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# Determination of Texture from Individual Grain Orientation Measurements

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**We present a technique for determining the texture of a polycrystalline material based on the measurement of the orientation of a number of individual grains. We assumed that the sample has fiber (i.e., axisymmetric) texture and that the texture can be characterized by a function (the March–Dollase function) with a single parameter. We simulated a large number, $N$, of orientation data sets, using the March–Dollase function for a total of five different texture parameters, $r^{init}$. Using the maximum-likelihood method, we solved for the texture parameter, $r'$, that best fits each simulated data set in order to determine the distribution of $r'$ and evaluate the precision and accuracy with which $r'$ can be determined. The 90% confidence limits of the ratio $r'/r^{init}$ vary as $N^{-1/2}$ but were independent of $r^{init}$. Using the texture of slightly textured $Al_2O_3$ as determined by X-ray diffraction, we calculated the 90% confidence limits for measurements of 131 grains. The orientations of 131 grains in textured $Al_2O_3$ were measured by electron backscatter diffraction, and the texture determined from those measurements lay within these 90% confidence limits.**

## I. Introduction

With the development of techniques for the rapid determination of the orientation of single grains in the surface of a polycrystalline sample,[1] there is the potential for determining the preferred crystallographic orientation, or texture, of a sample. Typically the crystallographic texture of a polycrystalline sample is determined by diffraction techniques. Pole figure[2] and rocking curve analysis[3] are the most commonly used techniques, but, for samples that exhibit fiber texture, where the preferred orientation of the crystallites is axisymmetric about a sample axis (texture axis), Rietveld analysis of conventional theta–two theta scans can also be used to measure the texture.[4,5] All these techniques give the average texture over the area illuminated by the X-ray beam. There are cases where it may be required to measure texture over a smaller scale, either to characterize small specimens or to investigate local texture variations in a larger specimen, and, in these cases, a smaller probe, such as an electron beam in a scanning electron microscope, can be used. The questions that arise are: how to compare the texture results from different techniques; and how many individual grains must be measured in order to achieve the desired accuracy and precision. In order to compare the measurements on individual grains to the results of Rietveld analysis of theta–two theta X-ray diffractions (XRD) scans, only samples with fiber texture are considered.

Texture arises when the crystallites that make up a polycrystalline sample do not have a random arrangement of their crystallographic orientations. To describe axisymmetric texture, we define a crystallographic direction (the preferred orientation direction) that is preferentially aligned with the texture axis. The preferred orientation is typically specified as the normal, **n**, to a specific crystallographic plane, (*hkl*); the texture axis is usually a sample direction or processing axis. The conventional measure of the degree of texture is the ratio of the volume fraction of crystallites in a textured sample with **n** at a specific orientation to the texture axis to the same volume fraction for a random (or untextured) sample. This ratio is called the multiple of a random distribution, MRD. If we assume that the diffracted intensity for a specific Bragg reflection in a diffraction pattern is proportional to (among other parameters) the volume fraction of crystallites correctly oriented to diffract into the Bragg peak, then one measure of the MRD profile of a polycrystalline sample is to compare the intensity of the diffraction peaks to the intensity of the same diffraction peaks for a random or untextured sample. The functional relationship of MRD to the angle between the texture axis and the orientation of the crystallites can be modeled for the case of axisymmetric rod- or disk-shaped crystallites.

## II. Texture Model

The model MRD function chosen for this work is the March–Dollase function[5] that has been incorporated in software packages (such as GSAS[6]—a Rietveld technique) used to analyze powder diffraction data. In these techniques, a number of functions that model the experimental and sample conditions are used to calculate a model diffraction pattern. The parameters in each model are adjusted to minimize the error between the experimental data and the pattern calculated from the model functions. The function $P(r,\alpha)$ models the MRD for crystallites with orientation **n** at an angle, $\alpha$, to the texture axis using a single parameter $r$:

$$P(r,\alpha) = (r^2 \cos^2 \alpha + r^{-1} \sin^2 \alpha)^{-3/2} \quad (1)$$

We set $M = P(r,0) = r^{-3}$; $M$ is the MRD at $\alpha = 0$, and it is frequently used as a parameter for describing crystallographic texture. For a random sample, the probability of a given crystallite orientation is uniform over orientation space. The probability of a volume element with **n** lying at an angle, $\alpha$, to the texture axis is proportional to $\sin \alpha$. Thus, for the textured specimen, the probability of a given crystallite orientation is $P(r,\alpha) \sin \alpha$.

Recently developed techniques allow the measurement of the crystallographic orientation of individual grains in the surface of a polycrystalline sample.[1] Such data can be used to determine the texture of the sample by fitting the data to Eq. (1). One method of doing this requires the data to first be put into bins to form a histogram. The March–Dollase equation is then fitted to the histogram data by allowing $r$ to vary. This technique may lead to poor results if the number of grains measured is low; in addition, the results are dependent on bin width.

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An alternative technique for fitting the experimental data is the maximum-likelihood method.[7] If we assume that the sample MRD distribution is given by the March–Dollase function, Eq. (1), then the probability of making any single-grain measurement resulting in an orientation of $\alpha_i$ is $P(r,\alpha_i)\sin\alpha_i$, which we call the March–Dollase distribution. The measurements of the orientations, $\alpha_i$, of the individual grains (the angle of the crystallographic axis to the texture axis) give a set of $N$ orientations that are assumed to be randomly taken from the entire sample orientation distribution with parameter value $r$. We assume that the texture of the sample is homogeneous and that the grains on the polished surface examined in SEM are representative of the bulk grains. While this may not be true for a fired surface, it is the case for an internal section. Given an experimentally measured (or simulated) set of orientations, taken from a population with an unknown degree of texture, $r$, the problem is to determine an estimate of the degree of texture of the entire sample orientation distribution. The estimate, $r'$, is the one that maximizes the likelihood that the sample set came from a population with degree of texture, $r'$. The relationship of $r'$ to $r$ and the confidence limits on $r'$ are investigated and related to the number of orientation measurements in the set.

For the chosen estimate, $r'$, the probability of measuring a value of $\alpha_i$ is given by the probability function, $P(r',\alpha_i)\sin\alpha_i$. For the entire set of $N$ orientation measurements, the probability of getting that particular set, $L(r')$, (the likelihood function), is given by the product of the individual probability functions:

$$L(r') = \prod_{i=1}^{N} P(r',\alpha_i)\sin\alpha_i \tag{2}$$

In order to solve Eq. (2) for $r'$, we take the logarithm of both sides to convert the product to a sum. This yields

$$\ln(L) = \sum_{i=1}^{N} \ln\left(P(r',\alpha_i)\sin\alpha_i\right) \tag{3}$$

Substituting $M' = r'^{-3}$, Eq. (3) can be rewritten as

$$\ln(L) = \sum_{i=1}^{N} \ln(M') - \frac{3}{2}\ln\left[M' - (M'-1)\cos^2\alpha_i\right] + \ln(\sin\alpha_i) \tag{4}$$

In the method of maximum likelihood, the value of the estimator, $M'$, that has the highest probability is assumed to be the best value for $M$, the parameter for the whole population. In order to find the maximum of the likelihood function, the derivative of Eq. (4) with respect to $M'$ is set equal to zero and then solved for $M'$.

$$\sum_{i=1}^{N}\left[\frac{M'(1-\cos^2\alpha_i)}{\cos^2\alpha_i + M'(1-\cos^2\alpha_i)}\right] - \frac{2}{3}N = 0 \tag{5}$$

Thus, the problem of determining the texture from a set of individual grain orientations, $\alpha_i$, is reduced to finding the value of the texture parameter, $M'$, which solves Eq. (5).

## III. Simulation

In order to determine the conditions where the maximum-likelihood method yields a better estimate of the texture than fitting the data to a histogram and to determine if there is any bias in the technique, we have simulated experimental results using a Monte Carlo method. First, a random set of angles, $\alpha_i$, is chosen from a March–Dollase distribution with a given texture parameter, $r^{\text{init}}$ or $M^{\text{init}}$. Then the maximum-likelihood technique is used to find an estimator of the texture parameter, $r'$, for that set of angles. This process is repeated a large number of times, and the average and distribution of the texture estimator are compared to the initial texture parameter, $r^{\text{init}}$.

To find the probability of measuring an angle $\leq\alpha_0$ for a sample with a texture parameter $M$, the function $P(M,\alpha)\sin\alpha$ can be integrated from 0 to $\alpha_0$ to give the cumulative distribution:

$$C(M,\alpha_0) = \int_0^{\alpha_0} P(M,\alpha)\sin\alpha\, d\alpha \tag{6}$$

which varies from 0 to 1 as $\alpha_0$ varies from 0° to 90°. In order to generate a set of angles, $\alpha_i$, with a March–Dollase distribution, we want to randomly sample probability space. To do this, random numbers, $R_i$, between 0 and 1 are generated, and the value of $\alpha_0$ that results in the cumulative distribution, $C(M,\alpha_0) = R_i$, is taken as the value of $\alpha_i$. Because $dC(M,\alpha)/d\alpha = P(M,\alpha_0)\sin\alpha_0$, this results in a set of angles that fit the March–Dollase distribution.

Equation (6) can be solved and inverted to give $\cos\alpha_i$ for an initial texture parameter, $M^{\text{init}}$.

$$\cos^2\alpha_i = \frac{(1-R_i)^2 M^{\text{init}}}{1 + (1-R_i)^2(M^{\text{init}} - 1)} \tag{7}$$

Using the maximum-likelihood method, we want to solve for the $M'$ value for the generated set of $\alpha_i$ values. Substituting Eq. (7) into Eq. (5) gives

$$\sum_{i=1}^{N}\left[1 + \left(\frac{M^{\text{init}}}{M'}\right)\left(\frac{(1-R_i)^2}{1-(1-R_i)^2}\right)\right]^{-1} - \frac{2}{3}N = 0 \tag{8}$$

The variable in Eq. (8) is the ratio $M'/M^{\text{init}}$; therefore, the results are expected to fall on a master curve independent of $N$ or $M^{\text{init}}$. We also fitted the simulated data by putting the data in bins and fitting the resultant histogram. Bin widths were varied from 15° to 3.75°, and the March–Dollase distribution was fitted to the data using a least-squares analysis.

## IV. Results

Sets of $\alpha_i$ were generated for $M$ = 8, 27, 64, and 125 (corresponding to $r$ = ½, ⅓, ¼, ⅕, respectively). The sets contained 10, 20, 50, 100, 200, 500, 1000, or 10 000 grain orientations. For each set, $M'/M^{\text{init}}$ was found using Newton's method to solve Eq. (8) and this was repeated 10 000 times. The mean of $M'/M^{\text{init}}$ and the 5% and 95% limits were determined and are shown in Fig. 1. The data normalized by the initial $M^{\text{init}}$ of the simulation lay on a single curve. Also, there was a bias in the calculated values of $\overline{M'}/M^{\text{init}}$ for small numbers of grain orientations measured. For 10 orientations measured, the maximum-likelihood method gave an estimate of $M'/M^{\text{init}}$ that was 4% too small, but the error decreased to 2% for 20 orientations measured and decreased to <1% when >40 orientations were measured.

The 90% confidence limits decreased as the number of orientations measured increased. The accuracy (standard deviation) of $M'/M^{\text{init}}$ was found to vary approximately as $N^{-1/2}$, which suggests that the main source of error is random error and not a systematic error due to the maximum-likelihood technique. The slope of the standard deviation as a function of $N$ was −0.518. When only the values for >100 orientations were used, the slope decreased to −0.503.

For fitting the March–Dollase distribution to a histogram of the data, the results are found to be dependent on the bin width chosen. The value of $M'/M^{\text{init}}$ is always smaller than the corresponding value from the maximum-likelihood method, except for the case of a 15° bin size and an $M^{\text{init}}$ value of 125 ($r^{\text{init}}$ = 0.2). Thus, the results are dependent on the initial texture value chosen for the simulation and do not fall on a single curve when normalized by $M^{\text{init}}$. When both the bin size and data set are small, it is likely that some bins have no orientations, which makes the fitting inaccurate. When analyz-

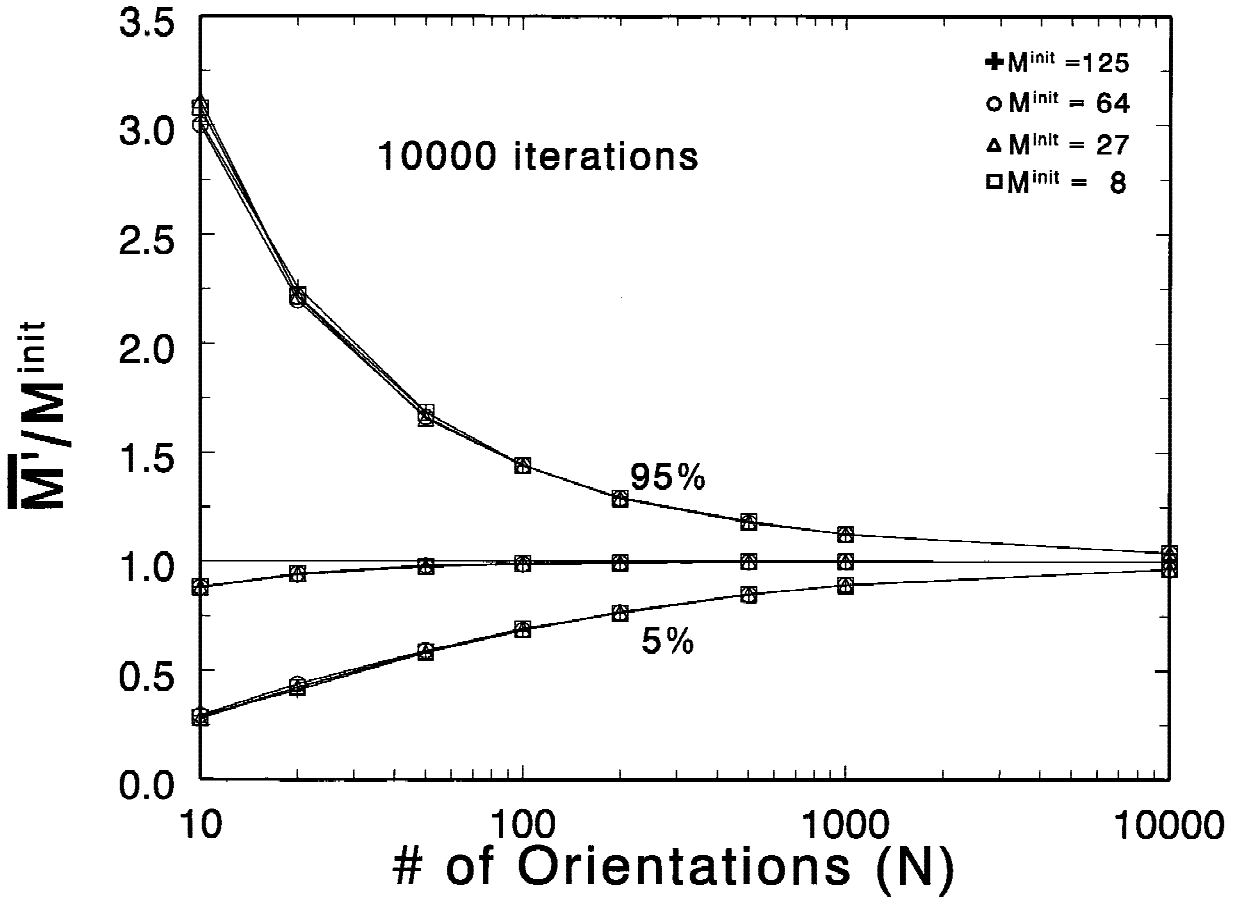


**Fig. 1.** Maximum-likelihood texture results ($\overline{M}'/M^{init}$) from simulations of $N$ individual grain orientations with varying texture levels ($M^{init}$).

ing highly textured samples, orientation density can change significantly across the bin, so that the center of the bin does not accurately represent the average value of the data in the bin. While there are methods for adjusting the bin size and location based upon the data, these methods can introduce artifacts that can be avoided by using the maximum-likelihood method.

The maximum-likelihood method was used to calculate the degree of texture for a set of 131 grain orientations measured using backscattered Kukuchi patterns (EBSP) generated in an SEM.[8] The sample was a polycrystalline $Al_2O_3$ substrate (SRM 1976[9]), and the fired surface was examined. The grain size was from 1 to 10 μm, and the orientation measurements were taken every 100 μm, so that the sampling position was chosen at random, no grain was sampled twice, and there was no knowledge of the size of each sampled grain. The EBSP data were analyzed both by using the maximum-likelihood method and by putting the data into bins and fitting the March–Dollase distribution to the resulting histogram. From the histogram data, $M' = 6.81$ ($r = 0.5277$); from the maximum-likelihood method, $M' = 6.20$ ($r = 0.5443$).

For comparison, the sample texture was also measured using an X-ray technique. Measurement of weak [0001] texture in $Al_2O_3$ cannot be performed simply by measuring the intensity diffracted by the basal planes using the 0006 or 000.12 peaks, because both those peaks have low structure factors and are extremely weak; therefore, they can be used only in rocking curve or single pole figure measurements on $Al_2O_3$ samples with considerable texture. Therefore, the texture was measured by performing a Rietveld refinement of standard theta–two theta XRD data using GSAS. From the Rietveld refinement, the texture parameter $M$ is 4.28 ($r = 0.616$), indicating that the sample has some small degree of [0001] texture for a texture axis normal to the surface of the substrate.

In order to estimate the confidence limits, the simulation was run for the case of 131 orientations with an $M^{init}$ of 6.20. The average of 10 000 iterations was $M = 6.14$ ($r = 0.5460$), and the normalized 90% confidence limits for 131 orientations were $M'(5\%)/M^{init} = 0.723$ and $M'(95\%)/M^{init} = 1.373$. This implies that, based on the measured $M'$, the true $M$ of the sample, with a 90% confidence limit, would lie between 4.46 and 8.00. The $M$ value determined from the measurement of the orientation of individual grains was above the measured X-ray $M$ value. For an $M^{init}$ of 4.28 ($r = 0.616$) the simulation yielded an $M$ of 4.07 ($r = 0.626$) with 90% confidence limits of $M = +2.23, -1.29$ ($r = +0.1504, -0.1278$, respectively). Thus, for measurements of 131 grains from a sample with a texture parameter of 4.28, 90% of the time, the measurements would lie between $M = 2.78$ and 6.30.

The single measurements we have made are within the range of expected values of the texture parameter for the sample, but they are at the high end of the range. This reflects the errors associated with the small number of grains and also may be due to the differences between the surface grains measured by EBSP and the grains measured by XRD. Thus, the discrepancy between the X-ray and EBSP measurements of the texture parameter is within the expected variation of the techniques and does not indicate a difference in texture.

The simulation we have performed allows an estimation of how different the texture parameters must be in different areas of a sample, or between two samples, in order for the texture of the regions to be considered distinct. As the above measurements show, different values of $M$ may just reflect the statistical variations for small sample size. Any technique based on individual grain orientation measurements is subject to these uncertainties. Clearly, if XRD techniques can be used, the results are a more accurate measure of the texture of the whole sample than when using the individual grain orientation measurements. The advantage of individual grain orientation mea-

surements is that the technique used to make the measurements has a probe that is smaller than the scale of the microstructure. This allows texture to be determined on a much finer scale as compared with X-ray techniques. Thus, local variations in texture can be measured. The advantage of the maximum-likelihood method of analyzing the orientation data, compared to a histogram, is that the results do not depend on the initial conditions, such as bin width for fitting a histogram.

## V. Summary

We have developed a method for evaluating the accuracy of the measurement of crystallographic texture of polycrystalline materials from individual grain orientation measurements. We assume that, for samples that have fiber texture, the March–Dollase distribution describes the orientation distribution of the crystallites. By fitting the orientation measurements to the March–Dollase distribution, the texture can be determined. The fitting is based on the maximum-likelihood method, which yields a better fit for small numbers of measured grains than the techniques of binning the data into a histogram and fitting the texture function to a histogram. In order to determine the accuracy and precision of the technique, we have simulated grain orientation distributions and fitted them to the March–Dollase distribution using the maximum-likelihood method. It was found that there was a bias in the calculated texture for small numbers of grains measured, but if >100 orientations were measured, the error was <1%. The 90% confidence limits decreased as $N^{-1/2}$, where $N$ was the number of orientations measured.

In order to compare the method to standard XRD techniques, measurements were made on a polycrystalline $Al_2O_3$ sample. The method was used to compare the results from the measurement of the 131 grains on the fired surface of the sample, and the texture parameters from the two techniques were found to be within the 90% confidence limits. Thus, the maximum-likelihood method was found to be a good method of fitting a model texture function to a data set of orientations to determine the texture of a polycrystalline sample.

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