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Contribution to quantification of highly inhomogeneous microstructures

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In highly inhomogeneous microstructures with a wide range of grain sizes, the problem arises of defining an effective average grain size. An original method for the estimation of effective average grain size and a parameter describing the homogeneity of microstructure are presented. The coefficients of homogeneity for equiaxed and columnar microstructures are calculated. The effective average grain size is calculated as a weighted sum of the average grain sizes of homogeneous grain groups.

1. Introduction

The microstructure of metals is often critical to their behaviour in some applications. It is necessary to be able to characterise microstructural parameters for predicting materials behaviour [1, 2]. Grain size of the material is an important engineering parameter which influences the material properties such as fatigue, creep, yield strength etc.

The planimetric method yields the number of grains per square millimeter area, from which can be calculate the average grain area [3]. It is common practice to take the square root of grain area and call this the grain diameter, although this assumes that the cross-sectional shape of the grain is a square, which it is not.

Test Methods E 112 [4], is chiefly concerned with the measurement of grain size when the grains are equiaxed in shape, although it does contain some information about measurement of grain size when the grains have been elongated by processing.

It is common to express grain sizes in terms of a simple exponential equation [4]:

$$n = 2^{(G-1)} \tag{1}$$

where: n—the number of grains per square inch at 100× magnification, and G—the ASTM grain size number.

Countries that used the metric system at that time developed an alternate equation that produces nearly identical grain size numbers:

$$m = 8(2^{Gm}) \tag{2}$$

where: m — the number of grains per mm^2 at magnification 100×, and Gm — the metric grain size number.

The coefficient Gm in (2) is slightly greater than G in (1), but the difference is negligible. Equation 2 is used in the Slovak [5], Russian [6], French [7], and Italian [8] standards.

The German standard also uses the metric system, but a different equation is employed [9]:

$$K = 3.7 + 3.33\mathrm{Log}(Z) \tag{3}$$

where: K = the photomicrograph serial number and Z = the number of grains per cm^2 at magnification 100×.

In modern non-oriented electrical steels stringent combinations of mechanical and magnetic properties are demanded. To achieve these, different technological approaches were developed that often result in a final microstructure with a high level of inhomogeneity. In microstructures with a wide grain size range, the problem arises of defining an effective average grain size. The evaluation methods described in above mentioned National Standards [3–9] are unsuitable for such microstructures.

Sevcik and Kohutek [10] explored various parameters for providing quantitative descriptions of inhomogeneous microstructures. Statistical parameters have been developed previously [11, 12] for the quantification of grain sizes in ferrite and austenite, respectively. However, only grain size for the homogeneous microstructure was evaluated.

DeHoff and others evaluated the grain size using a grain topology. It was shown that the topological complexity of a grain in a polycrystal is related to its diameter, as opposed to its area and volume [13–15].

Rhines evaluated the grain size utilising so-called "global parameters": length of line, area of surface, volume fraction, curvature of line and surface, etc.

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Topological parameters provide information about how many elements each feature of the structure is divided into and in how many places these elements are connected. These properties are dimensionless and depend in no way upon the scale of the microstructure [16].

Grain size measurement is complicated by a number of factors. First, the three-dimensional size of the grains is not constant and the sectioning plane will cut through the grains at random.

In most cases, the grains observed on a polished cross-sectional plane exhibit a range of sizes around a central mean and individual measurements of grain areas, diameters, or intercept lengths exhibit a normal distribution.

The different types of grain shapes that can be present in metals also complicate grain size measurement. For the most general case of irregular grains with varying shape, it is impossible to establish the true spatial distribution from planar or thin-section measurements. Bocksteigel [17] solved this problem by the assumption of a spherical geometry for the irregular grains. This model is based on the calculation of so-called "equivalent sphere-size distribution". However, the equivalent sphere-size distribution does not describe accurately real microstructures and, therefore, has limited applicability.

All methods proposed for the evaluation of the true spatial distribution of grains are restricted in some way. None of the above-mentioned methods are sufficiently representative nor give useful values of average grain size in the case of microstructures with a wide range of grain sizes.

This paper presents original methods for the estimation of the homogeneity of microstructures and establishment of the effective average grain size in highly inhomogeneous microstructures.

2. Experiment

Fully- and semi-processed non-oriented electrical steels were used as investigated material. The microstructure of the specimens was examined in the plane parallel to the sheet surface and in longitudinal cross-section using optical microscopy. Let b_i and c_i be the axes of the grains parallel to and perpendicular to the rolling direction in the plane of the sheet and a_i be the grain axis perpendicular to the rolling direction in longitudinal cross-section. In the microstructures under investigation, grains measurement in the sheet plane has shown that $b_i \approx c_i$, i.e., the microstructure in the sheet plane is equiaxed. If it is assumed that $b_i = c_i$, then the 3-D grain structure can be estimated from measurements of a_i and b_i made on longitudinal cross-sections.

Prior to analysis, the microstructure was traced manually to an acetate sheet and subsequently scanned. DIPS-5 image analysis software was used to make quantitative metallographic measurements. The software transforms the ith grain to an ellipse of similar eccentricity and unit area. The axes of the ellipse are A_i and B_i. The grain area S_i was estimated by the image analysis software, and the actual grain axes a_i and b_i calculated using the following equations:

$$a_i = \sqrt{S_i} A_i \tag{4}$$

$$b_i = \sqrt{S_i} B_i \tag{5}$$

3. Results and discussion

3.1. Microstructure homogeneity

The measurement of grain size is complicated by the different types of grain structures. So, it is important to characterize a microstructure in terms of its degree of homogeneity.

The typical examples of microstructure of non-oriented electrical steels are presented in Fig. 1. The microstructure of samples A and B have a uniform size distribution, and as a consequence, the grain diameter frequency histogram is a single peak and symmetrical in shape observed. However, the microstructure of samples C and D have more complex grain size distributions that will be discussed later.

Thus, within the non-oriented electrical steels under investigation, the following types of microstructure can be defined:

I Homogeneous microstructure (uniform grain size):

(a) equiaxed—aspect ratio approximately equal to 1 (Fig. 1A);
(b) non-equiaxed—microstructure with aspect ratio significantly greater than 1 (Fig. 1B);

II Inhomogeneous microstructure (non-uniform grain size):

(a) equiaxed microstructure with wide range of grain sizes (Fig. 1C);
(b) non-equiaxed microstructure with wide range of grain sizes (Fig. 1D).

The homogeneity of microstructure can be described using the following approach. The shape of each grain is approximated by an ellipsoid, Fig. 2. A coefficient of homogeneity for the 3-D microstructure G_v is defined as follows:

$$G_v = \frac{\bar{V}}{\frac{4}{3}\pi \bar{a}\bar{b}\bar{c}} = \frac{1}{N} \frac{\sum_{i=1}^{N} a_i b_i c_i}{\frac{\sum_{i=1}^{N} a_i}{N} \frac{\sum_{i=1}^{N} b_i}{N} \frac{\sum_{i=1}^{N} c_i}{N}}$$

$$= N^2 \frac{\sum_{i=1}^{N} a_i b_i c_i}{\sum_{i=1}^{N} a_i \sum_{i=1}^{N} b_i \sum_{i=1}^{N} c_i} \tag{6}$$

where: $\bar{V}$—average volume of a grain, N—total number of grains, a_i, b_i, c_i,—ith grain's axes.

If grains are of the same size and shape ($a_i = a$, $b_i = b$, $c_i = c$), then relationships exist between a, b and c:

$$a = lb = kc \tag{7}$$

where l and k are constants. Hence:

$$G_v = N^2 \frac{N \frac{a^3}{lk}}{N \cdot a \cdot N \cdot \frac{a}{l} \cdot N \cdot \frac{a}{k}} = 1 \tag{8}$$

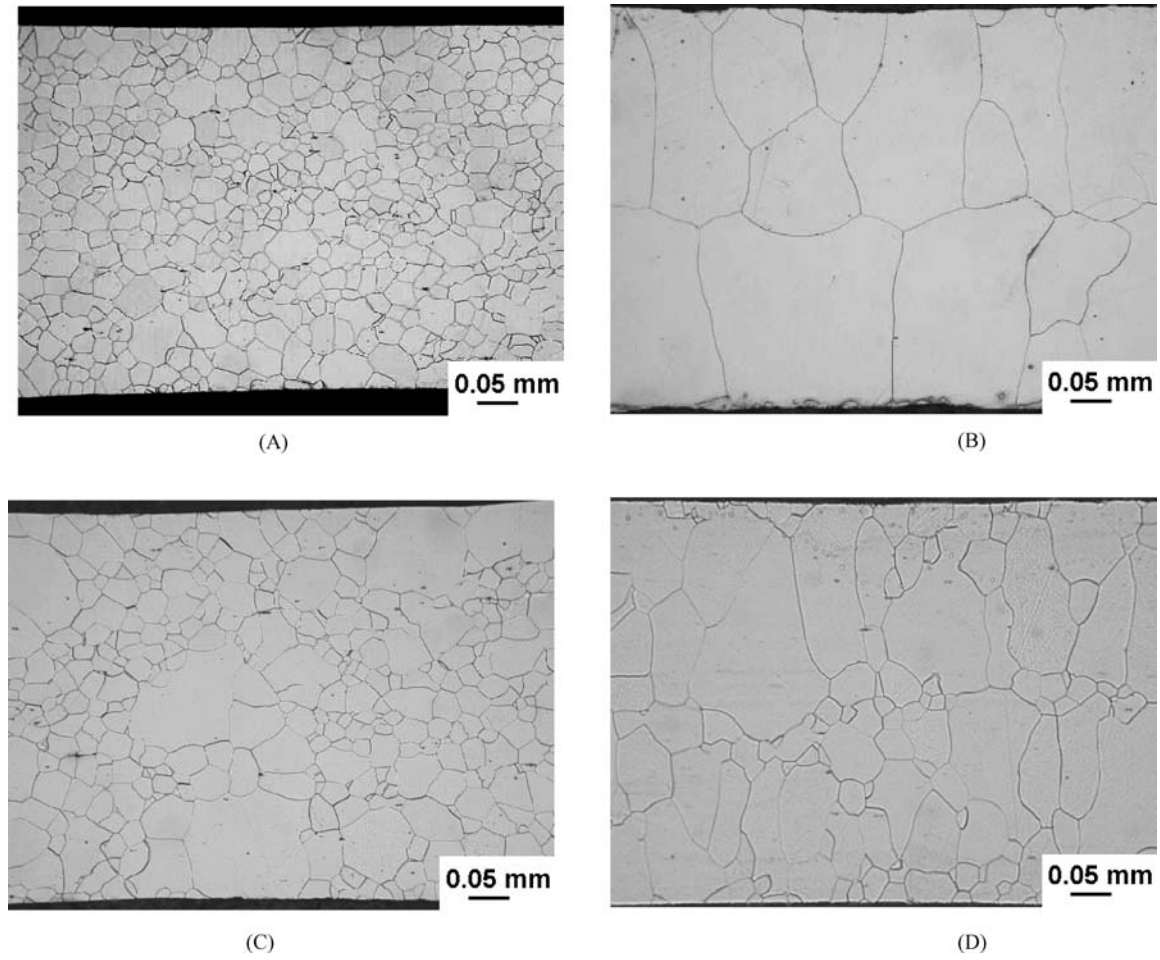

Figure 1 Microstructure of samples A–D.

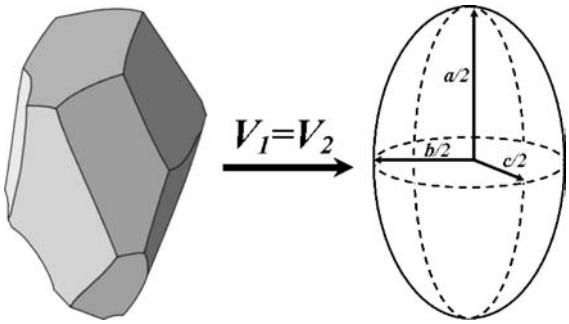

Figure 2 Scheme for changing of grain volume.

It is clear from Equations 6 and 8, that the proposed coefficient of homogeneity, G_v, is independent of the aspect ratio of the grain structure. The value G_v for "ideal" (equiaxed or non-equiaxed) homogeneous microstructures is 1.

The values of G_v, calculated by Equations 6 using experimental data for cases A–D, are presented in Table I. It can be seen that in the case of more homogeneous microstructures (samples A and B), the calculated coefficients of homogeneity are approaching the theoretical value of 1: $G_v \leq 1.25$. The difference between the theoretical G_v and calculated values can be explained by the non-uniform grain size. Hence, $G_v = 1.25$ can be chosen as the threshold conventional value for the homogeneous microstructure description. As noted in Table I, the higher value of coefficient G_v corresponds to wider grain size distributions. Thus, the coefficient G_v is a good indicator of the homogeneity of the microstructure.

3.2. Average grain size

The grain size distribution in samples A and B is reasonably uniform and the average grain size, $\bar{d}$, can be approximated by:

$$\bar{d} = \sqrt[3]{\bar{a}\bar{b}\bar{c}} \tag{9}$$

where the values of $\bar{a}$, $\bar{b}$, $\bar{c}$ are calculated by (10):

$$\bar{x} = \frac{\sum_{i=1}^{N} x_i}{N} \tag{10}$$

where x_i is one of the grain axes of ith grain and and $\bar{x}$ is average value of this axis.

In the other samples, C and D, the average grain size value cannot be used because of the wide spread in grain sizes. The grain size distribution in these microstructures can be approximated by a log-normal distribution:

$$y = y_0 + Ae^{-\frac{\ln^2 \frac{x}{x_c}}{2\sigma^2}} \tag{11}$$

where y is the number of grains within certain grain size range, x is grain size (grain axis), y_0 is the coefficient,

TABLE I Coefficients of homogeneity G_v, values of $\bar{a}$, a^*, $\bar{b}$, b^*, $\bar{c}$, c^*, $\bar{d}$ and $\bar{d}_{ef}$ in samples A–D

	G_v	$\bar{a}$ (μm)	a* μm	$\bar{b}$ (μm)	b^* (μm)	$\bar{c}$ (μm)	c^* (μm)	$\bar{d}$ (μm)	$\bar{d}_{ef}$ (μm)
Sample A	1.25	26.2	25.4	24.7	23.7	25.1	25.2	22.9	25.3
Sample B	1.22	195	187	96.3	94.2	97.7	93.4	118	122.4
Sample C	2.5	32	26	30	24.2	31.4	26.1	29.1	39.8
Sample D	3.1	52.8	23	30.3	16.8	29.9	17.8	64.2	102

σ is the standard deviation and x_c is the median value of the distribution.

The median values of the grain axis distributions a^*, b^* and c^*, calculated by (11), and the average values $\bar{a}$, $\bar{b}$, $\bar{c}$, calculated by Equation 10 are presented in Table I. It is obvious that for homogeneous microstructures, A and B, values of $\bar{a}$, $\bar{b}$, $\bar{c}$ and a^*, b^*, c^* are similar due to the narrow distribution of grain sizes.

However this is not true for inhomogeneous microstructures. In these cases the data must be divided into groups, such that for each group the coefficient of homogeneity, G_v, is close to 1. The more representative method of the microstructure evaluation is a combined histogram of the grain size distribution that consists of both grain's quantity ratio and volume fraction of one. All grain groups have to be homogeneous with, for example, $G_v \leq 1.1$, which is well inside of the conventionally chosen range $G_v \leq 1.25$, used for describing microstructure as homogeneous. Utilising this approach, it is possible to divide grains in respect to their size into three conventional groups: "large", "medium", "small". The representative grain size within the mentioned groups can be calculated by Equation 9. As an example, result of such calculation for the C and D microstructures is presented in Table II.

For inhomogeneous microstructure, the coefficients K_x, where $x = l$, m, s (large, medium, small, respectively) can be introduced as follows:

$$K_x = \frac{V}{V_x} \tag{12}$$

where V is overall volume and V_x is the volume of a homogeneous grain group. Then:

$$K_x = \frac{\sum_{i=1}^{N} V_i}{\sum_{x=1}^{X} V_l} = \frac{\sum_{i=1}^{N} a_i b_i c_i}{\sum_{x=1}^{X} a_x b_x c_x} = \frac{\sum_{x=1}^{n} \sum_{x=1}^{X} a_x b_x c_x}{\sum_{x=1}^{X} a_x b_x c_x} \tag{13}$$

where: X is the number of grains in the homogeneous group, N is the total number of grains and $\sum_x X = N$, n-number of homogeneous groups.

Rewriting the above equations gives for the large grains homogeneous group:

$$K_l = 1 + \frac{\sum_{m=1}^{M} a_m b_m c_m}{\sum_{l=1}^{L} a_l b_l c_l} + \frac{\sum_{s=1}^{S} a_s b_s c_s}{\sum_{L=1}^{L} a_l b_l c_l} \tag{14}$$

The coefficients K_m and K_s can be obtained in the same way as coefficient K_l. For homogeneous grain groups the next relationships can be introduced between average grain volumes of certain groups:

$$\frac{\bar{V}_m}{\bar{V}_l} = f_{ml} \qquad \frac{\bar{V}_s}{\bar{V}_l} = f_{sl} \qquad \frac{\bar{V}_s}{\bar{V}_m} = f_{sm} \tag{15}$$

where $\bar{V}_x$ is defined as follows: $\bar{V}_x = \frac{\sum_{x=1}^{X} V_x}{X}$. Then:

$$\frac{\sum_{m=1}^{M} a_m b_m c_m}{\sum_{l=1}^{L} a_l b_l c_l} = \frac{\sum_{m=1}^{M} V_m}{\sum_{l=1}^{L} V_l} = \frac{\bar{V}_m N_m}{\bar{V}_l N_l} = \frac{N_m}{N_l} f_{ml} \tag{16}$$

In the same manner the ratios $\frac{\sum_{m=1}^{M} a_m b_m c_m}{\sum_{s=1}^{S} a_s b_s c_s}$ and $\frac{\sum_{l=1}^{L} a_l b_l c_l}{\sum_{s=1}^{S} a_s b_s c_s}$ can be presented.

By substituting the above ratios into the corresponding form of the Equation 14, we have:

$$K_l = 1 + \frac{N_m}{N_l} f_{ml} + \frac{N_s}{N_l} f_{sl} \tag{17}$$

$$K_m = \frac{N_l}{N_m} \frac{1}{f_{ml}} + 1 + \frac{N_s}{N_m} f_{sm} \tag{18}$$

$$K_s = \frac{N_l}{N_s} \frac{1}{f_{sl}} + \frac{N_m}{N_s} \frac{1}{f_{sm}} + 1 \tag{19}$$

TABLE II Values of $\bar{a}$, a^*, $\bar{b}$, b^*, $\bar{c}$, c^* of homogeneous grain groups in samples C–F

	$\bar{a}_l$ (μm)	a^*_l (μm)	$\bar{b}_l$ (μm)	b^*_l μm	$\bar{c}_l$ (μm)	c^*_l (μm)
Sample C	75.4	72.1	74.6	74.2	75.1	73.2
Sample D	185.1	171.3	82.4	77.5	82.7	83.5
	$\bar{a}m$, μm	a^*m, μm	$\bar{b}m$, μm	b^*m, μm	$\bar{c}m$, μm	c^*m, μm
Sample C	31.2	29.3	32.2	31.8	29.2	30
Sample D	69.2	55.7	41.1	34.2	40.1	35.1
	$\bar{a}_s$, μm	a^*_s, μm	$\bar{b}_s$, μm	b^*_s, μm	$\bar{c}_s$, μm	c^*_s, μm
Sample C	17.3	16.1	17.1	16.9	16.8	16.3
Sample D	24.5	21.3	15.3	15.9	17.1	16.6

Equations 17–19 can be presented in matrix form as:

$$\begin{pmatrix} K_l \\ K_m \\ K_s \end{pmatrix} = \begin{pmatrix} \frac{N_l}{N} & f_{ml} & f_{sl} \\ \frac{1}{f_{ml}} & \frac{N_m}{N} & f_{sm} \\ \frac{1}{f_{sl}} & \frac{1}{f_{sm}} & \frac{N_s}{N} \end{pmatrix} \begin{pmatrix} \frac{N}{N_l} \\ \frac{N}{N_m} \\ \frac{N}{N_s} \end{pmatrix} \tag{20}$$

For the general case, as a microstructure subdivided into n homogeneous groups:

$$K_j = \sum_{j=1}^{n} t_{ji} r_i \tag{21}$$

$$t_{ji} = \begin{pmatrix} \frac{N_1}{N} & f_{21} & f_{31} & \dots & f_{n1} \\ \frac{1}{f_{21}} & \frac{N_2}{N} & f_{32} & \dots & f_{n2} \\ \frac{1}{f_{31}} & \frac{1}{f_{32}} & \frac{N_3}{N} & \dots & f_{n3} \\ . & . & . & . & . \\ . & . & . & . & . \\ \frac{1}{f_{n1}} & \frac{1}{f_{n2}} & \frac{1}{f_{n3}} & \dots & \frac{N_n}{N} \end{pmatrix}, \quad r_i = \begin{pmatrix} \frac{N}{N_1} \\ \frac{N}{N_2} \\ \frac{N}{N_3} \\ . \\ . \\ \frac{N}{N_n} \end{pmatrix} \tag{21}$$

Taking into account (22), the effective average grain size is simplified to form:

$$\bar{d}_{ef} = \sqrt[3]{\sum_{j=1}^{n} \frac{a_j}{K_j} \sum_{j=1}^{n} \frac{b_j}{K_j} \sum_{j=1}^{n} \frac{c_j}{K_j}} \tag{23}$$

The proposed way of calculating the effective average grain size has the essential difference compared to the traditional method, when the sum of sizes of all assessed grains is divided by the number of grains. Taking into account volume fractions gives us a more reasonable value of the (effective) average grain size and is logical from the point of view how big is the "weight" of the certain size grain group in the overall microstructure.

It is worth illustrating the advantage of the proposed method. Let us consider the following ideal array of grains (first digit is number of grains, second one is diameter of grains): {70 × 5, 60 × 8, 10 × 18, 5 × 25, 2 × 100, 1 × 120}. Average grain size calculated according to the traditional method is 9.83 units. However, it is possible to divide the microstructure into three homogenous grain groups of diameters 5–8, 18–25 and 100–120 units. The volume fractions of "small", "medium" and "large" grains are 0.01, 0.035 and 0.955 respectively. It is obvious that traditional calculation of average grain size gives a result, which is too far from the size of the dominating element of the microstructure. The value of the average grain size derived from the average value of the volume of grains is 47.79 units; that again does not describe properly the considered microstructure. In the case of the proposed approach, the value of effective average grain size is 102.63 units, which is fairly close to the diameter of the "big" grain covering more than 95% of the considered microstructure.

The microstructures of samples C and D have been divided into three groups containing "small", "medium" and "large" grains, and within each group $G_v \leq 1.2$. Furthermore, the values of $\bar{a}$ and a^*, $\bar{b}$ and b^*, $\bar{c}$ and c^* for each homogeneous group are similar, (see Table II). The average grain sizes of homogeneous groups are calculated utilising Equation 10.

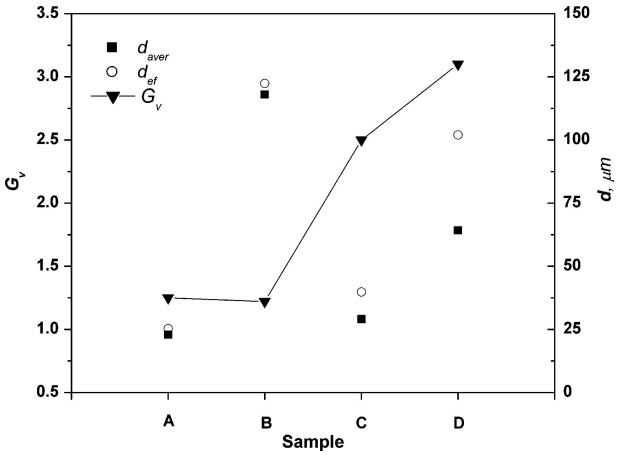

Figure 3 Relations between the $\bar{d}$, $\bar{d}_{ef}$ and homogeneity of microstructure.

The values of $\bar{a}$, $\bar{b}$, $\bar{c}$ calculated according to the Equation 22 for the actual microstructures are presented in Table I. The effective average grain size, $\bar{d}$, can be calculated utilising Equation 23.

Fig. 3 presents the relations between the estimated grain size by both standard techniques and above presented method and calculated value of homogeneity coefficient. As one can see, the high difference between $\bar{d}_{ef}$ and $\bar{d}$ corresponds to high value of coefficient G_v. For microstructures A and B values $\bar{d}_{ef}$ and $\bar{d}$ are similar, because of homogeneity. Consequently, for inhomogeneous microstructures C and D, the differences between $\bar{d}_{ef}$ and $\bar{d}$ increase with increasing level of the inhomogeneity. Thus, $\frac{\bar{d}_{ef}}{\bar{d}}$ ratio is also good indicator of microstructure homogeneity.

In the limit case $n = N$, i.e., each group comprises only one grain. Using such an approach, it is ease to calculate $\bar{d}_{ef}$ utilising each grain data directly from the image analysis results/EBSD grain boundary maps, without dividing grains into grain size groups.

4. Summary

1. The classical average grain size approach is not sufficient for the quantitative characterisation of the highly inhomogeneous microstructures.

2. The level of the microstructure homogeneity can be described by the proposed coefficient of homogeneity G_v:

$$G_v = N^2 \frac{\sum_{i=1}^{N} a_i b_i c_i}{\sum_{i=1}^{N} a_i \sum_{i=1}^{N} b_i \sum_{i=1}^{N} c_i}$$

A higher value of G_v corresponds to a wider grain size distribution.

3. In case of the microstructure with a wide range of grain sizes, the whole microstructure has to be divided into homogeneous grain groups with G_v approaching 1. The effective average grain size is evaluated as a

weighted sum of the average grain sizes of homogeneous grain groups.

4. In limit case and, simultaneously, for simplicity of the computer-assisted analysis of microstructure, each grain size group should comprise only one grain.

5. The ratio $\frac{\bar{d}_{ef}}{\bar{d}}$ can be also used for the quantitative description of the microstructure inhomogeneity. The threshold value should be chosen empirically.

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