

Two patterns indexed as  $M_2X$ , were searched with  $M=Cr, Ni, Fe, Mo, Nb$ . These yielded hits,  $Nb_2C$  (the same two database entries), for each pattern in Tables 10 and 11. None of the other four metals, singly or in combination, yielded hits for these patterns. In the full practice of this invention, the element Nb would have been determined experimentally, and other combinations of possible metals would be unnecessary to search. In this example three other phases were found to match one but not both patterns, demonstrating the advantage of obtaining alternate orientation patterns for confirmation.

When Ni was used as the metal in the search, three database entries for  $Ni(OH)_2$  were found for each pattern (Tables 12, 13). Though not mentioned by the authors of Reference 14, this ought to be considered as an additional possible solution. This  $MX_2$  formula was not among those considered by the authors, however. A third low index zone axis pattern was indexed by the authors as an  $M_6C$  phase. The best matches from our database were  $Cr_3Nb_3C$  (Table 14), and  $Fe_3Nb_3C$  and  $Fe_3Nb_3$  (Table 15). All possible combinations of one or two metals, M, were searched.

#### Example 8

A  $ZrH_2$  pattern was indexed in Reference 15. Taken as an unknown containing Zr, we obtained two correct hits from the database, both  $ZrH_2$ , which are listed in Table 16.

#### Example 9

Table 17 contains results of simulated ZONES searches for 10 phases, representing all crystal systems, in which input of the maximum  $d_1$ /maximum  $d_2$  and the minimum  $d_1$ /minimum  $d_2$  zones for the input phase in the database are, respectively, matched against the entire database. Although all input phases were minerals, this fact was not used in the search. The total number of "Hits" and the number of "Correct" hits are listed in each case (Hits/Corr.). Typical common phases like these have multiple database entries from different experimental sources, each having its own CODE. The large percentage of virtually identical correct hits in each case demonstrates that this search/match scheme is very robust. Many incorrect solutions were closely related solid solutions or isomorphs. Examples of these are: for halite— $Na(Cl, CN)$ ; for pyrite— $FeS$  and  $Fe_2S_3$ ; for calcite— $CaC_2B_2$ .

By way of comparison to the patented procedure mentioned above (Reference 9), the reduced unit cell of each phase (represented by its CODE) in Table 17 was searched against the database, and the total number of hits and correct hits (Hits/Corr. Red. Cell) were entered in the last column of Table 17. Generally similar results were obtained by both search methods, except in crystallographically less well defined cases (albite, kaolinite, and to a lesser extent quartz) where our approach produced more hits. Structurally similar phases with different unit cells (especially crystal symmetry) will not always be found in the reduced unit cell search of the NIST procedure. Since d-values are more closely related to structure than unit cells and are often nearly equal among polymorphs and isomorphs, ZONES does tend to produce more hits. Also, as discussed above, reduced unit cells can be difficult to obtain by electron diffraction.

The present invention allows the use of the reduced unit cell parameters instead of the more common procedure of using the full symmetry unit cell, and results in calculating d-spacings produced by double diffraction in addition to those produced by normal diffraction. Also, no d-spacings possible to be calculated from an experimental electron

diffraction pattern are missing from among the calculated d-spacings in the database, and no extra d-spacings are present in the database that are not possible to be observed in electron diffraction.

Because of the above, the candidate material or materials found in the output table tblZones2 described above, by using the methods and procedures of this invention, are very likely to be the actual material or materials in the sample from which the electron diffraction pattern was obtained. Additionally, the known materials which are in the database but which are not in the final list of candidate materials are very unlikely to be the actual material or materials in the sample from which the electron diffraction pattern was obtained.

The invention also provides a reduction in the time required to interpret a single crystal electron diffraction pattern of spots from that required by other methods and procedures.

This invention also allows transmission electron microscopists who are not trained crystallographers to identify small single crystal samples of known materials in the database, without resorting to other techniques or references, and in a time period which is comparable to that required by experts skilled in crystallography who employ methods and procedures of this invention or other methods and procedures. While skills in crystallography and database management are required to construct a useful embodiment of this invention, no such skills are required to use such a useful embodiment to identify an unknown single crystal, given the experimental data.

Also, the invention allows individuals who are not skilled in the art of transmission electron microscopy to interpret single crystal electron diffraction patterns obtained by other individuals who are skilled in the art of transmission electron microscopy. As shown in Examples 4–8, in which experimental data obtained by others and reported in the open literature was successfully interpreted by the inventor, who is not skilled in the art of transmission electron microscopy, using the methods and procedures of this invention, without resorting to other references or other methods and procedures.

As a result of the present invention the field of electron diffraction is made more useful to individuals who are not skilled in all three of the following arts: transmission electron microscopy, crystallography, and computer programming, and to the very few who are skilled in all three arts.

Illustrative examples of suitable method for the identification of polycrystalline materials from electron diffraction patterns, relevant crystallographic theory and application, and the techniques of electron diffraction can be found in the following literature and the description thereof are herein incorporated by reference:

1. Andrews, K. W., Dyson, D. J., and Keown, S. R., Interpretation of Electron Diffraction Patterns, Second Edition, Adam Hilger, London, 1971, p. 15.
2. Lally, J. S. and Lee, R. J., Computer Indexing of Electron Diffraction Patterns Including the Effect of Lattice Symmetry, Proc. Electron Microsc. Soc. Am., 1977, pp. 22–23.
3. Lally, J. S. and Lee, R. J., Computer Indexing of Electron Diffraction Patterns Including the Effect of Lattice Symmetry, Electron Microsc. X-ray Appl. Environ. Occup. Health Anal., Second Symposium, Phillip A. Russell, Alan E. Hutchings, eds., Ann Arbor Sci., Ann Arbor, Mich., 1978, pp. 169–174.