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STEREOLOGY IN QUANTITATIVE MICROSTRUCTURAL ANALYSIS: THE ORIENTATION FACTOR

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ABSTRACT

In order to describe orientation effects on the properties of multiphase engineering materials one needs more than a mathematically defined orientation factor. For practical purposes it is necessary that this orientation factor reflects three-dimensional orientation and can be derived from measurements made in two-dimensional cross sections by quantitative microstructural analysis. Furthermore it has to have a real meaning. This is what the present paper provides: an orientation factor which describes the orientation of particles or fibres in composites or other multiphase materials as a function of the angle between those features and a defined preferential direction (reference direction) as a spatial average and which results from values measured in two-dimensional sections via stereological relationships. Its derivation also implies, that the orientation factor reflects the orientation effect in microstructure-property-equations in agreement with experimental results.
Key Words: composites, microstructure-property-correlations, multiphase materials, orientation factor, quantitative microstructure analysis, stereology.

INTRODUCTION: ORIENTATION, MICROSTRUCTURAL FACTORS AND PROPERTIES

It is well known from reality, that the orientation of the phases has special influence on the properties of a multiphase material: fibre-reinforced composites as well as directionally solidified alloys are representative examples. But apart from engineering materials science also in biology, medicine or geology for example the determination of orientation effects by respective structural parameters is a stereological task, since the "orientation factor" desired

- has to provide three-dimensional information about the orientation of structural components and not only to refer to the orientation in two-dimensional sections
- has to follow from measurements made in two-dimensional sections made by stereologically supported quantitative microstructural analysis
- has to reflect the orientation effect quantitatively in microstructure-property-correlations

To derive this type of "spatial" and "engineering" orientation factor for multiphase materials is the subject of the present paper.

Considered more generally the phase orientation is only one microstructural factor affecting the properties of a multiphase material amongst others.

According to the literature a total of 5 parameters is required in order to take complete quantitative account of the microstructure of a multiphase material in microstructure-field property equations (Ondracek, 1987). They are

- the number of phases
- the type of microstructure
- the phase concentration factor
- the shape factor
- the orientation factor.

This, of course, is not rigorous but an engineering approach, which holds also for other than fields properties as for Youngs modulus of elasticity (Mazilu and Ondracek, 1990; Ondracek, 1989; Boccaccini et al., 1993), for Poisson ratio (Boccaccini and Ondracek, 1991) for rupture strength or thermal shock resistance (Boccaccini et al., 1991).
All these parameters are determined by means of quantitative microstructural analysis (Ondracek, 1987). Working from prepared sections through a two-phase material, the images are magnified microscopically, transferred to a monitor and again - electronically - magnified and measured. This operation may also be done with fotografic reprints from the image using an epidiascope instead of the microscope, which sometimes helps in contrasting and suppressing artefacts. The data measured in a flat plane are converted to three dimensional microstructural parameters by means of stereological equations in a primary computer

program and these parameters are then used in other computer programs for determining the required microstructural factors and properties of the two-phase materials.

Determination of the data on the number of phases, is particularly simple, since they can be seen directly in magnified microstructural images with adequate contrast.
Not simple, but possible on the basis of continuity measurements and probability calculations, is the distinction between the two types of microstructure - matrix and interconnecting microstructure - which has been treated in literature (Ondracek, 1987; Schulz, 1974) and is not subject of the present paper.

The phase concentration factor follows as phase volume content directly from the mean value of the area content of the phases on micrographs originating from sections cut statistically through the material (Delesse principle: Underwood, 1970) and does not need modeling of the real microstructure. This modeling, however, becomes compulsory for the determination of the shape and orientation factors, which in matrix type microstructures refer to the inclusion phase. The derivation of both is based on the spheroidal model mean value premise (Ondracek, 1987), in which real irregular shaped inclusions are substituted by spheroids, which have approximately the same surface-to-volume-ratio. To describe these spheroids quantitatively one needs to determine the ratio of the rotation axis (axis of revolution) to the minor axis of the spheroid by which the real particles of the inclusion phase in the model are replaced.

THE DETERMINATION OF AXIAL RATIOS

The fact that this "spatial" axial ratio (z/x) can be determined from the (mean) "planar" axial ratio of the sectional ellipses of the spheroids substituting for the real particles of the inclusion phase by means of stereological equations, was a crucial factor in the choice of the spheroid model for characterising real material.

In practice one proceeds as follows (compare fig.1): the area and perimeter are measured for each section of an (irregularly shaped) particle of the inclusion phase in a two-dimensional micrograph. From this the axial ratio is calculated for the sectional ellipse with the same area-to-perimeter-ratio as the measured section area of the particle (Ondracek, 1982; Pejsa, 1981). Since one can form the planar axial ratios not only from "minor axis b´ to major axis a´" but also from "major axis to minor axis", one obtains in each case two values of measured axial ratios of the sectional ellipse. Continuing this procedure for all sections of the particles, one obtains two mean values for the axial ratios of the sectional ellipses:

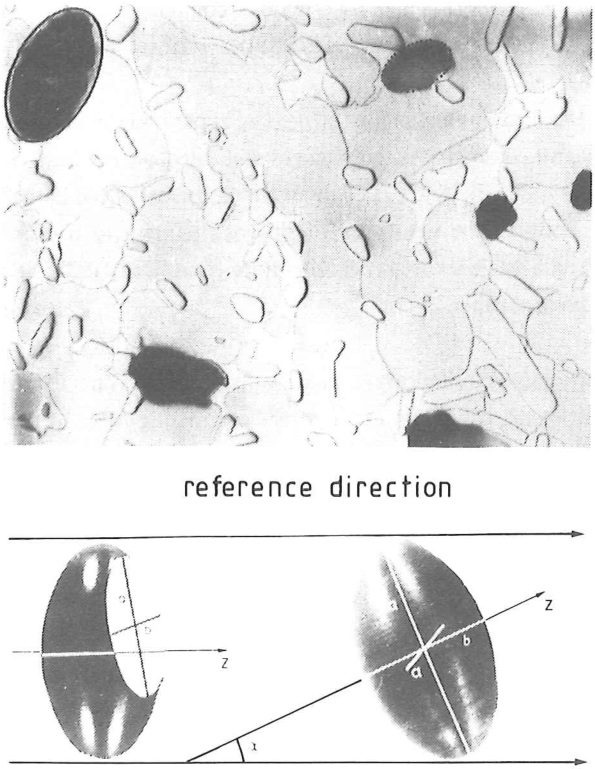

Fig. 1: Determination of axial ratios of sectional ellipses

$$\frac{\sum_{1}^{n} \frac{a'}{b'}}{n} = \left|\overline{\frac{a'}{b'}}\right| \qquad (1)$$

$$\frac{\sum_{1}^{n} \frac{b'}{a'}}{n} = \left|\overline{\frac{b'}{a'}}\right| \qquad (2)$$

For each of these axial ratios, the stereological equations provide an axial ratio for each oblate spheroid (denoted by subscript $=$) and one for each prolate sheroid (denoted by subscript //) (DeHoff, Rhines 1961; Ondracek 1982; Pejsa 1981; Schulz 1985), hence a total of 4 "spatial" axial ratios (z/x):

$$\overline{\left(\frac{b'}{a'}\right)} = \left(\frac{z}{x}\right) = \frac{\arcsin\sqrt{1-\left(\frac{z}{x}\right)_{=}^{2}}}{\sqrt{1-\left(\frac{z}{x}\right)_{=}^{2}}} \tag{3}$$

$$\overline{\left(\frac{b'}{a'}\right)} = \frac{1}{2} + \frac{1}{2\left(\frac{z}{x}\right)_{//}^{2}\sqrt{1-\frac{1}{\left(\frac{z}{x}\right)_{//}^{2}}}}\ln\left(1+\sqrt{1-\frac{1}{\left(\frac{z}{x}\right)_{//}^{2}}}\left(\frac{z}{x}\right)_{//}\right) \tag{4}$$

$$\overline{\left(\frac{a'}{b'}\right)} = \frac{1}{2} + \frac{\arcsin\sqrt{1-\left(\frac{z}{x}\right)_{=}^{2}}}{2\left(\frac{z}{x}\right)\sqrt{1-\left(\frac{z}{x}\right)_{=}^{2}}} \tag{5}$$

$$\overline{\left(\frac{a'}{b'}\right)} = \frac{1}{\sqrt{1-\frac{1}{\left(\frac{z}{x}\right)_{//}^{2}}}}\ln\left(1+\sqrt{1-\frac{1}{\left(\frac{z}{x}\right)_{//}^{2}}}\left(\frac{z}{x}\right)_{//}\right) \tag{6}$$

These equations are shown graphically in fig. 2 (Ondracek, 1982; Pejsa, 1981). In a notional experiment, let it now be assumed that there is a two-phase material with identical prolate spheroidal particles of inclusion phase. In the sections cut statistically through it, sectional ellipses then appear whose measurement provides the two axial ratios in accordance with equation (1) and (2). From equations (3,4) and (5,6) then follow 4 axial ratios of spheroids, of which only two, namely those for prolate spheroids corresponding to the assumed model material must be identical, whilst the associated two axial ratios for oblate spheroids are not. For quantitative microstructural analysis of a <u>real</u> material, this means that of the 4 axial ratios obtained from measurements for substitute spheroids, only two are approximately equal in each case for the same type of spheroid (prolate or oblate), or lie closer together than the other two. The spheroid corresponding to the approximately equal axial ratios would then be best suited for a model characterising the real particles of the inclusion phase. There might be real cases, however, in which the differences between the alternative spatial axial ratios will be so small, that no selection is possible for practical purposes. As a first approach one may either determine then a mean value from each pair of axial ratios for each type of spheroid, or one averages all 4 axial ratios.

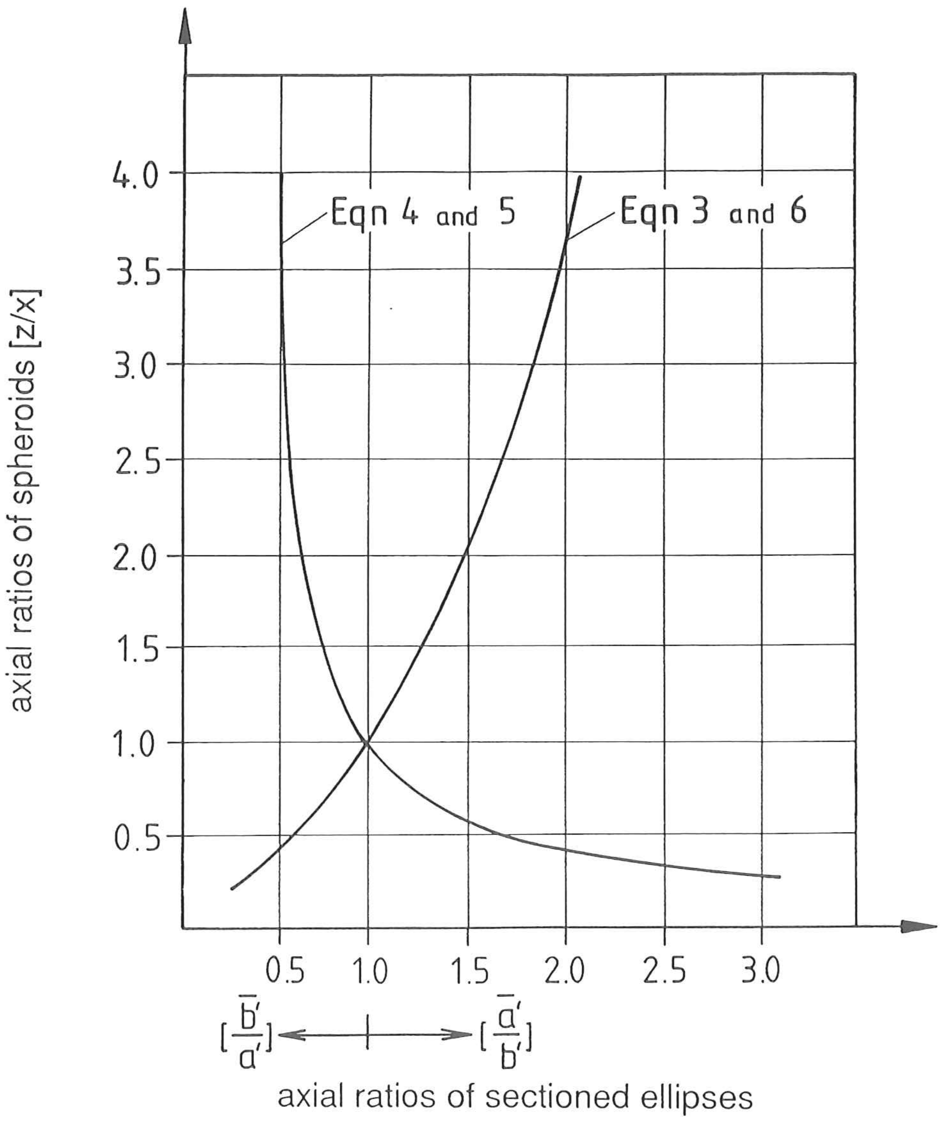

Fig. 2: "Spatial " axial ratio of spheroids as a function of "plane" axial ratio of sectioned ellipses

In order to test the stereological equations for determining axial ratios a model experiment has been made. Pills of equal prolate and oblate shapes have been included in a gypsum matrix. Since their spatial axial ratio can be measured, the mean axial ratio of their elipses in sections of plane can be determined by equations (4) and (6) or fig. 2. These calculated values are shown in the second and third columns of Table 1.
The gypsum samples containing pills were statistically cut with a diamond saw and the sections were observed under microscope. Measurements as described above were made to determine the experimental mean axial ratios of the ellipses in sections of plane. At least 30 ellipses were measured and the mean value was determined in each case. The measured values are shown in Table 1 for comparison with the calculated values.

Table 1: Comparison of calculated and experimental axial ratios of spheroidal inclusions (pills in a gypsum matrix)

Actual axial ratio (z/x)	Mean axial ratio in the plane				Rel.Error (%)	
	$\overline{(a'/b')}$ Eqn. 5, 6	$\overline{(b'/a')}$ Eqn. 3,4	$\overline{(a'/b')}$ Experiment	$\overline{(b'/a')}$ Experiment	$\overline{(a'/b')}$	$\overline{(b'/a')}$
1.49	0.79	1.285	0.78	1.27	1	1
0.65	0.74	1.37	0.75	1.33	1	3
0.56	0.66	1.55	0.66	1.53	0	1

THE ORIENTATION FACTOR

In accordance with the derivation of the microstructure-field property-equations (Grolier et al. 1991), the orientation factor is the cosine squared of the angle formed by the field strength gradient direction - in this case the reference direction - and the rotation axis of spheroids of identical form and size substituting the real particles in the model. For elastic properties the reference direction is given by the stress-strain field (Mazilu and Ondracek, 1990). Like the shape factor the orientation factor can also be calculated from axial ratios determined by quantitative microstructural analysis. To derive the relationship, one proceeds from the following equations (De Hoff and Rhines, 1961), where section A - as well as sections B and C - refer to fig. 3 (Ondracek, 1987):

- for prolate spheroids

$$\left|\frac{b'}{a'}\right|_A = \sqrt{\cos^2\alpha_A + \frac{\sin^2\alpha_A}{\left|\frac{z}{x}\right|_{//}^2}} \tag{7a}$$

or after transformation

$$\cos^2\alpha_A = \frac{\left|\frac{z}{x}\right|_{//}^2 \left|\frac{b'}{a'}\right|_A^2 - 1}{\left|\frac{z}{x}\right|_{//}^2 - 1} \tag{7b}$$

and respectively to sections B, C

$$\cos^2\alpha_B = \frac{\left|\frac{z}{x}\right|_{//}^2 \left|\frac{b'}{a'}\right|_B^2 - 1}{\left|\frac{z}{x}\right|_{//}^2 - 1} \tag{7c}$$

$$\cos^2\alpha_C = \frac{\left|\frac{z}{x}\right|_{//}^2 \left|\frac{b'}{a'}\right|_C^2 - 1}{\left|\frac{z}{x}\right|_{//}^2 - 1} \tag{7d}$$

- for oblate spheroids

$$\left|\frac{a'}{b'}\right|_A = \sqrt{\cos^2\alpha_A + \frac{\sin^2\alpha_A}{\left|\frac{z}{x}\right|_{=}^2}} \tag{8a}$$

or after transformation

$$\cos^2\alpha_A = \frac{\left|\frac{z}{x}\right|_{=}^{2}\left|\overline{\frac{a'}{b'}}\right|_{A}^{2} - 1}{\left|\frac{z}{x}\right|_{=}^{2} - 1} \tag{8b}$$

and respectively to sections B, C

$$\cos^2\alpha_B = \frac{\left|\frac{z}{x}\right|_{=}^{2}\left|\overline{\frac{a'}{b'}}\right|_{B}^{2} - 1}{\left|\frac{z}{x}\right|_{=}^{2} - 1} \tag{8c}$$

$$\cos^2\alpha_C = \frac{\left|\frac{z}{x}\right|_{=}^{2}\left|\overline{\frac{a'}{b'}}\right|_{C}^{2} - 1}{\left|\frac{z}{x}\right|_{=}^{2} - 1} \tag{8d}$$

For these equations the ("spatial") axial ratio of the spheroid is determined with equations (3,4) and (5,6) from the mean axial ratios of the sectional ellipses $\left(\overline{\left|\frac{b'}{a'}\right|}, \overline{\left|\frac{a'}{b'}\right|}\right)$, which were measured in sections cut statistically through the material. On the other hand, the mean axial ratios of the sectional ellipses $\left(\overline{\left|\frac{b'}{a'}\right|}_{A,B,C}, \overline{\left|\frac{a'}{b'}\right|}_{A,B,C}\right)$, contained in the equations themselves, are those measured in sectional planes (A,B,C) perpendicular to the field direction (see fig. 3). Considering $\cos^2\alpha_D$ as a proper measure for the - average - orientation of the included phase particles (compare fig. 4) as it follows from the derivation of microstruture - property - correlations (Grolier et al., 1991; Ondracek, 1987) one has to define a preference direction to which the orientation should refer. This direction in context with microstructure-property-correlation is given - for example - by the electric field or the stress-strain field direction. And the orientation angle as given in equations 7,8 refers to that section, which is perpendicular to the reference direction.

According to fig. 3 the orientation factor considered is

$$\cos^2\alpha_D = \cos^2\alpha_A$$

and its angle refers to that one given in fig. 4.

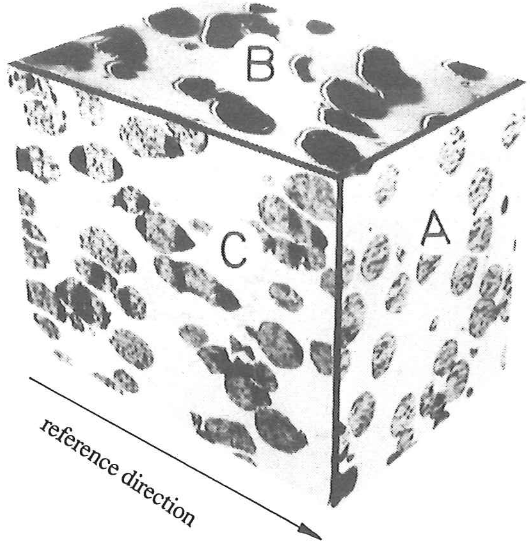

Fig. 3: Sections through a two phase material in order to determine the orientation factor

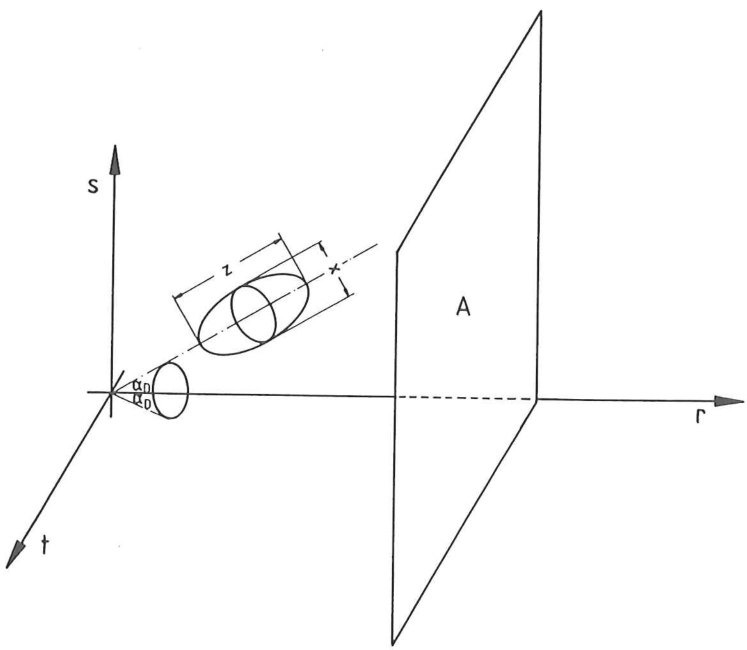

Fig. 4: Orientation angle and reference direction

In order to calculate that angle - or its cosine squared - the respective equations 7,8 should hold for all real microstructural configurations, which, in fact, is not the case. Assuming spheres - for example - the mean orientation angle has to follow from a statistically random angle distribution and the orientation factor would be (Underwood, 1970)

$$\cos^2\alpha_D=1/3$$

Equations 7 and/or 8 however provide undetermined solutions when inserting the respective terms $\left(\left|\frac{z}{x}\right|=1;\left|\frac{b'}{a'}\right|=\left|\frac{a'}{b'}\right|=1\right)$. This is why the following derivation has been made using axial ratios $\left(\left|\frac{b'}{a'}\right|_{A,B,C};\left|\frac{a'}{b'}\right|_{A,B,C}\right)$ together with the whole set of equations (7,8).

Furthermore the following condition is valid:

$$\cos^2\alpha_A+\cos^2\alpha_B+\cos^2\alpha_C=1 \tag{9}$$

This boundary condition, however, is only fulfilled on the ideal model, whilst for the real case the following will apply:

$$\sum_{i=A}^{C}\cos^2\alpha_i=X\neq 1 \tag{10}$$

In order to adjust the model material and the real case, one sets with a correction factor (Y)

$$Y\cos^2\alpha_A+Y\cos^2\alpha_B Y\cos^2\alpha_C=Y\bullet X=1 \tag{11}$$

and

$$Y=\frac{1}{\cos^2\alpha_A+\cos^2\alpha_B+\cos^2\alpha_C} \tag{12}$$

From equation (12) together with equation 7 follows

$$Y=\frac{\left|\frac{z}{x}\right|_{//}^{2}-1}{\left|\frac{z}{x}\right|_{//}^{2}\left\{\left|\frac{b'}{a'}\right|_{A}^{2}+\left|\frac{b'}{a'}\right|_{B}^{2}+\left|\frac{b'}{a'}\right|_{C}^{2}\right\}-3} \tag{13}$$

and - also from equation (12) - but with equation 8 follows

$$Y = \frac{\left|\frac{z}{x}\right|_{=}^{2} - 1}{\left|\frac{z}{x}\right|_{=}^{2}\left\{\overline{\left|\frac{a'}{b'}\right|}_{A}^{2} + \overline{\left|\frac{a'}{b'}\right|}_{B}^{2} + \overline{\left|\frac{a'}{b'}\right|}_{C}^{2}\right\} - 3} \tag{14}$$

The orientation factor $(\cos^2 \alpha_A = \cos^2 \alpha_D)$ which can be determined from analytical measurements of microstructure results then from equations (7,8,13,14).

$$\cos^2 \alpha_D = \frac{\left|\frac{z}{x}\right|_{//}^{2}\overline{\left|\frac{b'}{a'}\right|}_{A}^{2} - 1}{\left|\frac{z}{x}\right|_{//}^{2}\left\{\overline{\left|\frac{b'}{a'}\right|}_{A}^{2} + \overline{\left|\frac{b'}{a'}\right|}_{B}^{2} + \overline{\left|\frac{b'}{a'}\right|}_{C}^{2}\right\} - 3} \tag{15}$$

or

$$\cos^2 \alpha_D = \frac{\left|\frac{z}{x}\right|_{=}^{2}\overline{\left|\frac{a'}{b'}\right|}_{A}^{2} - 1}{\left|\frac{z}{x}\right|_{=}^{2}\left\{\overline{\left|\frac{a'}{b'}\right|}_{A}^{2} + \overline{\left|\frac{a'}{b'}\right|}_{B}^{2} + \overline{\left|\frac{a'}{b'}\right|}_{C}^{2}\right\} - 3} \tag{16}$$

Since for example for a model material with statistically random orientation of the inclusion phase particles

$$\overline{\left|\frac{b'}{a'}\right|}_{A} = \overline{\left|\frac{b'}{a'}\right|}_{B} = \overline{\left|\frac{b'}{a'}\right|}_{C} \tag{17}$$

or

$$\overline{\left|\frac{a'}{b'}\right|}_{A} = \overline{\left|\frac{a'}{b'}\right|}_{B} = \overline{\left|\frac{a'}{b'}\right|}_{C} \tag{18}$$

must apply, an orientation factor of

$$\cos^2 \alpha_D = \frac{1}{3} \tag{19}$$

follows from equation (15) and (16), as expected for an isotropic material (Ondracek, 1987).

It might be mentioned in this context that an isotropic material will always have an orientation factor of 1/3 but this orientation factor is not exclusively representative for an isotropic microstructure. It is possible to obtain that special orientation factor also by definitely oriented microstructures ($\alpha_D \approx 55°$), which means, according to theory, that the property of unique anisotropic material in one preferential direction should be identical with that of the same but isotropic material. Further theoretical treatment and experimental verification of this result is desired.

As a general engineering approach, however, the derivation demonstrates, that the orientation factor can be completely determined by quantitative microstructure analysis. It is shown extensively in the literature already, that it correlates the microstructure of two-phase materials, including porous materials, with their properties (Boccaccini and Ondracek, 1991; Boccaccini et al., 1993; Ondracek , 1982, 1987, 1988, 1989; Pejsa, 1981).

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