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DOI: 10.1017/S1431927606061678
Microsc Microanal 12(Supp 2), 2006

A General Approach to Unit-cell Determination in Electron Diffraction Experiments

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Determination of the unit-cell for unknown crystalline phase is a basic requirement for materials characterization and the first step of *ab initio* structure determination. Electron diffraction technique as a counterpart of X-ray and neutron diffraction techniques has been extensively used in material characterization and structure determination.

Due to the feature of electron diffraction technique, it is natural to determine the unit-cell for unknown crystalline phases by the method of reciprocal lattice reconstruction from an electron diffraction tilt series. A simple reconstruction method was shown in the book by Vanishtein [1]: a two-dimensional (2D) lattice was constructed from an electron diffraction tilt series. This method is troublesome in the application to crystalline phases belonging to monoclinic or triclinic systems. A general 3D reciprocal lattice re-construction method was discussed by Fraundorf [2] and a program with a visual interface for this purpose was recently developed by Zou *et al.* [3].

Kuo [4] applied the concept of the Niggli cell and cell reduction technique on the unit-cell determination in electron diffraction experiments. Similar techniques have been widely used in X-ray crystallography [e.g., 5-7]. Basically there are two steps in the determination of the unit-cell: the determination of a reduced direct primitive cell and the transformation to a conventional cell.

In the present paper, an approach for the determination of the unit-cell of an unknown crystalline phase in electron diffraction experiments is described. The approach for unit-cell determination by the cell reduction method is shown in Figure 1.

Primitive cell: Assuming that three consecutively tilted electron diffraction patterns have been obtained, two series of reduced primitive reciprocal cells can be calculated from these electron diffraction patterns by series of tilted angles near to the experimental measure values. The reciprocal cells from the two series with closest lattice parameters can be found. The corresponding tilt angles were then used for the calculation of the direct primitive cells and then the averaged one.

Conventional cell: The recognition and interpretation of the reduced form are difficult due to the effects of the errors in the cell parameters, rounding errors in calculation, and the equality or inequality conditions in calculation. Several methods have been proposed to solve the problem [e.g., 8-10]. The practical procedure proposed by Clegg [8], is used in the present approach for the determination of the conventional cell in electron diffraction experiments.

Indexing of reflections and Least-square refinement: The general procedure [11] for indexing electron diffraction patterns with known lattice parameters is to find the indices of the two basic reflections ($\mathbf{r}_1$, $\mathbf{r}_2$) by matching their vector lengths (r_1, r_2) and the angle ω between them (or r_1, r_2, $r_3 = |\mathbf{r}_1\text{-}\mathbf{r}_2$) to experimental results. Basic reflections in each pattern are indexed using the conventional

cell under a certain tolerated value. Least-square refinement was carried out in reciprocal space and then the direct lattice parameters are calculated.

The procedure described above shows that the optimum lattice parameters of the unit-cell depend on the lengths of the basic reflections in each diffraction pattern. A set of Java programs has been developed by the author and examples on the usage of the programs are given as demonstration [12].

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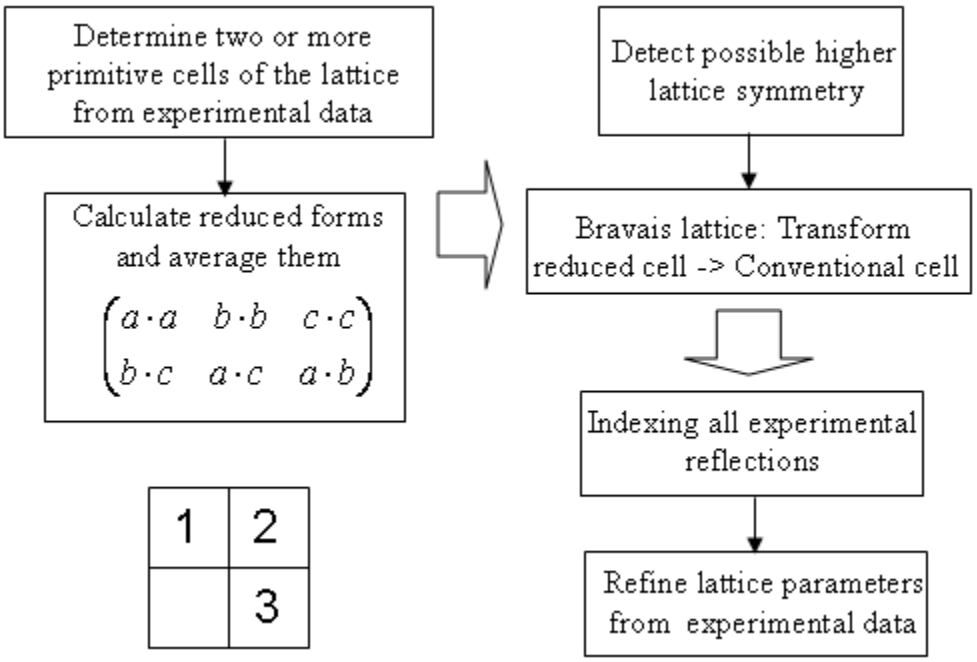

Fig. 1. Flow diagram showing the unit-cell determination by the cell reduction method. In step 1, the determination of the primitive cell. In step 2, the determination of the conventional cell. In step 3, the indexing of diffraction patterns and the refinements of the lattice parameters.

https://doi.org/10.1017/S1431927606061678 Published online by Cambridge University Press