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CALCULATION OF TILT ANGLES FOR CRYSTAL SPECIMEN ORIENTATION ADJUSTMENT USING DOUBLE-TILT AND TILT-ROTATE HOLDERS

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Abstract—In this communication we wish to present a group of new equations which can be used to calculate the tilt angle for crystal specimen orientation adjustment in the transmission electron microscope. The experiments were concerned with double-tilt and tilt-rotate holders and the new equations deduced using matrix geometry. The specimen orientation adjustment using the tilt angles calculated by these equations is considered to be more convenient and less time-consuming than following the Kikuchi map method. Our method avoids the difficulties associated with orientation adjustment of severely strained and small grain size specimens using the Kikuchi map procedure. The algorithms for deducing the new equations, together with an experimental example using the equations, are described.

Index key words: Crystal orientation adjustment, double-tilt and tilt-rotate holders, tilt angles, matrix description.

INTRODUCTION

For analysis with aid of transmission electron microscopy of crystal defects and the orientation relationship between two phases, it is necessary to make adjustments to the specimen in order to obtain the corresponding diffraction patterns from different zones. The Kikuchi map is normally used in practice as a type of 'road map' to make adjustments to the specimen from one orientation to another (Edington, 1975).

However, there are a number of problems when using the Kikuchi map method; first, the specimen area of interest is often observed to be too thin to produce Kikuchi line pairs. Second, for a severely strained specimen the visible Kikuchi line pairs are poorly defined, and third, in case of a small grained specimen, tilting of the specimen may cause the disappearance of the corresponding diffraction pattern, due to specimen shift.

In these situations it is difficult, or may not be possible to adjust the crystal orientation to follow the Kikuchi map. A computer program was developed by Chou (1987), to calculate the tilt angles for adjusting the specimen from one zone axis to another. However, it was found in practice that the method was both complex and time consuming when making the calculation. The aim of this communication is to present one group of new equations which allow the rapid calculation of the tilt angles and orientation adjustment of the specimen to be achieved.

MATRIX DESCRIPTION OF THE ROTATION AROUND THE TILT AXES

The relationship between a fixed reference orthogonal coordinate system and the tilt axes of double-tilt and tilt-rotate holders is shown in Fig. 1. The x-axis is coincident with the longitudinal axis (T_1) of the specimen holder, while the z-axis lies along the optical axis of the microscope. The second tilt axis (T_2) of the double-tilt holder (Fig. 1(a)) and the rotation axis (T'_2) of the tilt-rotate holder (Fig. 1(b)) lie in the yz-plane, the exact position depending on the first tilt angle (α_0) of the T_1-axis. For the double-tilt holder, when the specimen is tilted through an angle β around the T_2-axis (from β_0 to $\beta_0+\beta$), followed

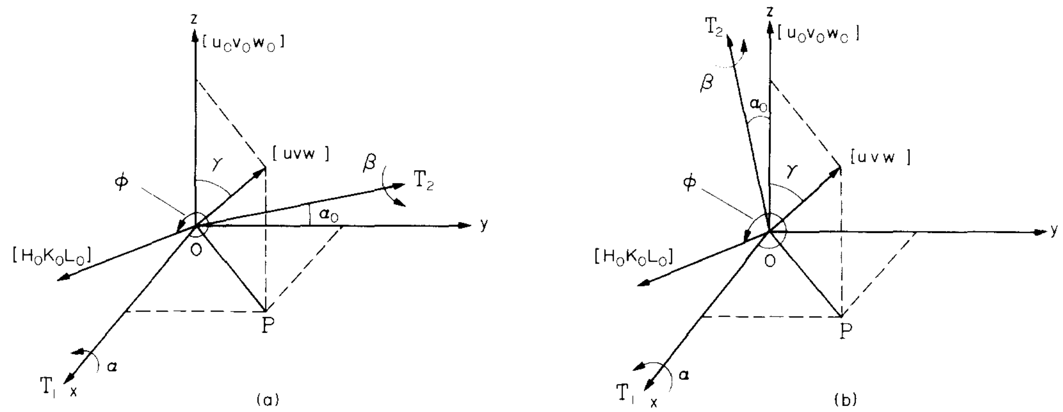

Fig. 1. Geometry relationships of the crystal specimen adjustment using (a) double-tilt holder and (b) tilt-rotate holder.

by a tilt α around the T_1-axis (from α_0 to $\alpha_0+\alpha$), the new position of a vector $\mathbf{g}$ is

$$\mathbf{g}'=T_1T_2\mathbf{g} \tag{1}$$

T_1 and T_2 are the transformation matrices related to rotations around the T_1- and T_2-axes, respectively, being:

$$T_1=\begin{pmatrix}1 & 0 & 0\\ 0 & \cos\alpha & -\sin\alpha\\ 0 & \sin\alpha & \cos\alpha\end{pmatrix} \tag{2}$$

and

$$T_2=\begin{pmatrix}\cos\beta & -\sin\beta\sin\alpha_0 & \sin\beta\cos\alpha_0\\ \sin\beta\sin\alpha_0 & \cos^2\alpha_0+\sin^2\alpha_0\cos\beta & \sin\alpha_0\cos\alpha_0(1-\cos\beta)\\ -\sin\beta\cos\alpha_0 & \sin\alpha_0\cos\alpha_0(1-\cos\beta) & \sin^2\alpha_0+\cos^2\alpha_0\cos\beta\end{pmatrix}. \tag{3}$$

Similarly, for the tilt-rotate holder, when the specimen is rotated through a angle β' around the T'_2-axis (from β'_0 to $\beta'_0+\beta'$), followed by a tilt α around the T_1-axis (from α_0 to $\alpha_0+\alpha$), the new position of a vector $\mathbf{g}$ is

$$\mathbf{g}'=T_1T'_2\mathbf{g} \tag{4}$$

T_1 and T'_2 are the transformation matrices related to rotations around the T_1- and T'_2-axes. The matrix T_1 is the same as that of the double-tilt holder, and

$$T'_2=\begin{pmatrix}\cos\beta & -\sin\beta'\cos\alpha_0 & -\sin\beta'\sin\alpha_0\\ \sin\beta'\cos\alpha_0 & \cos^2\alpha_0+\cos\beta'+\sin^2\alpha_0 & \cos\alpha_0\sin\alpha_0(\cos\beta'-1)\\ \sin\beta'\sin\alpha_0 & \sin\alpha_0\cos\alpha_0(\cos\beta'-1) & \sin^2\alpha_0\cos\beta'+\cos^2\alpha_0\end{pmatrix}. \tag{5}$$

CALCULATION OF TILT ANGLES FOR THE SPECIMEN ORIENTATION ADJUSTMENT

At the original position where the tilt angles about T_1-axis is α_0, one recognizable zone axis $[u_0\ v_0\ w_0]$ has been aligned to the incident beam direction, with the diffraction pattern of this zone axis being shown on the fluorescent screen. If the diffraction pattern of another zone axis $[u\ v\ w]$ is obtained from the original position, the angle between $[u\ v\ w]$ and $[u_0\ v_0\ w_0]$ is γ. The specimen should be tilted through the angle γ around the normal to the common crystallographic plane $(H_0\ K_0\ L_0)$ to the both zones (see Fig. 1). H_0, K_0 and L_0 can be determined from the following relations:

$$\begin{aligned}H_0&=vw_0-v_0w\\ K_0&=wu_0-w_0u\\ L_0&=uv_0-u_0v.\end{aligned} \tag{6}$$

The normal direction to the plane $(H_0\ K_0\ L_0)$ is the direction of the line drawn from the central spot to the spot $(H_0\, K_0\, L_0)$ in the diffraction pattern of the zone axis $[u_0\, v_0\, w_0]$. On the fluorescent screen, the movement trace of the Kikuchi pole during tilting the T_1-axis is normal to the projection of the T_1-axis. Thus, for a certain camera length, the projection position of the direction and sense of the x-axis can be determined. It follows that the angle ϕ between the normal to the plane $(H_0\, K_0\, L_0)$ and the x-axis can be measured on the fluorescent screen.

In order to obtain the diffraction pattern of the zone axis $[u\, v\, w]$, the vector $[u\, v\, w]$ (written as U) should be tilted to being coincident with the z-axis. In Fig. 1, the projection of the vector U is op, which is perpendicular to the normal to the plane $(H_0\, K_0\, L_0)$, then the angle $\angle x\,\mathrm{op} = \phi - 270°$. By assuming that the magnitude of the vector $|U| = 1$, in the fixed reference orthogonal coordinate system, the vector can be written as

$$U = \begin{bmatrix} \sin\gamma \cos(\phi - 270°) \\ \sin\gamma \sin(\phi - 270°) \\ \cos\gamma \end{bmatrix} = \begin{bmatrix} -\sin\gamma \sin\phi \\ \sin\gamma \cos\phi \\ \cos\gamma \end{bmatrix},$$

When the vector U is tilted to being coincident with z-axis, the new position of this is

$$U' = \begin{bmatrix} 0 \\ 0 \\ 1 \end{bmatrix}.$$

For the double-tilt holder, according to the Equation (1), the relationship between U and U' is given by:

$$U' = T_1(\alpha) T_2(\beta) U$$

then

$$\begin{bmatrix} 0 \\ 0 \\ 1 \end{bmatrix} = T_1(\alpha) T_2(\beta) \begin{bmatrix} -\sin\gamma \sin\phi \\ \sin\gamma \cos\phi \\ \cos\gamma \end{bmatrix}. \qquad (7)$$

Similarly, for the tilt-rotate holder, according to the Equation (4)

$$\begin{bmatrix} 0 \\ 0 \\ 1 \end{bmatrix} = T_1(\alpha) T_2'(\beta') \begin{bmatrix} -\sin\gamma \sin\phi \\ \sin\gamma \cos\phi \\ \cos\gamma \end{bmatrix}. \qquad (8)$$

For the double-tilt holder, using Equations (2) and (3) in Equation (7) leads to the equations:

$$\alpha = \sin^{-1}(\sin\gamma \cos\phi \cos\alpha_0 + \sin\alpha_0 \cos\gamma) - \alpha_0 \qquad (9a)$$

$$\beta = tg^{-1}\left(\frac{tg\gamma \sin\phi}{\cos\alpha_0 - \sin\alpha_0 tg\gamma \cos\phi}\right). \qquad (9b)$$

Similarly, for the tilt-rotate holder, using Equations (2) and (5) in Equation (8) leads to the equations:

$$\alpha = (-1)^n \cos^{-1} (\cos\alpha_0 \cos\gamma - \sin\alpha_0 \sin\gamma \cos\phi) - \alpha_0 \qquad (10a)$$

$$\beta' = tg^{-1}\left(\frac{\underline{-}\sin\phi}{ctg\,\gamma \sin\alpha_0 + \cos\alpha_0 \cos\phi}\right) \qquad (10b)$$

where $n = 0$ if $0° < \phi < 180°$ and $\beta' < 0°$ or $180° < \phi < 360°$ and $\beta' > 0°$; $n = 1$ if $0° < \phi < 180°$ and $\beta' > 0°$ or $180° < \phi < 360°$ and $\beta' < 0°$.

Having obtained the diffraction pattern of the zone axis $[u_0\, v_0\, w_0]$, in order to obtain the diffraction pattern of another zone axis, the common crystallographic plane $(H_0\, K_0\, L_0)$ to both zones can be determined by the Equation (6). Moreover, the angle ϕ between the normal to the plane $(H_0\, K_0\, L_0)$ and x-axis, can be directly measured on the fluorescent screen using a beam tilt technique described by Tambuyser (1983). Thus, after having known the original angle α_0 of T_1-axis from the meter reading of the specimen holder and the angle γ between both the zone axes, for the double-tilt or tilt-rotate holders, the tilt angles for obtaining the diffraction pattern of any zone axis can be rapidly calculated using Equation (9) or Equation (10).

Example

With the aid of a Phillips CM12/STEM electron microscope, a small hexagonal β-SiC whisker (which was found to consist of hcp crystal layers) in a SiC_w/6061Al composite (see Fig. 2) was used to test the orientation adjustment by applying Equation (9) in the double-tilt holder. The original position was the zone axis [0 0 0 1] being coincident with the beam direction, where the number of the tilt angles about the two axes were $\alpha_0 = 9°$, $\beta_0 = 12.4°$. Since the diameter of the hexagonal whisker was about 0.5 μm, the microdiffraction was obtained using a small beam spot size (150 nm), and the diffraction patterns of the other eight zone axes

Fig. 2. Transmission electron micrograph of a hexagon β-SiC whisker (hcp) in an Al–SiC composite. (× 57,000)

obtained by adjusting the specimen using the tilt angles calculated by Equation (9). Table 1 shows the good agreement between the experimental and calculated tilt angles. The maximum error being $\leqslant 2.0^\circ$.

Similarly, for the tilt-rotate holder, an aluminium alloy specimen was chosen to test the orientation adjustment using Equation (10). The comparison between the calculated and experimental results are shown in Table 2. For tilt axis, the maximum error was $\leq 2.0^\circ$; for the rotate axis, the maximum error was $\leq 4.0^\circ$.

DISCUSSION

The close agreement between the experimental and calculated tilt angles shown in Tables 1 and 2 allows the rapid and accurate adjustment of the crystal specimen orientation without reference to the Kikuchi pattern. The main sources of the errors arise from the following.

(1) The determination of the projection position of the x-axis. The projection position of the x-axis can be determined by observing the movement trace of the Kikuchi pole when only tilting the T_1-axis as mentioned above. If the movement trace of the cross point of the higher order Kikuchi lines in a convergent beam diffraction pattern is observed, it will be more accurate to determine the projection of the x-axis. It should be noted

Table 1. Experimental and calculated tilt angles for a β-SiC whisker (hcp) orientation adjustment using double-tilt holder

				1st tilt angle		2nd tilt angle	
$u\ v\ t\ w$	γ	$H_0\ K_0\ I_0\ L_0$	ϕ	α_{exp}	α_{cal}	β_{exp}	β_{cal}
$0\ 0\ 0\ 1$	0			9		12.4	
$0\ \bar{1}\ 1\ 3$	19.47	$\bar{2}\ 1\ 1\ 0$	−10	28.0	28.14	10.3	8.64
$0\ 2\ \bar{2}\ 3$	35.26	$2\ \bar{1}\ \bar{1}\ 0$	170	−25.5	−25.71	20.7	18.79
$\bar{1}\ 1\ 0\ 3$	19.47	$1\ 1\ \bar{2}\ 0$	−130	−4.0	−3.68	−1.5	−2.42
$\bar{2}\ 0\ 2\ 3$	35.26	$\bar{1}\ 2\ \bar{1}\ 0$	−70	17.0	18.83	−22.9	−22.57
$0\ \bar{2}\ 2\ 3$	35.26	$\bar{2}\ 1\ 1\ 0$	−10	43.0	43.57	2.8	4.45
$\bar{2}\ 2\ 0\ 3$	35.26	$1\ 1\ \bar{2}\ 0$	−130	−15.5	−13.81	−13.6	−14.69
$1\ \bar{2}\ 1\ 6$	17.02	$\bar{1}\ 0\ 1\ 0$	20	24.0	24.91	17.5	18.74
$\bar{1}\ \bar{1}\ 2\ 3$	31.48	$\bar{1}\ 1\ 0\ 0$	−40	30.2	31.91	−9.3	−10.89

Table 2. Experimental and calculated tilt angles for an aluminium alloy orientation adjustment using tilt-rotate holder

				1st tilt angle		Rotation angle	
$u\ v\ w$	γ	$H_0\ K_0\ L_0$	ϕ	α_{exp}	α_{cal}	β'_{exp}	β'_{cal}
$0\ 1\ 1$	0			−7.5		233.8	
$\bar{1}\ 1\ 1$	35.26	$0\ 2\ \bar{2}$	3	27.6	27.77	232.6	230.08
$\bar{1}\ 1\ 2$	30	$\bar{1}\ 1\ \bar{1}$	38.26	23.6	24.51	187.2	185.54
$\bar{1}\ 2\ 1$	30	$1\ 1\ \bar{1}$	−32.26	25.0	23.96	278.5	274.88
$0\ 1\ 0$	45	$2\ 0\ 0$	−87	−43.3	−45.10	148.5	148.30
$0\ 0\ 1$	45	$\bar{2}\ 0\ 0$	93	−44.5	−45.87	316.5	313.45
$1\ 1\ 2$	30	$\bar{1}\ \bar{1}\ 1$	147.74	−37.5	−36.54	257.3	260.43
$1\ 1\ 1$	35.26	$0\ \bar{2}\ 2$	183	−43.2	−42.75	229.5	231.25
$1\ 2\ 1$	30	$1\ \bar{1}\ 1$	218.26	−37.5	−36.16	205.5	202.15

projection position of the x-axis changes with the camera length.

(2) If the microscope is equipped with a device for the direct measurement of the diffraction spots positions (Tambuyser, 1983), it will be convenient and accurate to measure the angle ϕ.

(3) The measurement of the tilt angles; the mechanical inaccuracy of the specimen holder is the main cause of error. From the examples given above, the error of the tilt axis is smaller than that of the rotate axis. From our experience, the slower the specimen is rotated the closer the accuracy between the experimental and calculated angles.

(4) The accuracy in obtaining exact zone axis diffraction. After tilting the specimen using the tilt angles calculated by the equations are presented in this paper, only a small adjustment is needed to obtain the exact zone axis diffraction. It should be noted that the first zone axis $[u_0\, v_0\, w_0]$ should be carefully aligned to the incident beam direction.

The equations presented in this paper provide a rapid method for crystal specimen orientation adjustment. All these equations are simple, and the tilt angles for orientation adjustment can be calculated using a conventional calculator. For a severely strained specimen and a small grain-size crystal specimen (such as a small whisker mentioned above), it is very convenient to adjust the orientation using the tilt angles calculated by Equations (9) or (10), instead of the difficult adjustment following the Kikuchi map, since the specimen can be tilted under image mode and the specimen shift can be adjusted during tilting.

In order to determine an unknown crystal structure, the Niggli reduced bases are usually constructed by the measurement of a couple of electron diffraction patterns. However, it is usual to encounter errors following the poor results for Bravis lattice determination by electron diffraction. The equations described here can be rapidly used to examine whether the suggested unit cell parameters are correct. It is also valuable in the examination of unknown structure determination.

CONCLUSIONS

(1) One group of new equations to calculate the tilt angles for crystal orientation adjustment using double-tilt and tilt-rotate holders are deduced.

(2) The method presented in this paper overcomes the difficulty for orientation adjustment of strained and small grain-size crystal specimens following the Kikuchi map.

(3) This method can be used if the suggested unit cell parameters are correct when determining an unknown structure.

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