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ORIENTATION DETERMINATION OF SINGLE-CRYSTAL BY COMPUTER GENERATED ALGORITHM

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A computer generated algorithm is written to determine the orientation of single crystals by directly taking the pole intensity data from the diffractometer by completely eliminating the use of Wulff net ensuring greater precision. The advantages of using a diffractometer method over conventional laue method for orientation determination is also compared and discussed.

Key words: Diffractometer method, Laue method, Wulff net, Stereographic projection, Pole figure.

Introduction

Much of our present-day knowledge in the field of physical metallurgy is based on the physical and mechanical properties that are measured on the individual single crystals of various orientations. Mechanical properties such as yield strength, elongation and ultimate tensile strength, as well as physical properties such as thermal expansion and diffusion co-efficients exhibit a wide range of anisotropy in a single crystal depending upon the direction in the single crystal along which they are measured.

In certain practical applications very precise crystal orientation is desired, for example in the operation of piezoelectric crystals used in acoustic detectors. Like wise, the choice of operative slip systems during plastic deformation in a single crystal also depends on the initial orientation of the crystal axis in relation to the applied stress axis. It is sometimes required to measure the precise orientation of undeformed and deformed single crystals [1], for example to determine the reorientation characteristics of tensile or compression axes of crystals to form a theory of plasticity, which is a field of continuing research.

Although various methods of orientation determination of crystals based on neutron diffraction, electron channelling patterns and electron backscattered patterns are available, however, mainly due to practical reasons, Laue or rotating crystal methods based on X-rays are generally used. In case of deformed crystals the lattice planes are severely distorted, the severity of which depends on the amounts of plastic deformation which in turn puts a limit to the precise determination of the crystal orientation by conventional techniques such as Laue or rotating crystal methods. These techniques are sensitive to the crystal quality and in case of even slightly deformed crystals, the X-ray spots are no longer sharp enough which make these techniques inadequate for precise determination of crystal orientation. Moreover, when the tensile or compression axis lies near the symmetry boundary of a stereographic triangle, it is not possible to determine precisely the overshooting of the crystal axis with these methods.

Another method of orientation determination is diffractometer method which is rarely used in such studies, although it allows to measure the reorientation of crystal with great precision compared to Laue or rotating crystal method. This method also allows precise determination of crystal orientation even though the crystal is heavily deformed.

In this paper diffractometer method is used to collect pole intensity of various planes in a single crystal. A computer algorithm was written to analyze the intensity data collected from the diffractometer for precise determination of crystal orientation. The orientation of same crystal was also determined by conventional Laue technique. The accuracy and limitations of two methods are discussed and compared. Although the results presented in this paper is based on undeformed single crystal only, however, the method used can be extended to determine the orientation of deformed crystals with ease and great precision, which is a subject of another publication.

Materials and Methods

Pure aluminium was directionally solidified under controlled cooling environment which resulted in large equiaxed grains [2]. Specimen of 3 x 3 x 3 cm^3 was sectioned within one grain which was checked metallographically from all six sides for any grain boundaries to ensure that it is a single crystal. Laue patterns from three orthogonal sections confirmed it. One side of the crystal was selected and its orientation i.e. crystallographic planes parallel to the crystal surface was determined by taking X-ray back reflection Laue pattern.

The same crystal surface as used in Laue method was mounted in a diffractometer and the orientation of the crystal was determined.

Orientation determination by Laue method. X-ray back reflection Laue pattern was taken by allowing white radiation to fall upon 50 x 25 x 10 mm^3 single crystal which was placed 13 mm away from the collimeter. An X- ray film to record the back reflection Laue pattern was placed behind the crystal at a distance of 23 mm. Figure 1a shows the Laue pattern obtained from the crystal, and Fig. 1b shows the selected

diffraction spots used in solving the pattern. Angular coordinates of these spots were measured by Greninger chart, poles of which were plotted on a stereographic projection which is shown in Fig. 2. The diffraction spots lying on any curve represent planes from one zone. With the help of a Wulff net great circles are drawn through various sets of diffraction spots in order to pinpoint low index planes which are the intersections of two or more great circles [3]. By trial and error, low index planes such as (100), (110) and (111) types were identified which are shown in Fig. 2. The projection centre of the stereographic projection in Fig. 2 represent the orientation of the crystal axis, the Miller indices of which were calculated by measuring angles between the crystal axis (centre of the stereographic projection) and low index planes i.e. (001), (011) and (111) which are marked on the stereographic projection with appropriate symbols.

The orientation of the crystal as determined by Laue method is found to be (5 3 3).

Orientation determination by diffractometer. The diffractometer technique utilized in this study involves the use of a rotating goniometer which is similar to that used in texture analysis [4]. Figure 3 shows the basic arrangement of the goniometer used in this work.

The sample is mounted at the geometrical centre of the goniometer and the detector is present to receive intensities from low index set of lattice planes e.g. (111) type reflections. By means of two independent rotations i.e. χ and ϕ the position of these low indexed planes on the standard stereographic projection are determined. Angle χ represents the tilt angle of the normal of the (111) crystal plane with respect to the crystal axis and angle ϕ represents the rotation angle of (111) plane against the crystal axis. The intensities of (111) poles were recorded and stored onto a magnetic tape. With the help of a computer program, the maximum intensities of (111) planes with respect to the tilt angles were identified. These tilt angles corresponding to maximum intensities were used as input data for the computer program developed to measure precise crystal orientation by completely eliminating measurement errors which may otherwise be incorporated using a Wulff net.

An algorithm, based on the coordinate system transformation from a non-orthogonal to an orthogonal system [1] is generalized to develop a computer program for orientation determination of FCC single crystal.

The generalized transformation is given by the following matrix equation:

$$[C_i] = [f_j]\,[a_{ij}] \quad ; \quad i,j = 1,2,3$$

where (a_{ij}) is a non-singular matrix and (C_i), (f_j) are row vectors of direction components in non-orthogonal and orthogonal systems, respectively.

The above matrix equation is solved for FCC single crystal, which has provided the following computer algorithm:

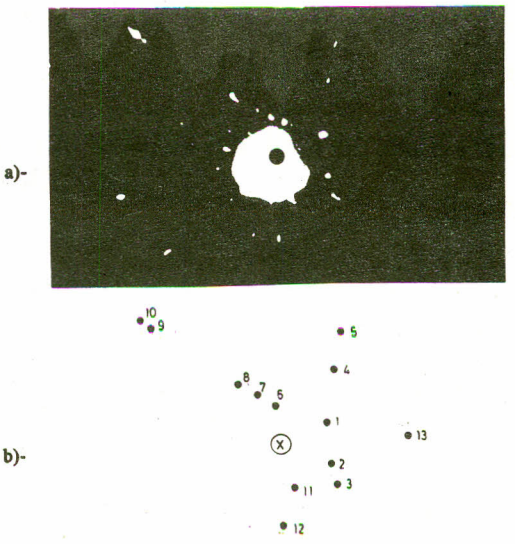

Fig. 1. Back reflection X-ray Laue pattern of single crystal (1a). Selected diffraction spots used to construct zones of low index planes (1b).

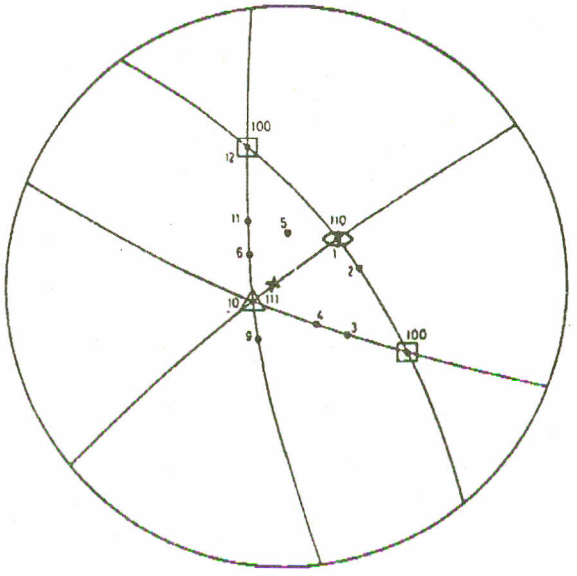

Fig. 2. Stereographic projection on which diffraction spots (from Fig. 1b) are plotted.

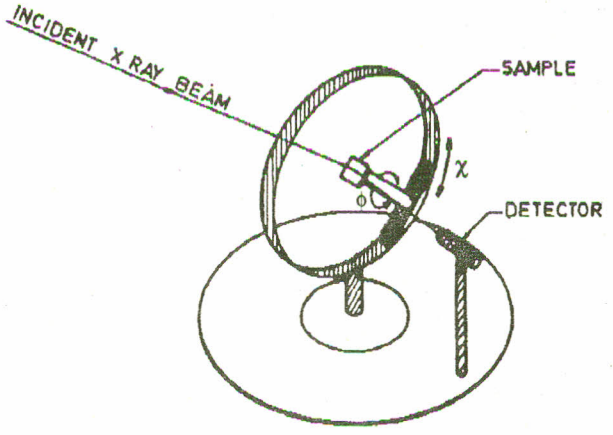

Fig. 3. Schematic diagram of a four-circle texture goniometer.

$f_1 = 1/D\ [(k_2l_3 - k_3l_2)c_1 + (k_3l_1 - k_1l_3)c_2 + (k_1l_2 - k_2l_1)c_3]$

$f_2 = 1/D\ [(h_3l_2 - h_2l_3)c_1 + (h_1l_3 - h_3l_1)c_2 + (h_2l_1 - h_1l_2)c_3]$

$f_3 = 1/D\ [(h_2k_3 - h_3k_2)c_1 + (h_3k_1 - h_1k_3)c_2 + (h_1k_2 - h_2k_1)c_3]$

where $C_1 = \cos \chi_1$, $C_2 = \cos \chi_2$, $C_3 = \cos \chi_3$; χ_1; χ_2, χ_3 are the tilt angles between the sample normal and normals to the projection planes $(h_1\ k_1\ l_1)$, $(h_2\ k_2\ l_2)$ and $(h_3\ k_3\ l_3)$ respectively. The determinant "D" thus formed is given as follows:

$$D = \begin{vmatrix} h_1 & k_1 & l_1 \\ h_2 & k_2 & l_2 \\ h_3 & k_3 & l_3 \end{vmatrix} \neq 0$$

If R is the radius of pole figure obtained by the diffractometer method, and r is the linear distance between the point on the pole figure representing maximum pole density of the respective plane and the centre of pole figure, then angle χ between sample normal and projection plane normal, can be evaluated by using the following relation:

$$\chi = 2 \arctan (r/R)$$

The types of projection planes can be indexed by adopting the sign convention as given by Cullity [3]. Thus by substituting the values of χ_1, χ_2, χ_3 and Miller indices of respective projection planes in the algorithm, the direction components f_1, f_2, f_3 of the orientation of FCC single crystal can be obtained directly by diffractometer method.

An example is presented by taking aluminium single crystal whose surface normal was determined by Laue method was mounted in the goniometer as described in earlier section. (111) pole figure is obtained as shown in Fig. 4. The tilt angles χ_1, χ_2, χ_3 were found to be 61, 19, 62 respectively and the respective projection planes were indexed as $(\bar{1}\bar{1}1)$, $(\bar{1}11)$, $(1\bar{1}1)$. Then the direction components f_1, f_2, f_3 of surface normal (by using the computer algorithm) were found to be (1 1 2).

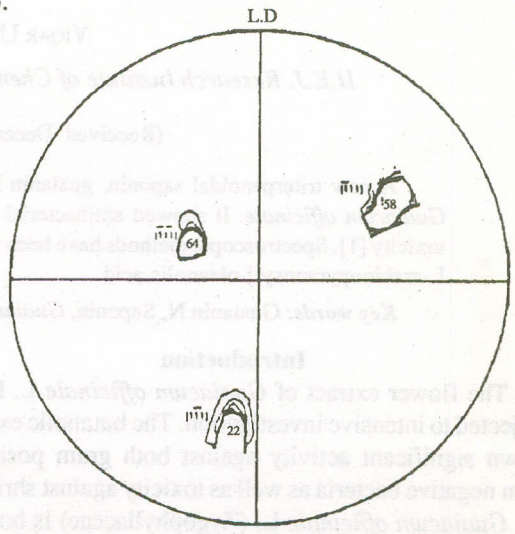

Fig. 4. (111) Pole figure of pure aluminium single crystal.

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