Materials Transactions, JIM, Vol. 32, No. 11 (1991), pp. 1042 to 1047

Estimation of Three-dimensional Grain Size Distribution in Polycrystalline Material

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A new method has been proposed for the estimation of the three-dimensional grain size distribution from the two-dimensional distribution measured on the cross section of polycrystalline material. In this method, twelve types of polyhedra were employed as the grain models. The distributions of the cross-sectional diameters of the individual polyhedra were expressed as probability density functions. On the basis of the functions for each polyhedron, the supposed grain size distribution on the cross section of the material was calculated, and it was compared with the measured one. The comparison was repeated until the agreement between the both distributions. By operating this two-dimensional distribution reversely, the three-dimensional grain size distribution was estimated.

The distribution of the vertex number of polygon-shaped grains on the cross section and that of the face number of polyhedron-shaped grains in three dimensions were calculated from the obtained results and were compared with the measured distributions. There was a good agreement between these distributions.

(Received March 28, 1991)

Keywords: *grain size distribution, three dimensions, grain shape, grain size, polycrystalline*

I. Introduction

It is well known that the grain size in polycrystalline material plays an important role in controlling its mechanical properties such as strength or toughness. When the relation between the grain size and the properties is disscused, the average grain size measured on a cross section is generally employed. However, the information of the grain size distribution may be much more important than that of the average grain size to predict the fracture initiation and the crack propagation in a material, as pointed out by Wasẽn and Warren[1].

Takayama *et al.*[2] proposed a method to estimate the three-dimensional grain size distribution from the measured linear intercept length distribution. Their method is based on the assumption that the grain size distribution in three dimensions is a log-normal one. This assumption will be valid for the general structure in polycrystalline material. However, the grain size distribution after an abnormal grain growth or the artificial grain size distribution in a green compact in a powder metallurgy process, for example, may not be a log-normal one.

The purpose of the present paper is to propose a new method for the estimation of the three-dimensional grain size distribution in polycrystalline material from the measured two-dimensional grain size distribution on the cross section without any assumption of the distribution type.

Many reseachers have often employed tetrakaidecahedron (type 14-C in Fig. 1) as the grain model[2]-[4], in spite of the fact that the actual shape is various and complex[5]. On the contrary, we employed many types of polyhedra and introduced mathematical formulae for the cross-sectional diameter distributions of those polyhedra.

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II. Procedure

1. Model of grain shape

In this work, twelve types of polyhedra given in Fig. 1 were employed to comply the variety of the grain shape in actual material. These are regular polyhedra or semi-

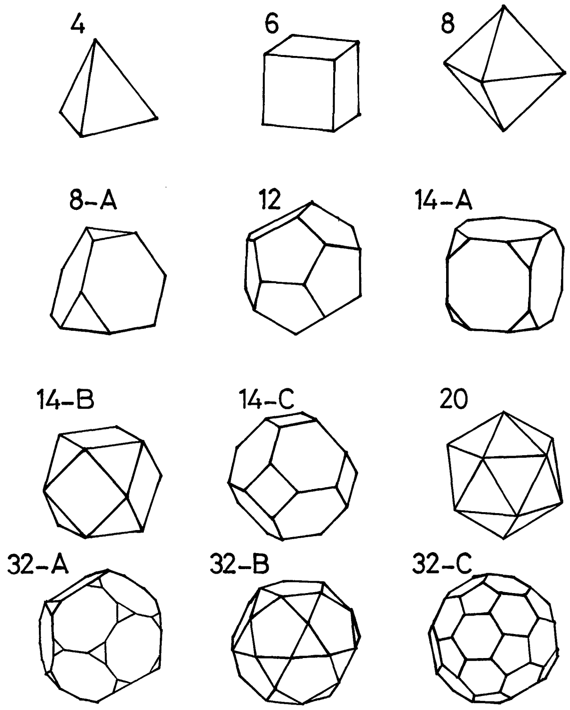

Fig. 1 Twelve types of polyhedra employed as the grain model.

regular ones (truncated regular ones). Because each polyhedron has each circumscribing sphere and the all edge lengths of each polyhedron are the same, the application of the present method is effective for materials with equiaxed grains.

On the basis of those grain models in Fig. 1, the present authors[6] have proposed the relation between the number of the grain faces, m, and the relative grain size, $D/\bar{D}$.

$$m=17D/\bar{D}-3 \tag{1}$$

where D is the diameter of the individual grains and $\bar{D}$ is the average one. The diameter of a grain was defined as the equivalent volume diameter. Equation (1) can be obtained from the calculation of the ratio of the radii of the two spheres in contact with each other, one of which is placed at the center of a polyhedron and another of which is placed on the corner of the polyhedron, and the diameter of the latter sphere is equal to the edge length of the polyhedron.

Rhines and Patterson[5] measured the weights of individual grains after disintegrating the sample into separate grains, and they investigated the relation between the weight W and the number of the grain faces m. Their result showed the following relation:

$$\log(W)=a\cdot\log(m)+b \tag{2}$$

Equation (2) can be rewritten as

$$\log(V)=a\cdot\log(m)+b-\log(\rho) \tag{3}$$

where a and b are constants, V is the volume of a grain and ρ is the density.

Equation (1) can be changed into the similar type to eq. (3), which shows that the relation between the logarithms of the volume of a grain and the number of the faces is linear. Equation (4) is derived from eq. (1):

$$\log(V)=3\log(m+3)+3\log\bar{D}-\log\{3(17\times2)^3/(4\pi)\} \tag{4}$$

Equations (3) and (4) are essentially equivalent in a sense that the relation between the logarithms of the volume of a grain and the number of the faces is linear. Consequently, eq. (1) which was derived from the geometrical features of several polyhedra agrees with the experimental result.

2. Basic distribution functions of the size and the shape of the cross section of the polyhedron-shaped grains

Each polyhedron in Fig. 1 was cut mathematically by arbitrary planes (5000 planes produced by the generation of random numbers in a computer), and the distributions of the cross sectional diameters were obtained for each polyhedron. The diameter was defined as the equivalent area diameter, and it was normalized by dividing by the polyhedron's diameter in three dimensions. Some examples of the distributions are given in Fig. 2.

In Fig. 2, the relative diameter 1.0 shows the polyhedron's diameter in three dimensions. The reason why some ralative diameters are larger than the value of 1.0 is related to the definition that the polyhedron's diameter in three dimensions is the equivalent volume diameter.

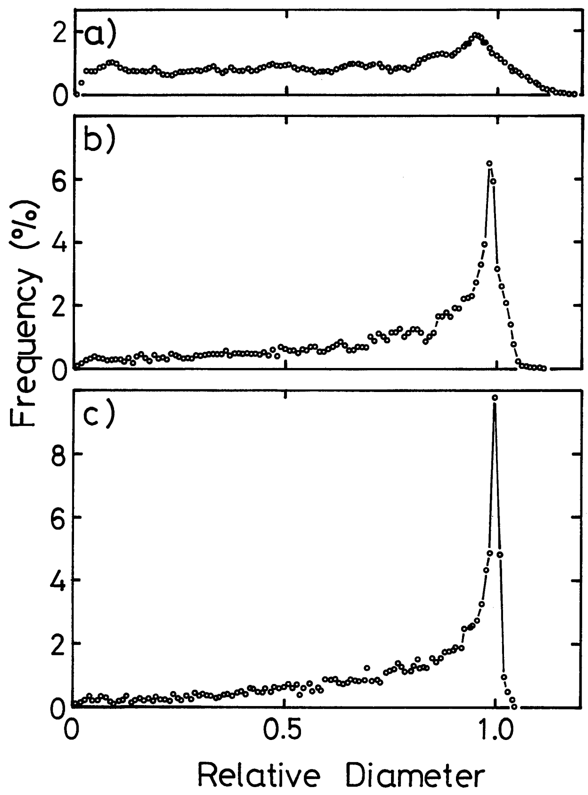

Fig. 2 Diameter distributions of the cross section of some polyhedra. (a), (b) and (c) are those of types 4, 14-C and 32-C in Fig. 1, respectively.

The distribution was broad when the number of the faces of a polyhedron was small. The distribution, however, became sharper with the increasing number of the faces, and the position of the peak moved toward the value of 1.0. At the same time, the value of the maximum diameter decreased toward 1.0.

The characteristic features of the distribution curves in Fig. 2 are as follows:

(1) $P_l>0$ for $0\leqq l\leqq l_{\mathrm{Max}}$, where l is the relative cross sectional diameter, l_{Max} is the maximum relative diameter and P_l is the probability density for the diameter l.

(2) $P_l\to0$, when $l\to0$ and $l\to l_{\mathrm{Max}}$.

(3) The curve has only one peak at (l_P, K_l), and l_P is comparatively near the maximum relative diameter.

After trial and error, we found that eq. (5) fitted the characteristic features (1) to (3) of the distribution curves in Fig. 2. Although there may be some other functions which can describe the curves in Fig. 2, eq. (5) was employed as the probability density function for the distribution of the cross-sectional diameters of a polyhedron because eq. (5) is simple and it fits very well to the curves.

$$P_l=K_l\cdot\exp\left[-\left\{\ln\frac{l_{\mathrm{Max}}-l}{l_{\mathrm{Max}}(1-l_P)}\right\}^2\right] \tag{5}$$

The values of K_l, l_{Max} and l_P are respectively dependent

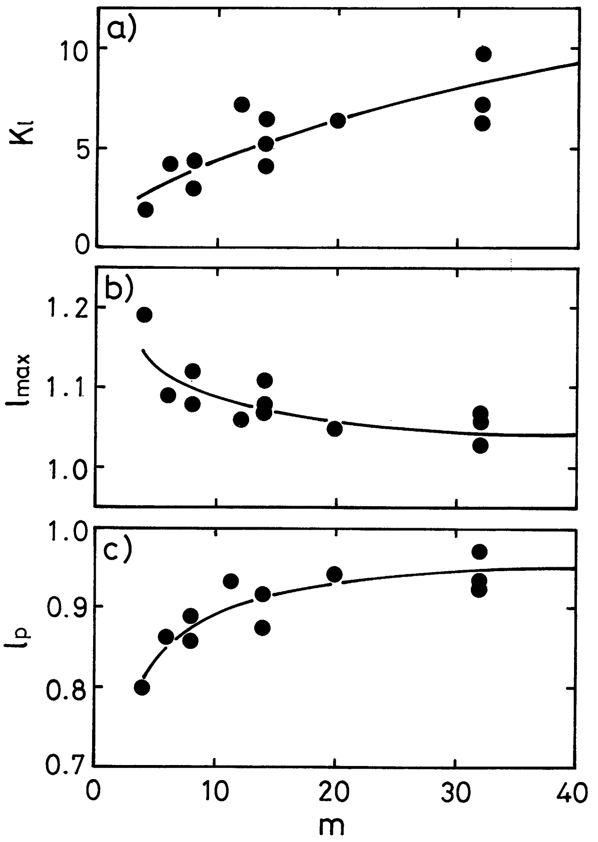

Fig. 3. Relation between m and K_l (a), l_{Max} (b) and l_P (c). m: number of faces of a polyhedron, K_l: maximum probability density, l_{Max}: maximum relative diameter, l_P: peak position in the distribution.

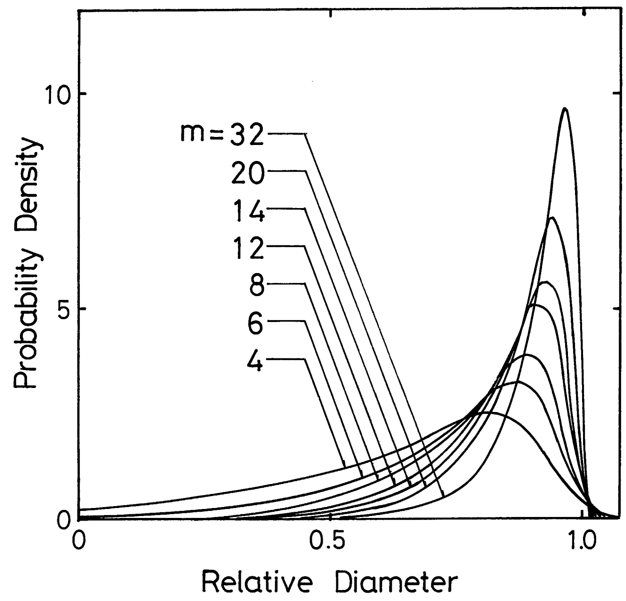

Fig. 4 Relation between the relative cross sectional diameter of polyhedra and the probability density.

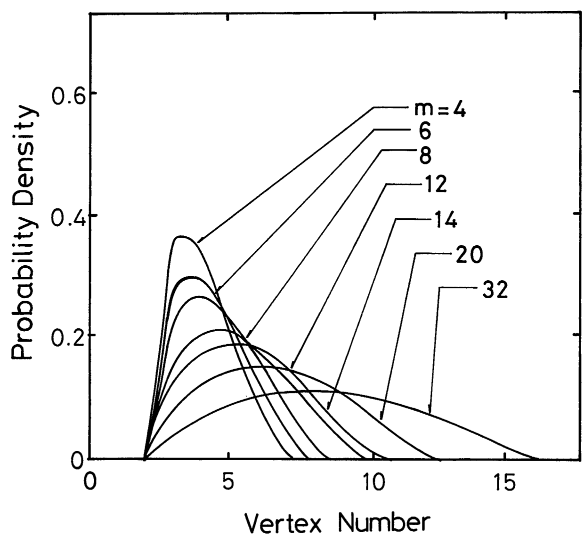

Fig. 5 Relation between the vertex number of polygons on the cross sections of polyhedra and the probability density.

on the type of the polyhedron, as seen in Fig. 2. Figure 3 shows the relations between these values and the number of polyhedron's faces, m. Equations (6) to (8) are the regression formulae for Figs. 3(a) to (c), respectively.

$$K_l = 1.22m^{0.55} \tag{6}$$

$$l_{Max} = 0.31m^{-0.53} + 1.0 \tag{7}$$

$$l_P = -0.47m^{-0.63} + 1.0 \tag{8}$$

The probability density P_l becomes the function of m and l by substituting eqs. (6) to (8) into eq. (5). In Fig. 4, several curves of l vs. P_l are drawn for the representative values of m.

One can calculate the number of the faces of a grain from its relative size in eq. (1) and the values of K_l, l_{Max} and l_P from the number of the faces in eqs. (6) to (8), and draw the distribution curve of the cross-sectional diameters of the grain by eq. (5) after substituting eqs. (6) to (8) into eq. (5). Therefore, one can calculate the probability density function of diameters of polygons observed on the cross section of a sample for any polyhedron-shape grain from the relative grain size in three dimensions.

On the other hand, the distribution of the shape (the vertex number) of the cross section of each polyhedron in Fig. 1 was also expressed by similar way to the distribution of the equivalent area diameter. The results were given in eqs. (9) to (12) and in Fig. 5.

$$P_k = K_k \cdot \exp\left[\left\{\ln\frac{k_{Max}-k}{k_{Max}(1-k_P)}\right\}\left\{\ln\frac{k-2}{k_P-2}\right\}\right] \tag{9}$$

$$K_k = 1.22m^{-0.47} \tag{10}$$

$$k_{Max} = 0.32m + 4.8 \tag{11}$$

$$k_P = 0.55k_{Max} \tag{12}$$

where

k : vertex number of the polygon on the cross section of a polyhedron
P_k : probability density of the vertex number
K_k : maximun probability density of the vertex number
k_{Max} : maximun vertex number
k_P : peak position in the grain shape distribution
m : number of grain's faces

3. Compounding of the several basic distribution functions of the cross-sectional diameters

Figure 6(a) shows the schematic distributions of the equivalent area diameter from eq. (5). The distribution curve of C_i, for example, is regarded as the apparent grain size distribution observed on an arbitrary cross section of a polycrystalline material which is composed of grains with the equivalent volume diameter D_i. Therefore, if the curves of C_1 to C_n are compounded and if the compounding ratios of f_1 to f_n are chosen to make the cpompounded curve, C_{comp}, correspond to the measured one, C_{meas}, in Fig. 6(b), then the relation between D_i and f_i indicates the distribution of the equivalent volume diameter. This is the fundamental idea of the present method.

The procedure of the estimation of the three-dimensional grain diameter distribution is as follows.

Process 1

(a) Measurment of the distribution of the equivalent area grain diameter on a cross section of the material.

(b) Preparation of the histogram as the diameter l_i vs. the measured frequency g_i.

Process 2

(a) Determination of m values for each class in the histogram of the grain size distribution from eq. (1).

(b) Substitution of the values into eqs. (6) to (8) in order to obtain the values of K_l, l_{Max} and l_P.

(c) Substitution of these values into eq. (5) in order to obtain basic distribution function of the cross-sectional diameters for each class.

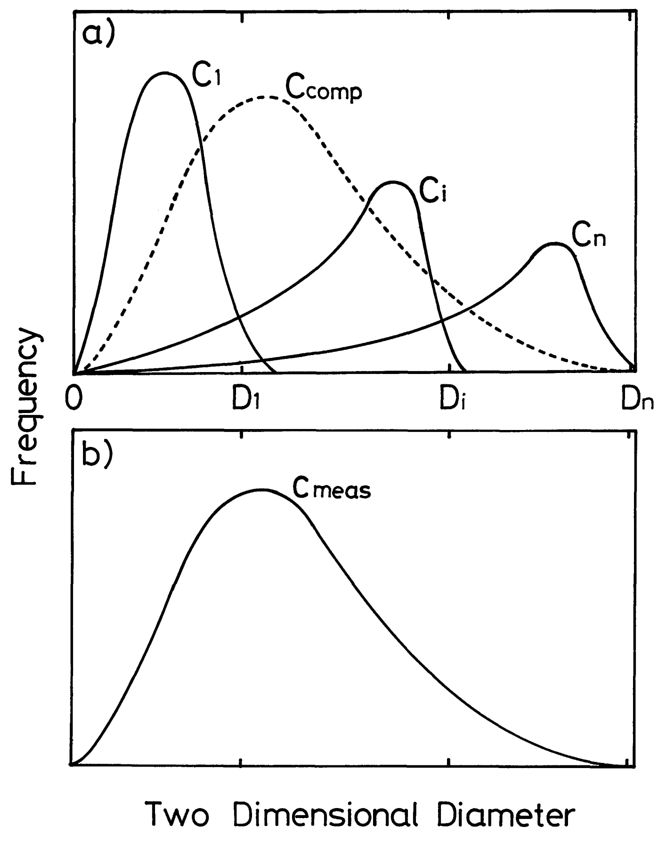

Fig. 6 Shematic curves (a) drawn from eqs. (1) and (5), and the measured grain diameter distribution on the cross section (b).

$\bar{D}$ in eq. (1) is the average equivalent volume diameter, but the value is unkown at first. Therefore, the measured average equivalent area diameter is taken as a temporary value.

Process 3

Integration of eq. (5) in order to evaluate the compounded frequency h_i of each class such that

$$h_i = f_i \cdot \int_{(i-1)w}^{i \cdot w} P_l^i \mathrm{d}l + \cdots + f_n \cdot \int_{(i-1)w}^{i \cdot w} P_l^n \mathrm{d}l + f_{i-1} \cdot \int_{(i-1)w}^{l_{Max}^{i-1}} P_l^{i-1} \mathrm{d}l \tag{13}$$

where

i : class number between 1 to n

w : class width

n : total class number

Process 4

Seeking of the compounding ratios, f_1 to f_n, to minimize the value of

$$\Delta^2 = \sum_{i=1}^{n} (h_i - g_i)^2.$$

The ratios, f_1 to f_n, indicate the values of the frequency for each class in the histogram of the three-dimensional grain diameter distribution. It should be, however, recollected that the average diameter used to obtain the m value in *Process 2* was the equivalent area diameter. The m value should be determined from the equivalent volume diameter as described in Sec. II·1.

Process 5

Calculation of the average equivalent volume diameter as

$$\bar{D}_1 = \sum_{i=1}^{n} (f_i \cdot l_i) \tag{14}$$

and feeding it back to *Process 2*.

The subscripted number of $\bar{D}$ in eq. (14) is the number of the feed-back. The operation from *Processes 2* to *5* was repeated until the change in $\bar{D}_i$ with the number of the feed-back was very small.

III. Results

The austenitic structure of a low carbon steel quenched after holding 18.0 ks at 1473 K was used as a sample to be analyzed. The number of the measured grains was approximately 1700.

The measured and the compounded distributions on the cross section are given in Fig. 7(a). There was a fairly good agreement between them. The distribution of the compounding ratios (the estimated three-dimensional diameter distribution) was given in Fig. 7(b).

The average grain diameters in the measured and the estimated distributions were 164.8 and 170.0 μm, while the values of the standard deviation were 75.8 and 69.7 μm, respectively.

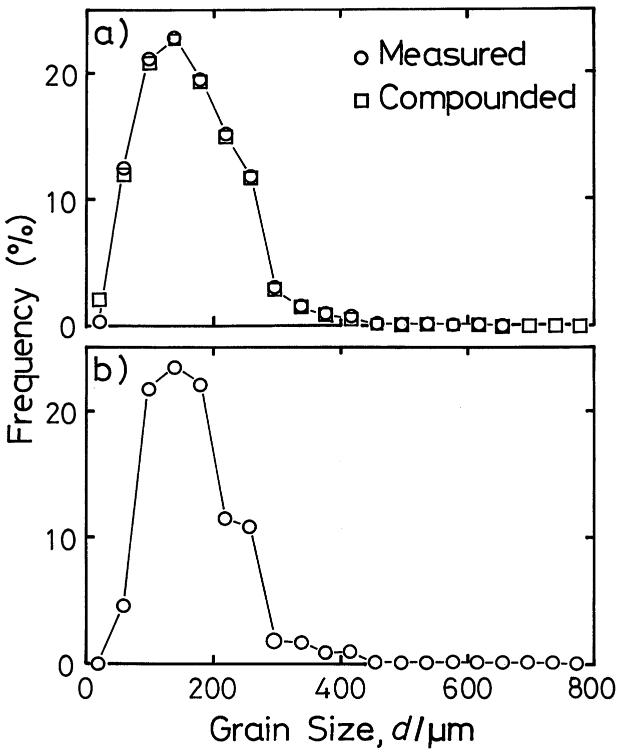

Fig. 7 Measured and compounded equivalent area diameter distributions (a) and the estimated equivalent volume diameter distribution (b).

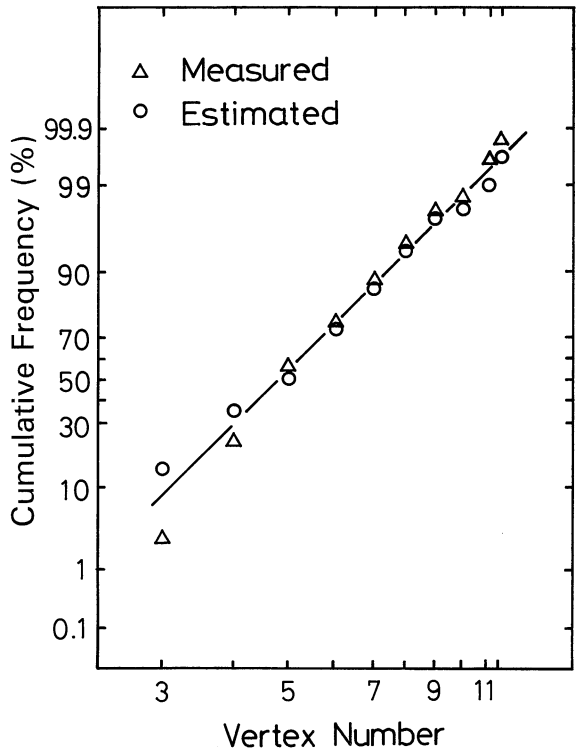

Fig. 8 Cumulative frequency of the vertex number of grains on the cross section.

IV. Discussion

The method described in Sec. II contains some processes of approximation. The first one is the grain model shown in Fig. 1, the second is the transformation from the statistical data to the mathematical equations given in Figs. 2 to 5 and eqs. (1) to (12), and the third is the curve fitting process described in Sec. II·3. These processes may have produced an error. We will discuss the accuracy of the result.

In Fig. 7, the average grain diameter of the estimated three-dimensional distribution was larger than that of the measured one, while the value of the standard deviation in the former was smaller than that in the latter. This result seemed to be reasonable from Fig. 2 or Fig. 4. These figures show that a grain with a certain equivalent volume diameter can be observed as grains with various equivalent area diameters on a cross section and that in most cases grains on the cross section are recognized as smaller ones than their true diameters in three dimensions. The features shown in those figures seemed to result in the larger average diameter and the smaller standard deviation in the distribution of equivalent volume diameters than of the equivalent area diameters.

The relations between the cumulative frequency and the measured and estimated vertex numbers on the cross section were plotted on a log-normal paper and are shown in Fig. 8. The latter relation was calculated from eq. (1) and eqs. (9) to (12) and from the estimated three-dimensional grain diameter distribution shown in Fig. 7(b). As a whole, there was a good agreement between these relations, and the respective average vertex numbers of 5.6 and 5.5 were very close.

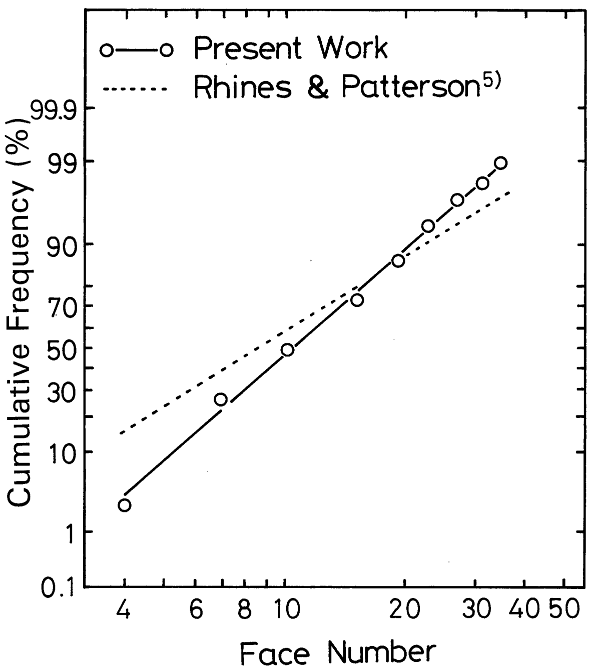

Fig. 9. Cumulative frequency of the face number of grains in three dimensions.

Figure 9 shows that the distribution of the grain face number is almost perfect log-normal. Rhines and Patterson[(5)] reported a similar result. In their work, pure

Al annealed 0.9 ks at 873 K after 6% tensile deformation was used as a sample. The distribution in their work was analyzed by disintegrating the sample into separate grains, while the present result was calculated from eq. (1) and the three-dimensional grain diameter distribution shown in Fig. 7(b). The both distributions seemed to be fairly close.

From the results shown in Figs. 8 and 9, it was suggested that the present method for the estimation of the three-dimensional grain size distribution was valid and that the error in the result was not considerable.

V. Conclusions

A new method has been proposed for the estimation of the three-dimensional grain size distribution from the measured distribution on the cross section of a polycrystalline material. This method is characterized by the employment of many types of polyhedra as the grain model and by the introduction of the formula for the apparent grain size distribution on the cross section of the material.

As an example of the application of this method, the three-dimensional grain size distribution was estimated for an austenitic structure in low carbon steel. The result seemed to be valid from the comparison between the estimated and the measured distributions of the vertex number on the cross section and the face number of grains in three dimensions.

Acknowledgment

The authors are very grateful to Prof. Ola Hunderi, the division director of SINTEF in Norwegian Institute of Technology, for many helpful conversations.

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