Quantitative neutron diffraction texture measurement applied to $\alpha$-phase alumina and Ti$_3$AlC$_2$

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Many research topics in condensed matter research, materials science and the life sciences make use of crystallographic methods to study crystalline and non-crystalline matter with neutrons, X-rays and electrons. Articles published in the Journal of Applied Crystallography focus on these methods and their use in identifying structural and diffusion-controlled phase transformations, structure-property relationships, structural changes of defects, interfaces and surfaces, etc. Developments of instrumentation and crystallographic apparatus, theory and interpretation, numerical analysis and other related subjects are also covered. The journal is the primary place where crystallographic computer program information is published.
Quantitative neutron diffraction texture measurement applied to α-phase alumina and Ti₃AlC₂

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Orientation distribution functions, essential for making a quantitative connection between single-crystal and polycrystal properties, have been determined for extruded α-phase alumina, hot-pressed Ti₃AlC₂ and cold isostatically pressed Ti₃AlC₂ using experimental pole figures recorded on the fixed-wavelength neutron diffractometer KOWARI. Some practical improvements to the calculation of the pole-figure density from the raw area-detector data, and for constructing pole figures on an n × n' hemispherical grid, are presented. The textures give some insight into particle flow during manufacture. Directly measured material textures were compared with one-dimensional pole density functions, such as the March and Rietveld functions commonly used for the correction of preferred orientation in Rietveld refinements, as a means of assessing the utility of the latter for the computation of diffraction elastic constants and other polycrystal properties from a given set of single-crystal properties.

1. Introduction

The physical properties of a polycrystalline material depend on the properties of the individual crystallites and their orientations, as well as the effects of grain boundaries. Evidently, the computation of physical properties should take these factors into account. Crystallites within the polycrystal are not always uniformly distributed. This non-randomness or texture mainly arises from manufacturing, geological or biological processes and may be beneficial or disadvantageous. It can have a significant effect on the average properties of the polycrystal, owing to the inherent anisotropy of most single-crystal properties.

One example where texture can have a strong effect on the polycrystal properties is in elastic constants and their computation. With an appropriate micro-mechanical approximation, methods have been developed for the determination of the bulk elastic constants. More difficult is the computation of the diffraction elastic constants, which relate an applied or residual stress to strains measured by X-ray or neutron diffraction. This is particularly important for the estimation of single-crystal elastic constants (SCEC) for new materials when sizable single crystals are difficult to grow, and also in residual stress analysis. Several groups have published accounts of the determination of SCEC from polycrystalline materials based on in situ loading during neutron or synchrotron diffraction studies (Hauk & Kockelmann, 1979; Hayakawa et al., 1985; Kisi & Howard, 1998; Gnäupel-Herold et al., 1998; Howard & Kisi, 1999; Mattheis et al., 2001). In the diffraction situation, the scattering vector defines a unique direction common to all crystallites contributing to a given reflection. Averaging over all crystallites having this common direction is accomplished by integration around the scattering vector. The simplest case is that of an un-textured polycrystalline material (Howard & Kisi, 1999) where the bulk or diffraction properties can be easily calculated for a given set of SCEC using equations in Tables 1 and 2 of Howard & Kisi (1999) (see, for example, Kisi et al., 2010). However, owing to anisotropy of the single-crystal properties and varying proportions of crystallites with different orientations in a textured polycrystalline material, the properties of the textured material will be altered. Mattheis et al. (2001) have considered the concept of texture-weighted averages but specific methods for implementing these were not discussed.

In general, the full description of a polycrystalline structure, by specifying the orientation of the crystallographic axes of each grain as well as its shape, size and position within a specimen, is complicated. The practical description usually adopts a statistical distribution function, such as the mean grain shape or the grain size distribution, and so on. The orientation distribution function (ODF) is widely used to represent the statistical form of all crystallographic orientations in a polycrystalline material (Roe, 1965). If a dedicated texture scanning diffractometer is not available, a few one-dimensional pole density models based upon a particular vector [HKL] are widely adopted to deal with preferred orientation (texture) in powder diffraction. Rietveld (1969) proposed describing preferred orientation in an axially...
symmetric sample using a single texture parameter \((G_z)\) density function and a scale factor \(G_2\):

\[
P_H(\Phi) = G_z \exp(-G_z \Phi^2)
\]

(1)

where \(\Phi\) is the angle the preferred orientation vector \([HKL]\) makes with the axis of symmetry. Setting \(G_1 = 0\) leads to \(P_H(\Phi) = 1\) which makes no preferred orientation. The main disadvantage of the Rietveld function is that it is not normalized. Dollase (1986) compared a number of different models and showed that the March function,

\[
P_H(\Phi) = (R^2 \cos^2 \Phi + R^{-1} \sin^2 \Phi)^{-3/2},
\]

(2)

has a number of advantages in dealing with material texture in powder diffraction patterns. For example, the March function is correctly normalized such that the integral of the density function over the sphere is independent of the value of the March coefficient \(R\); therefore it can be used to handle preferred orientation in quantitative phase analysis (Hill, 1993; Hill & Howard, 1987; Howard & Kisi, 2000). Although Dollase suggested a follow-up investigation comparing the March function with pole distributions from full texture analysis, this study appears not to have been completed. The possibility has been raised of using a one-dimensional pole density function determined during Rietveld refinement to apply the required texture weighting to the computation of diffraction elastic constants (Kisi & Howard, 2008). The procedure is one of forming an intermediate Cartesian coordinate system defined such that the scattering vector \((\mathbf{x})\), i.e. the direction of applied stress \((\mathbf{x})\) and preferred orientation vector \([HKL]\) are coplanar. Under these conditions, the distribution of crystal poles receiving the applied stress is the same as the distribution of the preferred orientation vector and hence the integration about the scattering vector may be weighted by the distribution of \(HKL\). The veracity of using the March function for this purpose has not been fully explored, although it is expected to be relatively robust for materials prone to one-dimensional texture and rather less so for other materials.

The elasto-plastic self-consistent model has been reported to simulate texture development during plastic deformation (Tome, 1999). However, to have a reasonable chance of simulating the texture, precise manufacturing conditions must be known, which is reasonable for experimental metallurgical samples but rather less so for geological, ceramic and industrial metallurgical products.

A more rigorous approach would be to measure the full three-dimensional texture of the material and use the measured ODF of the sample to weight the integral of the elastic compliances around the scattering vector in each crystal direction of interest. If the exact population of the grains is taken into account in the computation of the elastic properties of the polycrystalline material, the most accurate set of diffraction elastic constants will be determined from a given set of single-crystal elastic constants and micromechanical state. This will extend the determination of SCEC from polycrystalline materials by \(\text{in situ}\) loading experiments to include textured materials. Furthermore, with precise texture measurement, it should be possible to improve the accuracy of the determination of residual stress in engineering materials. To be effective, these methods require that the distribution of poles along the integration path around the chosen scattering vector be accurately determined. Quantitative texture analysis therefore becomes an essential tool for analysing the material properties of textured samples.

There are many different methods to measure the ODF of a polycrystalline material. These include electron back-scattered diffraction within scanning electron microscopes, laboratory and synchrotron X-ray diffraction, and neutron diffraction. Neutron diffraction texture measurement has several advantages. These include low neutron absorption by most materials, enabling a statistically larger population of crystallites to be sampled with minimal attenuation effects to be corrected. Textures can be measured and their evolution tracked during various types of \(\text{in situ}\) experiment under applied stress, or under an electric or a magnetic field. Unfortunately, unlike commercial X-ray texture diffractometers, standard software for pre-processing neutron diffractometer texture data is generally written for each diffractometer. Here we present a generalized methodology for pre-processing neutron diffraction area-detector data, which meshes seamlessly with the MTEX (Hielscher & Schaeben, 2008) texture analysis toolbox in MATLAB (The MathWorks Inc., Natick, MA, USA). The method is used to analyse the texture of extruded \(\alpha-\text{Al}_2\text{O}_3\) ceramic, uniaxially hot-pressed \(\text{Ti}_3\text{AlC}_2\) and cold isostatically pressed \(\text{Ti}_3\text{AlC}_2\). Integrated one-dimensional pole densities for various \(hkl\) are extracted for comparison with the same pole densities obtained from Rietveld refinements using the March and Rietveld preferred orientation functions.

2. Experimental setup

The samples used in this work were (i) an 8 mm-diameter cylinder of 100% dense \(\alpha-\text{Al}_2\text{O}_3\) sown from a 1 m-long commercially produced rod, (ii) a 100% dense \(10.5 \times 10 \times 18.5\ mm\ \text{Ti}_3\text{AlC}_2\) block produced by uniaxial hot-pressing of pre-reacted powders at 1673 K for 20 min and (iii) a 100% dense 10 mm-diameter 12.2 mm-high \(\text{Ti}_3\text{AlC}_2\) cylinder produced by cold isostatically pressing pre-reacted powders at 40 MPa followed by pressureless sintering at 1723 K for 2 h. Standard laboratory X-ray diffraction and powder neutron diffraction patterns were used to characterize the samples before texture measurement.

The texture experiments were conducted on the residual stress diffractometer KOWARI at the OPAL research reactor at ANSTO in Sydney. The incident wavelength was set to 1.7 Å using a double-focusing Si-wafer monochromator. With the samples mounted on an Eulerian cradle, diffracted intensities for a set of chosen diffraction peaks were recorded by setting the detector \(2\theta\) and then tilting \((\chi)\) and rotating \((\phi)\) the sample, typically on a \(15 \times 5^\circ\) grid in a three-dimensional sphere. The detector covers at least \(15^\circ\) in the vertical direction and so may be further partitioned to give finer angular resolution in the recorded pole figures. Fig. 1 shows typical raw
data from the area detector showing a curved portion of a Debye–Scherrer ring for each reflection. Detected intensity is indicated by the darkness of each pixel. If the sample is textured, the diffracted intensity around the Debye–Scherrer ring is modulated according to the orientation of the sample. The variation of the intensity of these peaks can then be processed to derive three-dimensional pole figures for any pole.

3. Analysis of experimental data

Several data-analysis and texture-presentation processes are introduced here. Two of the steps were completed using MTEX (Hielscher & Schaeben, 2008) which was developed for texture analysis using MATLAB. All additional computer programming was completed in the MATLAB environment.

3.1. Understanding the detector image and raw data structure

The detector used is square with an area of \(280 \times 280\) mm containing 421 channels in both the vertical and horizontal directions, giving \(421 \times 421\) pixels in the detector image. Fig. 1 shows an image of the detector obtained from the texture measurement of an \(\alpha\)-phase \(\text{Al}_2\text{O}_3\) sample. The peak shown inside the marked area comes from crystallites that have the direction normal to their \(\{11\bar{2}3\}\) planes (hereafter \(\{11\bar{2}3\}\) poles) aligned with the scattering vector.

A full texture determination requires data from a number of reflections. For each reflection, the sample is scanned, recording 72 area-detector images at \(5^\circ\) steps around the \(\varphi\) axis at a fixed value of the azimuthal angle \(\chi\). The raw data for each scan are stored in an HDF5-format file, each file containing a matrix with \(421 \times 421 \times 72\) cells. A full hemisphere will be covered by running seven such scans, taken at \(15^\circ\) intervals in \(\chi\). This amounts to a four-dimensional matrix which contains \(421 \times 421 \times 72 \times 7\) cells and provides the intensity information from one or more reflections in a single frame in all \(5 \times 5^\circ\) orientations. Unlike time-of-flight neutron diffractometers, a fixed-wavelength diffractometer cannot generally give the full range of peaks in one detector image and the detector \(2\theta\) position must be changed.

The minimum number of reflections (pole figures) to be measured in order to have a unique solution depends on the crystal symmetry (Bunge & Esling, 1982). At least four pole figures are required to completely specify the texture of a hexagonal material. In general, increasing the number of reflections measured beyond the minimum can reduce the error in texture calculations. Over-determined problems can be solved by a least-squares method.

3.2. Circumferential summation of the neutron intensity

The integration of neutron counts captured by the detector is not straightforward because of the curvature of the Debye–Scherrer rings and the flatness of the detector. The detector plane on KOWARI is generally not perpendicular to the axis of the diffraction cone. That means the intersection of the diffraction cone with the two-dimensional area detector will form a hyperbola in the detector plane and the radius of curvature is not constant along the curve. The key point is that the actual diffraction angle \(2\theta\) is constant at any position on a diffraction cone. The intensities of those cells which have the same angle of \(2\theta\) can be summed to get the total number of neutrons corresponding to the angle \(2\theta\). Before the \(2\theta\) calculation, the position of a pixel corresponding to an intensity stored in an array will have its position converted from array indices to a point referred to two-dimensional Cartesian coordinates; then the position of this point will be transformed to \(\gamma\) and \(2\theta\) in a polar coordinate system [equation (3)]. The transformation is similar to the method described by Bunge & Klein (1996); however, our method is more straightforward:

\[
I_{\text{raw}} \rightarrow I_{(x,y)} \rightarrow I_{(2\theta,\gamma)}.
\]  

Fig. 2 shows the coordinate system for the first transformation. A Cartesian coordinate system in \(x\) and \(y\) is invoked.

---

**Figure 1**
Detector image from \(\alpha\)-\(\text{Al}_2\text{O}_3\) \((421 \times 421\) pixels\) and partitions of \(5^\circ\) in \(\gamma\) for data processing. \(x\) and \(y\) axis labels refer to raw detector channels.

**Figure 2**
The axis convention for the Cartesian system on the KOWARI area detector.
The values of \(x\) and \(y\) can be easily calculated from the detector architecture. The centre pixel \((211, 211)\) is considered to be the origin of the coordinate system, \(m_i\) and \(n_i\) are the total number of channels along the \(x\) and \(y\) axes, respectively (in our case both \(m_i\) and \(n_i\) are 421), and \(l_i\) and \(l_y\) are the length of the detector in millimetres along the \(x\) and \(y\) directions (in our case both are 280 mm):

\[
\begin{align*}
x &= l_i (211 - m)/m_i, \\
y &= l_y (211 - n)/n_i.
\end{align*}
\]

(4)

A three-dimensional coordinate system \((X_L, Y_L, Z_L)\), centred on the sample position, is also required (Fig. 2). The incident neutron beam travels along \(X_L\), the \(2\theta\) axis is \(Z_L\) and \(Y_L\) is mutually perpendicular. The detector centre lies in the \(X_L Y_L\) plane with the central pixel at \(2\theta = \alpha\). Once the position of a pixel in the Cartesian coordinate system is known, its 2\theta and angle \(\gamma\) above or below the mid-plane of the Debye–Scherrer ring can be calculated, referring to Fig. 3 and using the following relationships:

\[
2\theta = \cos^{-1}\left[\frac{x \sin \alpha + d \cos \alpha}{(d^2 + x^2 + y^2)^{1/2}}\right] \quad (0 < 2\theta < \pi),
\]

(5)

\[
\gamma = \sin^{-1}\left[\frac{y}{(y^2 + (x \cos \alpha + d \sin \alpha)^2)^{1/2}}\right] \quad \left(-\frac{\pi}{2} < \gamma < \frac{\pi}{2}\right).
\]

(6)

The next step is to total the counts for a particular \(2\theta\) within a \(5^\circ\) segment of the Debye–Scherrer ring. This is equivalent to collapsing the observed intensities within the \(5^\circ\) segment (Fig. 1) onto the centreline of the detector. As the values of \(m\) and \(n\) are integers, whereas \(2\theta\) and \(\gamma\) are real numbers, these need to be discretized before numerical integration. When an intensity corresponding to a position \((x, y)\) on the detector has been transformed to intensity in the \(2\theta–\gamma\) domain and rotated to lie on the centreline, in the general case, its position will no longer exactly match the centre of the corresponding pixel on the centreline. Taking the integration cell on the centreline to be a single pixel, and supposing this matches the peak full width at half-maximum (FWHM), it is beneficial to distribute the intensity to adjacent integral cells to accommodate the Gaussian distribution of neutrons captured by the detector in order to have a smoother integration curve of neutron intensity with respect to \(2\theta\).

As shown in Fig. 4, when a cell intensity \(I_{i(2\theta, \gamma)}\) rotates to lie within the bounds of the integral cell \(i_{(2\theta, \gamma)}\), it is not rigorous to simply attribute the whole intensity to the cell \(i_{(2\theta, \gamma)}\). To correct this effect, the whole intensity can be divided into three main parts. The proportion for the cell \(i_{(2\theta, \gamma)}\) is the largest; that for \(i_{(2\theta) + 1}\) is the second largest; and the cell \(i_{(2\theta) - 1}\) is the smallest. If the proper normal distribution parameter \(\sigma\) and integral range of \(2\theta\) have been chosen, we may ignore the very small proportion of intensity that falls into cell \(i_{(2\theta) + 2}\) and put this part into the cell \(i_{(2\theta) + 1}\).

Such an argument can be used to evaluate the proportion of any intensity \(I(2\theta, \gamma)\) falling in the \(2\theta\) range \(2\theta_{i-2} \leq 2\theta < 2\theta_{i+1}\) [i.e. in the \((i - 1)\)th, \(i\)th or \((i + 1)\)th cell] to be counted in the \(i\)th cell as

\[
p_i = \frac{1}{(m(2\theta_i) - m(2\theta_{i-1}))} \int_{2\theta_{i-1}}^{2\theta_i} I(2\theta, \gamma) \mathrm{d}2\theta \quad (2\theta_i \leq 2\theta < 2\theta_{i+1}),
\]

\[
\frac{1}{(m(2\theta_i) - m(2\theta_{i-1}))} \int_{2\theta_{i-1}}^{2\theta_{i-1} + 2\theta_{i-2}} I(2\theta, \gamma) \mathrm{d}2\theta - \frac{1}{(m(2\theta_i) - m(2\theta_{i-1}))} \int_{2\theta_{i-1}}^{2\theta_{i-1} + 2\theta_{i-2}} I(2\theta, \gamma) \mathrm{d}2\theta \quad (2\theta_{i-1} \leq 2\theta < 2\theta_i),
\]

\[
\frac{1}{(m(2\theta_i) - m(2\theta_{i-1}))} \int_{2\theta_{i-1} + 2\theta_{i-2}}^{2\theta_{i-1}} I(2\theta, \gamma) \mathrm{d}2\theta \quad (2\theta_{i+1} \leq 2\theta < 2\theta_{i-1}).
\]

(7)

The area detector was separated into three partitions each \(5^\circ\) of \(\gamma\) high and so the intensity of three different areas was integrated separately from each detector image. Next, the intensity of neutrons within the \(5 \times 5^\circ\) area was summed to give totals within the integration unit \(i\) (Fig. 4):

\[
I_i = \sum_{\gamma' = \Delta\gamma, 2\theta_{i-2} \leq 2\theta < 2\theta_{i+1}} p_i I(2\theta, \gamma'),
\]

(8)

where the \(I(2\theta, \gamma)\) are the counts recorded in pixels with \(2\theta_{i-2} \leq 2\theta < 2\theta_{i+1}\) in the desired range of \(\gamma\). If more partitions
are required for better $\gamma$ resolution, $\Delta\gamma$ simply needs to be changed from 2.5° to a smaller angle.

3.3. Raw pole-figure output

After defining the bounds of $2\theta$ and $\gamma$, and applying the procedure in §3.2 for a chosen reflection, the intensities of the 421 × 421 cells in one detector image (i.e. at particular $\alpha$) are processed into three one-dimensional arrays of intensity with respect to $2\theta$ (summed around the Debye–Schererr ring). The width of the array ($p$) is defined by the left and right boundary of the chosen peak. The number of data has reduced significantly to an array of dimension 1512 by $C$. The detector position ($\alpha$) and sample rotation and tilt angles ($\varphi$ and $\gamma$) are now amended to account for the $\gamma$ offsets. These data were further reduced to the 1512 integrated intensities defining the measured pole figure for a given $hk\ell$ using a customized Gaussian fitting function developed in MATLAB. To make the Gaussian fitting function work efficiently, reasonable starting values for the parameters are required. Usually, the peak position, FWHM and background intensity are quite consistent regardless of the sample orientation; the estimated values from a single peak are good enough for processing all the detector images. However, the intensities of the chosen peak can vary significantly and this variation may cause the curve fitting function to be unable to find a solution in a limited number of iterations. To avoid this issue, an initial estimate for peak intensity is obtained for each data set from the difference between the averaged background intensity and the maximum peak intensity. In obtaining this starting value for intensity, the whole detector image (rather than 5°/C14) was collapsed onto the detector centreline according to the method described in §3.2 to improve the statistical precision. The pole density resulting from the integrations is stored in an output array with corresponding values of $\varphi$ and $\gamma$. Discarding the data set at $-5°$ and $95°$ of $\gamma$, a total of 1368 sets of data were then sorted in ascending order according to $\gamma$ and $\varphi$, and saved for use in the next process.

3.4. Pole-figure data input and ODF calculation

Input of the measured pole-figure data into the MTEX toolbox was via a script containing the crystal class, lattice parameters, sample symmetry and file format. For each material, pole-figure densities for several $hk\ell$ were input to MTEX and the pole figure for each reflection plotted to help visualize and check the raw intensity data. ODFs for each sample were calculated by the MTEX solver by iterative solution of equation (9):

$$f_{\text{est}} = \sum_{j=1}^{N_r} \sum_{i=1}^{N_r} \frac{|\delta_iQ(h_i, r_j) - I_{ij}|^2}{I_{ij}},$$

where the index $j$ runs over the densities recorded for a measured pole figure, the index $i$ runs over different reflections $h_i$ and $r_j$ are directions relative to the specimen axis. $\delta_i$ is the weight for each reflection, and $f$ is the pole density function, which depends on reflection $h_i$, $r_j$ and the operator $Q$ of the ODF. A full description of the estimator and the algorithm is given by Hielscher & Schaeben (2008). It is worth highlighting here that the ODF calculated from MTEX has already been properly normalized.

4. Experimental results

4.1. Texture of commercial $\alpha$-phase alumina

Fig. 5 shows a pole density figure (PDF) of a commercial alumina rod for different poles identified by their Miller–Bravais indices. Clearly the majority of crystallites have their [0001] poles aligned perpendicular to the cylinder axis $(z)$ whilst very few are aligned parallel with the $z$ axis of the specimen. The orientation distribution of the other poles shown in this plot is entirely consistent with the [0001] pole. For instance, since the normal of the [1010] planes is perpendicular to the normal of the [0001] planes, the [1010] pole figure has a high density parallel to the $z$ axis and a very low density in the $xy$ plane of the specimen coordinate axes. In general, the texture appears to be primarily of the fibre type with [001] normal to the cylinder axis. The fibre texture is not perfect as there is a small rise in the [0001] pole density near the centre of the figure. From this example, it is clear that the material texture is directly related to the manufacturing process, which involves cold extrusion of the green ceramic paste through a die followed by sintering. As the ceramic particles pass through the die, they assume a preferred alignment with the material flow according to their shape. Since the particle shape is linked to the crystallographic axes, a strong fibre texture develops. As the particles along the centreline of the die experience little shear, the small rise at the centre of the [0001] pole figure is likely due to this effect.

Owing to the different manufacturing orientation, the texture direction in our specimen is perpendicular to that observed by neutron diffraction for alumina slip cast under a 10 T magnetic field and sintered (Guilmeau et al., 2005). In those results, the initial alignment in the field is greatly enhanced by preferred grain growth during sintering. Hence, it

Figure 5
Reconstructed pole figures for six lattice directions in an $\alpha$-Al$_2$O$_3$ cylinder with the cylinder axis pointing out of the figure.
is possible that the texture observed here is likewise initiated during early processing and enhanced during sintering.

4.2. Texture of hot-pressed Ti$_3$AlC$_2$

The longer $z$ axis of the $10.5 \times 10 \times 18.5$ mm Ti$_3$AlC$_2$ specimen was parallel to the direction of applied stress during compression at high temperature. From the pole-figure plots in Fig. 6, it is apparent that a majority of crystallites have their $c$ axes ($\{0001\}$ poles) aligned with the $z$ axis of the specimen coordinate. The maximum density in this pole figure is nearly 45 times its minimum value. As shown in the figure, the pole figures of other Miller indices are purely related to this pole i.e. once again the texture is of the fibre type. This gives us a clear image that the process of compression changes the orientation of the crystallites at high temperature. In this case, the applied pressure deformed the material along the $z$ axis of the specimen coordinate system; and this deformation aligned the $c$ axis of the crystallites along this flow direction at 1673 K. Therefore the crystallites are platelike with the $c$ axis normal to the plate surface. The normalized texture index (Bunge, 1982),

$$J = \frac{\bar{f} (f(g))}{d g},$$

for this hot-pressed Ti$_3$AlC$_2$ is 3.153, which is considered to be a very strong texture.

4.3. Texture of cold isostatically pressed (CIP) Ti$_3$AlC$_2$

This example provides a contrast with the strongly textured samples in §4.1 and 4.2 as well as demonstrating an alternative way of viewing texture information. Instead of the PDF for various poles of interest, Fig. 7 shows a plot of the ODF for the Ti$_3$AlC$_2$ CIP sample directly in a series of sections.

From Fig. 7 it can be seen that the distribution of crystallites in the material is close to random. The maximum and minimum of the preferred orientation are 1.2 and 0.9 compared to the maximum 7.3 and minimum 0.16 for the hot-pressed sample of the same compound. In contrast to the texture index of 3.153 for the hot-pressed sample, the texture index for CIP Ti$_3$AlC$_2$ is only 1.0026 (1 indicating no texture). The geometric shape of the compressed solid sample before trimming was slightly elongated with a neck in the middle. Though the compressive stress was applied to the powder isostatically, the deformation of the material was not perfectly isotropic. This non-uniformity of deformation produced a very slight texture. It is considered that an even weaker texture would be observed if the powder was compressed in a more uniform spherical shape. Therefore, it should be possible to produce a completely un-textured sample using the CIP method.

4.4. Evaluating the distribution function around a specific scattering vector

Though visualization of the ODF is a basic requirement for the quantitative analysis of texture, it is rather important that texture information be included in the quantitative analysis of the material properties. In particular, for the determination of single-crystal elastic constants using diffraction, the extraction of the required pole density around a chosen scattering vector is the key issue with which we need to deal. Such experiments, which combine in situ loading with neutron diffraction, normally have the scattering vector either parallel or perpendicular to the loading direction. For the former the results are largely independent of texture but for the latter they are strongly affected.

Fig. 8(a) shows a three-dimensional plot of the $\{0001\}$ pole figure of the alumina (see §4.1). For this example, we assume that the applied load aligns with the $z$ axis and the scattering vector aligns with the $y$ axis of the specimen coordinate system, as shown. The macroscopic strain measured along the scattering vector is connected with the strain measured along the loading direction by a bulk property, Poisson’s ratio $\nu$. This is the macroscopic sum of the individual response of all the crystallites in the material. However, when the strain is measured by diffraction, only those crystallites with a common...
scattering vector contribute to the average strain measured from a given reflection. The observed strain measured along the scattering vector is therefore strongly related to the ODF of the material.

The texture of the α-phase alumina we examined is directly related to the preferred orientation of crystallites in the [0001] direction. As shown in Fig. 8(a), ω is the angle between the scattering vector for an individual group of crystallites all contributing to the reflection hkl and the [0001] direction, and it can be easily calculated from the respective Miller indices. The lattice strain that is measured along the scattering vector for each individual group of crystallites is a mean value of the strains within every single crystallite that has the same lattice direction aligned with the scattering vector (within the gauge volume). Though all these crystallites have a common lattice direction aligned with the scattering vector, there is still one degree of freedom available as they are rotated different amounts around that lattice direction (angle ξ in Fig. 8a). It is our task to obtain the average over these rotated crystallites as seen by the neutrons. The pole densities of all individual crystal groups are correlatively connected with the [0001] pole density. For a specific group of crystallites with the direction normal to [hkl] aligned with the scattering vector, the density of [0001] poles is known once the angle ξ is given. Although not all the crystallites whose [0001] pole is in this orientation have their [hkl] plane normals coinciding with the scattering vector, the volume fraction of these crystallites must be proportional to the pole density of [0001].

The preceding arguments are only valid if there is no secondary texture present which may be readily ascertained from the inverse pole figures in Fig. 9 for four different directions of the specimen coordinate axes. Therefore the density of crystallites having their specified direction parallel to the scattering vector is linearly proportional to the pole density of [0001]:

\[ I_{hkl}(ξ, ω) ∝ I_{0001}(ξ, ω). \]  

The elastic compliance is one crystal property strongly dependent on crystal orientation. With an appropriate micromechanical approximation, the elastic anisotropy of the single crystal makes the stress exerted on a single crystallite vary when its orientation has changed; hence, as a result of differing stress and elastic compliance, the strain on each individual crystallite will be different. The averaged strain can be calculated by

\[ \bar{ε}_{hkl} = \frac{\int_0^{π/2} ε_{hkl}(ξ, ω) I_{0001}(ξ, ω) \, dξ}{\int_0^{π/2} I_{0001}(ξ, ω) \, dξ}. \]  

Evidently, there is no way to calculate the averaged strain without knowing the distribution of pole density along a specific integration path (the yellow line in Fig. 8a). The methods of March and Rietveld for ODF estimation are adopted here for the purpose of comparison with direct measurement.

The pole density of [0001] was extracted at ω equal to 90° and ξ in the range from 0 to 90°, and is plotted for the purpose of comparison with the March and Rietveld estimates in Fig. 10(a). The March coefficient \( R_d = 0.72 \), hence \( R = 1.93 \) for estimation of the ODF was obtained from Rietveld analysis of a neutron diffraction pattern recorded using the instrument HRPD at the ISIS Facility, Rutherford Appleton Laboratory, England. The parameters for the Rietveld function were obtained by manual adjustment until a best fit was obtained. It is worth pointing out that both March estimation of the ODF and direct measurement are normalized. Fig. 10(a) shows that the March estimation of the ODF matches the directly measured texture relatively well when ξ ranges from 45 to 90° owing to the nature of the texture being largely one-dimensional. However, they do not match very well when ξ is smaller than 45°. For the purposes of elastic constant integrals, the overall match is acceptable in this case if a full texture analysis is not available.

The three-dimensional pole-figure density plot for hot-pressed Ti₃AlC₂ in Fig. 8(b) shows three integration paths drawn on the sphere, which correspond to [10T0], [10T7] and [2025] poles at 90° and approximately 45 and 70° to the [0001] poles. The March coefficient \( R \) in this case is 0.51, which was
obtained from Rietveld refinements using diffraction patterns acquired from the diffractometer ENGIN-X at the ISIS Facility, Rutherford Appleton Laboratory, England. Fig. 10(b) shows that, in this case, the March estimation agrees reasonably well with the direct texture measurement. In general, the March estimate of pole density for $\omega = 90^\circ$ has greater curvature than the directly measured one, being slightly higher below $10^\circ$ of $\xi$, then lower from 10 to 45$^\circ$ of $\xi$, and higher again when $\xi$ is between 45 and 90$^\circ$. The situation is reversed when $\omega$ equals 75 and 45$^\circ$ i.e. the March curve underestimates the curvature of the actual pole densities. It is obvious that the integration of pole density along these paths at $\omega$ of 75 and 45$^\circ$ will be significantly different for the cases of direct measurement and March estimation. Depending upon the single-crystal constants, in the case of fibre textures such as these, the differences may be sufficient to affect the estimated physical (e.g. elastic) properties to a moderate extent. The differences will be more severe for two-dimensional textures or co-existing textures.

5. Conclusions

In this work, texture measurement using a neutron diffractometer equipped with an area detector has been streamlined and improved using MATLAB programs developed to preprocess the raw data for input to the quantitative texture analysis toolbox MTEX. Improvements include rigorous partitioning of large area detectors and proper integration along Debye–Scherrer rings.

Results are presented for three samples showing the utility of the routines and the MTEX suite. In two samples, $\alpha$-Al2O3 and hot-pressed Ti3AlC2, the c axes showed a strong preference for alignment with the material flow direction during processing, even though these flow directions are perpendicular in the two samples. A similar preference has been reported for textures in near-ideal hexagonal close-packed metals even though the deformation processes of these ceramics are completely different from metallic materials. The third sample demonstrates the near-texture-free nature of materials prepared using cold isostatic pressing and subsequent pressureless sintering.

The experimental textures were used to extract one-dimensional pole densities for particular $hkil$ such as would be required for the computation of diffraction elastic compliances (constants) in residual stress analysis or in the experimental determination of single-crystal elastic constants from neutron diffraction experiments. The directly measured densities and the March and Rietveld functions for one-dimensional texture estimation were compared and the agreement for the Rietveld function was poor. Although reasonable overall agreement was observed for the March function, the differences are great enough to cause minor to moderate errors in the computed elastic properties compared with full texture measurement. These discrepancies would be far greater for two-dimensional textures or samples containing co-existing textures. These results are also of interest in the computation of other physical property tensors for polycrystals.

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