quantitative characterisation.

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