Letters

On the measurement of the misorientation across low-angle boundaries

During the course of electron microscopical investigations of, for instance, crept or recovered microstructures, it may become necessary, or desirable, to measure the misorientation across low-angle grain boundaries. The simplest possible estimate of this parameter is found by measuring the angular displacement between identical reciprocal lattice vectors in the plane of a diffraction pattern taken across a boundary region. However, this type of measurement can be considerably in error. For a series of such measurements, and in a specimen where the true rotation axes of the low-angle boundaries are at random angles to the beam direction, 33% of the measured angles will underestimate the true misorientation by more than 50%.

The presence of Kikuchi lines in diffraction patterns allows a far more accurate assessment of such misorientations. However, the post-experimental analysis of Kikuchi patterns taken across grain boundaries usually involves rather complex procedures [1] which, while necessary for the analysis of high-angle boundaries, can be avoided in the case of low-angle boundaries. It is the intention of the present communication to illustrate that by manipulating diffraction conditions, it is possible to reduce the procedure for post-experimental analysis of misorientation angles to a simple level.

The basis of the technique is that the misorientation between any two subgrains (grains) can be described in terms of a rotation (θ_2) about a plane normal, followed by a rotation (θ_1) about an axis contained within the same plane. For a low-angle grain boundary, these rotations are small (in general less than 5°) with the result that adjacent subgrains exhibit identical spot patterns but displaced Kikuchi lines. Thus, if a specimen is tilted until the deviation parameter (s) for a pair of identically indexed reflections ($g_{hkl_{\mathrm{A}}}$, $g_{hkl_{\mathrm{B}}}$) is the same in two neighbouring subgrains (A and B), the true rotation between these subgrains can be found from a measurement of just two angular components. It should be noted that the most convenient way of setting up the diffraction conditions referred to above is in dark field; both subgrains are brought into bright contrast simultaneously.

The first component of the rotation, θ_1, is the angular displacement between the reciprocal lattice vectors $g_{hkl_{\mathrm{A}}}$ and $g_{hkl_{\mathrm{B}}}$ (i.e. measured about $g_{hkl_{\mathrm{A}}} \times g_{hkl_{\mathrm{B}}}$), while the second component, θ_2, which is measured along the traces of $(h\,k\,l)_{\mathrm{A}}$ and $(h\,k\,l)_{\mathrm{B}}$, is the angular separation of zone axes which must be indexed identically in the Kikuchi line patterns of each subgrain; θ_2 is a rotation about an axis normal to that for the rotation θ_1.

The true angle of misorientation is found as arc cos $(\cos\theta_1 \cos\theta_2)$ or $(\theta_1^2 + \theta_2^2)^{1/2}$ (since θ_1 and θ_2 are small). The axis of rotation can be obtained after indexing $g_{hkl_{\mathrm{A}}} \times g_{hkl_{\mathrm{B}}}$. This can be accomplished using the simple method developed by Helfmeier and Feller-Kniemeier [2].

The above technique has one further advantage, which arises since $g_{hkl_{\mathrm{A}}}$ and $g_{hkl_{\mathrm{B}}}$ are both strongly excited and only slightly misoriented; a system of purely rotational Moiré fringes is superimposed on the image of the low-angle boundary. Thus the component of misorientation, θ_1, which is usually hard to measure accurately for a low-angle boundary, is magnified in the Moiré pattern of the bright or dark-field image. The spacing of the Moiré fringes (D) and the angle of rotation (θ_1) are related by $D = (g_{hkl} \sin\theta_1)^{-1}$.

The above technique is illustrated by the example given in Fig. 1. Fig. 1a and b are diffraction patterns obtained (from grains A and B, respectively) from either side of the low-angle grain boundary shown in Fig. 1c. the material is aluminium. In both grains, the diffraction pattern is close to a reciprocal lattice section normal to $[1\,1\,\bar{2}]$. In (a) and (b), $g_{hkl_{\mathrm{A,B}}}$ are $1\,1\,1_{\mathrm{A}}$ and $1\,1\,1_{\mathrm{B}}$, respectively. The position of zone axis $g_{111_{\mathrm{A}}} \times g_{111_{\mathrm{B}}}$ is indicated in each pattern at point C. A point $\mathrm{D_A}$ has been arbitrarily selected and indicated along the trace of $(1\,1\,1)_{\mathrm{A}}$ (dashed line in Fig. 1a). The identical point in the Kikuchi pattern of grain B is indicated at $\mathrm{D_B}$ along the trace of $(1\,1\,1)_{\mathrm{B}}$ (dashed line in Fig. 1b). It can be seen that these identical zone axes are mutually

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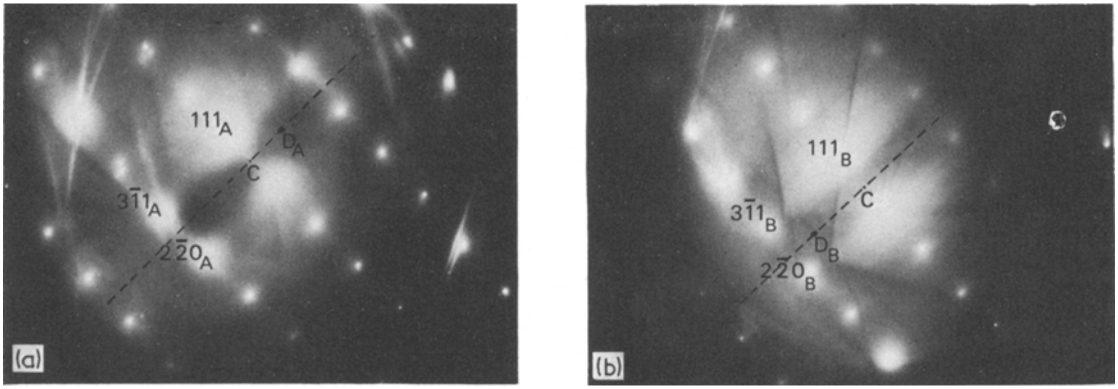

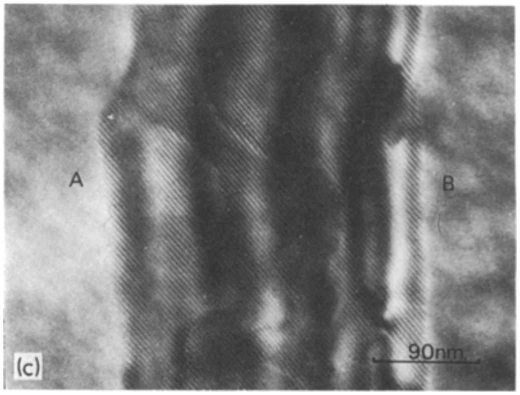

Figure 1 (a) and (b) The diffraction patterns from grains A and B, respectively, seen in (c). Both patterns are close to that of a reciprocal lattice section normal to [1 1 $\bar{2}$]. (*c*) A low-angle grain boundary in aluminium. In this micrograph, the finely spaced lines showing strong contrast are rotational Moiré fringes.

displaced; the angular misorientation between them (from D_A to C and C to D_B) represents θ_2. This was measured as 1.89 ± 0.03°. Measurement of the spacing of the Moiré fringes seen in Fig. 1c gave θ_2 (about $g_{111_A} \times g_{111_B}$) as 3.9 ± 0.1°. Hence the angle of misorientation between subgrains A and B is 4.3 ± 0.1°. The axis of rotation was found after indexing $g_{111_A} \times g_{111_B}$ in Fig. 1a (as $[\overline{21}\ \overline{20}\ 41]$). The rotation of the diffraction pattern in Fig. 1a into full coincidence with that shown in Fig. 1b is accomplished by a rotation of 4.3 ± 0.1° about $[\overline{130}\ \overline{127}\ 100]$.

The errors in the two components of the misorientation lead to an uncertainty of about ±1° (in the present case) in determining the true axis of misorientation. A similar error arises from the uncertainty in indexing $g_{111_A} \times g_{111_B}$, since for an exact solution, s_{111_A} and s_{111_B} have to be identical (but not necessarily at $s = 0$).

It should be noted that the errors in the analysis lead to an accuracy of ±0.1° for the angle of misorientation; the error in determining the axis of misorientation increases with smaller angles of misorientation (since the smallest detectable difference in s between g_{hkl_A} and g_{hkl_B} is not a function of the angle θ_1).

References

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