

define lattice structures that are in a derivative subcell or supercell relationship if step 130 finds a matrix H such that either i) the matrix H has integer elements, a determinant HDET > 1 and will transform cell Z to cell Y; or ii) its inverse matrix H' has integer elements, a determinant HDET > 1, and will transform cell Y to cell Z. A typical range of matrix elements in such a case includes the integer and non-integer reciprocals thereof in the range ± 6 , i.e., -6, -5, -4, -3, -2, -1, -1/8, -1/4, -1/5, 0, 1/5, 1/4, 1/2, 1, 2, 3, 4, 5, 6.

c) Unknown cell Z and matching known cell Y define lattice structures that are in a composite relationship if step 130 finds a matrix H such that i) the matrix H has one or more fractional elements and will transform cell Z to cell Y; and ii) its matrix inverse H' also has one or more fractional elements and will transform cell Y to cell Z. A typical range of matrix elements in such a case includes the integers and non-integer reciprocals thereof in the range ± 6 , i.e., -6, -5, -4, -3, -2, -1, -1/2, -1/3, -1/4, -1/5, 0, 1/5, 1/4, 1/3, 1/2, 1, 2, 3, 4, 5 and 6.

It will be appreciated that in most phase characterization studies, only the first two relationships described above are of interest. Routinely checking for sub/supercell relationships is particularly useful in that it permits identification despite certain categories of experimental errors. The known lattices cells Z determined to have either the same or a sub- or super-cell relationship to unknown cell Y are thus advantageously stored as a first data set 1.

In the next step 150, database 96 is searched to determine all known compounds with the same elemental composition as the unknown material. The results of the search are saved in a second data set 2. With the NIST CRYSTAL DATA database, this is a straightforward operation, since the database contains an empirical formula with the elements in alphabetical order for each material. Preferably, the element search is set to find all database compounds that have precisely the same elements as the unknown and no other elements. In this type of search, knowledge of the elements not present allows the database to be screened to usually obtain a highly limited set of potential matches.

Finally, in step 160, the unknown material is identified by analyzing the results of steps 140 and 150. Advantageously, this is done simply by logically combining the data sets 1 and 2 using the Boolean AND operation to form a third data set 3 containing the data entries which are present in both sets 1 and 2. Hence, the entries in data set 3 have the same lattice structure and element types as the unknown material. Since research work has shown that materials can be accurately characterized on the basis of their lattice structures and chemical composition, it can be reasonably assumed that if an unknown material has the same lattice structure and "element types" as a known material in database A, the unknown material is the same compound.

The use of the converse transformation matrix generation method of the present invention in conjunction with symmetry analysis of lattice structures by an automated diffractometer will now be described. Referring to FIG. 8, an automated diffractometer according to the present invention comprises a conventional diffractometer 900 which includes a microprocessor-based controller 902 for controlling the orientation of a sample A relative to the diffractometer, and detecting and analyzing the diffraction signal data produced by the diffractometer. Referring to FIG. 9, the analysis of lattice symmetry according to the present invention is accom-

plished by collecting edge and angle parameter data with diffractometer 900 (step 910); defining any primitive cell and using the converse transformation matrix generation method of the present invention described hereinabove to generate a set (or sets) of symmetry matrices Hs which transforms the primitive cell into itself (step 920). More specifically, the symmetry matrices Hs in the following equation are determined:

$$a_i = \sum_j H_{sij} a_j \quad (i, j = 1, 2, 3),$$

where a_j and a_j define two primitive triplets of noncoplanar translations. Only matrices Hs with integer elements and a determinant HDET of +1 are considered. The primitive cell advantageously is defined as described hereinabove in connection with step 10 of FIG. 2. It will be appreciated that the data collection can be carried out by diffractometer 900 with respect to any basis.

The next step (930) is to determine the metric symmetry, which is accomplished through analysis of the tolerance matrices generated with the matrices Hs produced in step 920, and to assess the experimental error of unit cell parameters (which errors are directly related to the refined cell parameters). As discussed hereinabove, generated with each symmetry matrix Hs is a tolerance matrix T, which represents the tolerances (either absolute or relative percentage tolerances) in the cell parameters required to transform the primitive cell into itself by the specified matrix Hs. If the converse transformation method of the present invention generates a symmetry matrix Hs having a tolerance matrix:

$$\begin{matrix} tol a & tol b & tol c \\ tol \alpha & tol \beta & tol \gamma \end{matrix}$$

then the transformation of a first cell by the matrix Hs will produce a second cell having the parameters

$$\begin{matrix} a' = a + tol a \\ b' = b + tol b \\ c' = c + tol c \\ \alpha' = \alpha + tol \alpha \\ \beta' = \beta + tol \beta \\ \gamma' = \gamma + tol \gamma \end{matrix}$$

Thus the matrix method of the present invention enables a direct comparison of the calculated errors with the experimental errors for the refined unit cell. Further, by initially assuming very large experimental errors, a menu of all possible symmetries can be obtained from which the highest possible metric symmetry can be determined.

By analyzing the symmetry matrices Hs and associated tolerance matrices T, the metric symmetry groups are defined. The tolerance matrices T for the group(s) of symmetry matrices indicate precisely how the initially selected primitive cell deviates from exact metric symmetry and pseudosymmetry. In theory, it is the nature of the matrices themselves that defines the sets to be analyzed (i.e., those defining a symmetry group). In practice, however, the usual result is that the tolerance matrices alone clearly define the groups. Thus, with this approach, all possible symmetries and pseudosymmetries to within any specified maximum acceptable tolerance are immediately apparent. After a group of sym-