

symbols, assigning appropriate numeric indicators for each element symbol found according to 5. (a) (iii), and totaling these numeric indicators within each atomic number range (1 through 7) as in 5. (a) (iii).

The CODE for each known material is consistent throughout the relational database, so that it is possible to relate it to the elements present, the FORMULA, and all stored values of 100D1, 100D2, and 10PHI in a unique manner. Similarly, in the macro macZones, the CODE for each known material is used consistently throughout all queries, intermediate tables, macros, modules, and reports.

#### Running a Search/Match

The steps required to run a search/match of a single crystal electron diffraction pattern in ZONES, using the present embodiment of this invention, are:

- (1) Open the macro macZones.
- (2) In Input box 1: From the keyboard, enter a symbol of an element known to be present ( $Z > 10$ ). Repeat for each element ( $Z > 10$ ) known to be present. No unspecified heavy elements will be allowed in the solution, and all specified elements must be present. This requires a complete x-ray fluorescence (or other comparable elemental) analysis. Enter "0" (zero) to stop adding elements. All elements with  $Z \leq 10$  cannot be entered and are considered to be possibly present (i.e., any combination of these light elements, including none, is allowed).
- (3) Input box 2a: Enter  $d_1$ , the largest d-value of the zone, in Angstroms.
- (4) Input box 2b: Enter  $d_2$ , the second largest d-value of the zone, in Angstroms.
- (5) Input box 2c: Enter  $\phi$ , the angle between the repeat distances in the diffraction pattern corresponding to  $d_1$  and  $d_2$ , respectively, in degrees.
- (6) Input box 3a: Enter the experimental error limit on d-values in % (recommended input 1.5). A match occurs when a database d-value is within this percent of the experimental value, i.e. when

$$(100 - \text{err. } d) * d(\text{exptl.}) \leq [100D(\text{database, 1 or 2})] \leq (100 + \text{err. } d) * d(\text{exptl.})$$

- (7) Input box 3b: Enter the experimental error limit on  $\phi$  in degrees (recommended input 1.0). A match occurs when

$$10 * (\phi(\text{exptl.}) - \text{err. } \phi) \leq [10PHI(\text{database})] \leq 10 * (\phi(\text{exptl.}) + \text{err. } \phi)$$

#### BRIEF DESCRIPTION OF THE TABLES

- Table 1. Stored ZONES.
- Table 2. Fluorite.
- Table 3. Zircon.
- Table 4. Molybdenite.
- Table 5. Hornblende, first zone.
- Table 6. Hornblende, second zone.
- Table 7.  $\text{Fe}_3\text{C}$ .
- Table 8. Hollandite, first zone.
- Table 9. Hollandite, second zone.
- Table 10.  $\text{M}_2\text{X}$ , first zone, Nb.
- Table 11.  $\text{M}_2\text{X}$ , second zone, Nb.
- Table 12.  $\text{M}_2\text{X}$ , first zone, Ni.
- Table 13.  $\text{M}_2\text{X}$ , second zone, Ni.
- Table 14.  $\text{M}_6\text{C}$ , Cr, Nb.
- Table 15.  $\text{M}_6\text{C}$ , Fe, Nb.
- Table 16.  $\text{ZrH}_2$ .
- Table 17. Search Simulations.

#### EXAMPLES

In the following examples all searches were of the entire database of 79,136 phase entries. No other information about the sample was used, such as mineral, alloy, etc.

#### Example 1

Table 2 shows output for real input data from fluorite,  $\text{CaF}_2$ , as measured on film:  $d_1=3.19 \text{ \AA}$ ,  $d_2=3.18 \text{ \AA}$ ,  $\phi=70.2 \text{ deg}$ . All phases are correct except one (Ca), which has the poorest d-matches.

#### Example 2

Table 3 shows output for a search with uncalibrated film data from zircon,  $\text{ZrSiO}_4$ ,  $d_1=4.51 \text{ \AA}$ ,  $d_2=2.69 \text{ \AA}$ ,  $\phi=78.6 \text{ deg}$ . Three zircon phases are found.

#### Example 3

Molybdenite,  $\text{MoO}_3$ , is a TEM standard used to calibrate the rotational effect in going from transmission mode to diffraction mode. A common diffraction pattern has  $d_1=3.96 \text{ \AA}$ ,  $d_2=3.70 \text{ \AA}$ ,  $\phi=90.0 \text{ deg}$ . Table 4 gives the results of such a search, with six  $\text{MoO}_3$  phases found. These reflections are produced by double diffraction and are absent in the Powder Diffraction File (see below) for  $\text{MoO}_3$ .

#### Example 4

Table 5 shows output for literature experimental data on hornblende,  $\text{Ca}_2(\text{Mg, Fe})\text{Si}_8\text{O}_{22}(\text{OH, F})_2$  (Reference 10). The pattern was successfully identified with ZONES, without the need to index. Table 6 shows results from a second zone of hornblende (assumed) from another reference (Reference 11) by the same author. Both literature references assume hornblende to index the reflections, but we assumed only its elemental information (Ca, Fe, Mg, Si) and searched for the structure as an unknown.

#### Example 5

Table 7 shows output for  $\text{Fe}_3\text{C}$  from (Reference 12) considered as unknown data for a phase containing Fe. The experimental data had large errors of 4% and 2 deg., yet the correct phase solution was uniquely obtained, plus one boron isomorph. The reference assumed the known structure to index the reflections.

#### Example 6

Tables 8 and 9 are output for two zones from hollandite,  $\text{BaMn}_8\text{O}_{16}$  (Reference 13.). This example illustrates the case of obtaining two different zone axis patterns from the same physical crystal or chemically identical crystals in different orientations. Obviously, the same phase should be obtained as the solution for each, which is the case here. The authors used their own Minerals Database of 3822 minerals. Our search was over the entire NIST CD-derived inorganic database of 79,136 phases.

#### Example 7

Example 7 (Tables 10–15) is multifaceted (Reference 14). In this article the authors index forty zone axis electron diffraction patterns from various grains of Inconel 625 alloy containing the following major elements: Cr, Ni, Fe, Mo, Nb, plus others: C, Si, Mn, P, S, Ti, Al, N. They assumed six unit cells corresponding to the following possible phases ( $\text{M}=\text{metal}$ ,  $\text{X}=\text{nonmetal}$ ):  $\text{M}_{23}\text{C}_6$ ,  $\text{MoNi}_3$ ,  $\text{M}_6\text{C}$ ,  $\text{MX}$ ,  $\text{MX}$  ( $-2\%$  unit cell edges),  $\text{M}_2\text{X}$ . The authors search on four d-values computed from reciprocal lattice distances forming the sides and diagonals of a parallelogram. This data is redundant, and was reduced to the two largest d-values and included angle (as described above) for the search with ZONES.