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diffractometers The powder diffractometer has a proportional, scintillation or Geiger counter or an image plate detector which scans a range of values about a constant angular velocity (it is common practice to refer to the angle 2θ between diffracted beams, Fig. 5.12, rather than to the Bragg angle, θ). Usually, the range $10\text{--}80^\circ$ 2θ is sufficient to cover the most useful part of the powder pattern. A typical diffractometer trace is shown in Fig. 5.15(a) for the cristobalite polymorph of SiO₂. The scale is linear in 2θ and d-spacings of the peaks may be calculated from Bragg's law or obtained from standard tables of d versus 2θ . The scanning speed of the counter is usually $2\text{--}20$ min^{−1} and, therefore, about 30 min are needed to obtain a trace. Intensities are taken as either peak heights or, for accurate work, peak areas; the most intense peak is given an intensity of 100 and the rest are scaled accordingly. For accurate d-spacings, an internal standard (a pure material, such as KCl, whose d-spacings are known accurately) may be mixed with the sample. A correction factor, which may vary 245 Crystallography and Diffraction Techniques (a) Intensity Cristobalite (b) Silica glass 10 20 30 40 2 θ /" Figure 5.15 X-ray powder diffraction patterns of (a) cristobalite and (b) glassy SiO₂; Cu K α radiation, with 2θ , is obtained from the discrepancy between observed and true d-spacings of the standard and is then applied to the pattern that is being measured. Samples for diffractometry take various forms: they include thin layers of fine powder sprinkled onto a glass slide smeared with vaseline and thin flakes pressed onto a glass slide. The objective is always to obtain a sample which contains a random arrangement of crystal orientations. If the crystal arrangement is not random, then preferred orientation exists and can introduce errors, sometimes very large, into the measured intensities. Preferred orientation is a serious problem for materials that crystallise in a characteristic, very non-spherical shape, e.g. clay minerals, which usually occur as thin plates, or some cubic materials, which crystallise as cubes and, on crushing, break up into smaller cubes. In a powder aggregate of such materials, the crystals tend to sit on their faces, resulting in a non-random average orientation. 5.3.6.4 Guinier focusing cameras The arrangement of a focusing or Guinier camera, which uses a crystal monochromator M and also makes use of the theorem of the circle described above, is shown in Fig. 5.16(a). A convergent beam of monochromatic radiation passes through the sample at X. Radiation that is not diffracted comes to a focus at A, where a beam stop is placed in front of the film to prevent its blackening. Various beams diffracted by the sample focus at B, C, etc. We know from the theorem of the circle that A, B, C and X must lie on the circumference of a circle. The film is placed in a cassette which is in the form of a short cylinder and lies on the circle ABC. The scale of the film is linear in 2θ . A schematic film is as shown in Fig. 5.16(b) except that instead of peaks of different height, lines of different intensity or different degrees of blackness are seen. Film dimensions are $\sim 1 \times 15$ cm, which makes them very convenient to handle. The line at 0° 2θ or ∞ d-spacing corresponds to the undiffracted beam at A in (a) and is the reference position on the film. The Guinier method is capable of giving accurate d-spacings with results comparable to those obtained by diffractometry. Intensities are either estimated visually or measured using microdensitometry. Sample sizes are small, ≤ 1 mg, and exposure times vary between 5 min and 1 h, depending on factors such as the crystallinity of the sample and the presence or absence of heavy elements which absorb X-rays. Solid State Chemistry and its Applications, Second Edition, Student Edition 246 C B A S 2 θ X M Intensity (a) ∞ d - spacing/A 0° Bragg angle θ (or 2θ) (b) • Figure 5.16 (a) Crystal monochromator M, source S and sample X, in a focusing camera. (b) Schematic Guinier powder XRD pattern. 5.3.6.5 A powder pattern of a crystalline phase is its 'fingerprint' There are two main factors which determine XRD patterns: (i) the size and shape of the unit cell and (ii) the atomic number and position of atoms in the cell. Thus, two materials may have the same crystal structure but almost certainly will have distinct powder patterns. For example, KF, KCl and KI all have the rock salt structure and should show the same set of lines in their powder patterns, but, as can be seen from Table 5.3, both the positions and intensities of the lines are different in each. The positions or d-spacings vary because Table 5.3 X-ray powder diffraction patterns for potassium halides KF, a = 5.347 Å KCl, a = 6.2931 Å KI, a = 7.0655 Å (hkl) $d/\text{Å}^{\circ}$ 1 $d/\text{Å}^{\circ}$ 1 111 200 220 311 222 400 3.087 2.671 1.890 1.612 1.542 1.337 29 100 63 10 17 B 3.146 2.224 - 1.816 1.575 - 100 59 - 23 8 4.08 3.53 2.498 2.131 2.039 1.767 42 100 70 29 27 15 247 Crystallography and Diffraction Techniques the unit cells are of different size and, therefore, the a parameter in the d-spacing equation varies. Intensities differ because different anions with different atomic numbers and therefore different scattering powers are present in the three materials, even though the atomic coordinates are the same for each (i.e. cations at corner and face centre positions, etc.). KCl is a rather extreme example because the intensities of 111 and 311 reflections are too small to measure, but it serves to illustrate the importance of scattering power of the atoms present. Intensities are discussed in more detail in the next section. A powder XRD pattern has two characteristic features, therefore: the d-spacings of the lines and their intensity. Of the two, the d-spacing is more useful and capable of precise measurement. The d-spacings should be reproducible from sample to sample unless impurities are present to form a solid solution. Intensities are more difficult to measure quantitatively and often vary from sample to sample, especially if a preferred orientation is present. Thus, the differences in tabulated intensities for, say, the (220) reflection of the three materials in Table 5.3 are probably not absolute, quantitatively. The likelihood of two materials having the same cell parameters and d-spacings decreases considerably with decreasing crystal symmetry. Thus, cubic materials have only one variable, a, and there is a fair chance of finding two materials with the same a value. On the other hand, triclinic powder patterns have six variables, a, b, c, α , β and γ , and so accidental coincidences are far less likely. Problems of identification, if they occur, are most likely to be experienced with high symmetry, especially cubic, materials or in cases where similar-sized ions may replace each other in a particular structure as indicated in Section 5.3.6. 5.3.6.6 Powder patterns and crystal structures Although powder patterns are invaluable and unique for fingerprint identification of crystalline materials, a great deal of additional crystallographic information can be obtained from powder patterns without the necessity for full structure determination (which is also possible, as discussed later). As a first step, it is necessary to be able to index the powder patterns, i.e. to assign h, k, l indices to all lines in the powder diffraction pattern, and at the same time obtain the unit cell symmetry and dimensions. A general observation is that crystals with higher symmetry have simpler powder patterns, i.e. with fewer lines, than samples with lower symmetry recorded under the same experimental conditions. Thus, a cubic material may have 10 lines in a powder pattern recorded by a conventional diffractometer whereas a similar-sized triclinic crystal may have ~ 100 lines, many of which are of low intensity. This is because in a cubic powder pattern, each line represents the overlap of a number (up to 48) of lines of the same d-spacing, whereas in a material of lower symmetry, these lines do not overlap if they have different d-spacings. The number of lines that overlap as a consequence of crystal symmetry is the multiplicity. For example, in a cubic material, a line with indices h00 has multiplicity of 6 (h00, h00, 0k0, 0k0, 00l, 00l). In a tetragonal crystal for which a = b, the multiplicity of h00 is 4 (i.e. h00, h00, 0k0, 0k0), whereas the multiplicity of 00l is only 2 (00l, 00l). In an orthorhombic powder pattern, a good example of this effect is seen in the powder patterns of different polymorphs of BaTiO₃, shown in Fig. 5.17. BaTiO₃ shows the following sequence of polymorphs and symmetries with increasing temperature: -110° C 28 - C 125 - C 1470 - C rhombohedral - - - - - orthorhombic - - - - - tetragonal - - - - - cubic - - - - - hexagonal The cubic polymorph has a single line for the h00 reflection whereas the tetragonal polymorph shows a doublet with intensity ratio 2:1, reflecting the fact that the multiplicity ratio is 2:1; the orthorhombic Solid State Chemistry and its Applications, Second Edition, Student Edition 248 110 100 BaTiO₃, Cubic polymorph 111 100 101 0 100 BaTiO₃, Tetragonal polymorph 110 50 0 20 100 001 111 Relative intensity 50 30 2 θ /" 40 Figure 5.17 Section of the powder XRD patterns of BaTiO₃ showing the cubic polymorph and line splittings associated with the tetragonal polymorph. Note the difference in intensities of 001 and 100 lines and of the 101 and 110 lines of tetragonal BaTiO₃, which are a direct consequence of their different multiplicities. By contrast, the 111 reflection is a singlet in both cubic and tetragonal polymorphs, polymorph (not shown) gives a triplet. The powder patterns enable us not only to identify the material as BaTiO₃, but also to specify which polymorph, and symmetry, is being measured. This is important if one is interested in the material properties since, for instance, tetragonal BaTiO₃ is ferroelectric whereas cubic BaTiO₃ is not. Order-disorder phenomena are very important in many inorganic materials and can be seen clearly from changes in the XRD powder patterns. In Fig. 5.18 are shown powder patterns of several phases that have the basic rock salt structure but with various cation arrangements. In CoO (a), the structure is fcc rock salt and the first two lines in the cubic XRD pattern have indices (111) and (200). In LiCoO₂, (b), Li and Co cations occupy layers of alternate octahedral sites within the ccp oxide array; this cation ordering gives a larger unit cell, which is rhombohedral. Extra lines appear in the XRD pattern; the lines that correspond to the rock salt subcell are still present, but appear at higher 2θ (lower d-spacing) since there is a contraction in interatomic distances. In addition, the hk indices are different since the unit cell is different. In Li₂MnO₃, Fig. 5.18(c), the cation arrangement is also layered but fully occupied Li layers alternate with layers containing a mixture of Li and Mn arranged in ordered fashion; this structure is monoclinic and again, extra reflexions appear in comparison with CoO (a). All three powder patterns therefore contain the same basic sub-cell lines that are observed in a cubic rock salt structure, such as that of CoO, (a), but in addition, the patterns of LiCoO₂ and Li₂MnO₃ contain extra sets of reflexions associated with the cation order, which is different in these two cases. 5.3.7 Intensities Intensities of X-ray reflections are important for two main reasons. First, quantitative measurements of intensity are necessary in order to solve crystal structures. Second, qualitative or semi-quantitative intensity 249 Crystallography and Diffraction Techniques 111 200 100 CoO, Cubic 50 104 012 101 003 50 (b) 0 100 Li₂MnO₃, Monoclinic 50 0 10 Figure 5.18 202 111 133 002 Relative Intensity (a) 0 100 LiCoO₂, Rhombohedral (c) 20 30 2 θ / " 40 XRD patterns of some rock salt-related phases, data are needed in using the powder fingerprint method to characterise materials and especially in using the Powder Diffraction File to identify unknowns. Although this book is not concerned with the details of the methods of structure determination, it is important that the factors which control the intensity of X-ray reflections be understood. The topic falls into two parts: the intensity scattered by individual atoms and the resultant intensity scattered from the large number of atoms in a crystal. 5.3.7.1 Scattering of X-rays by an atom: atomic scattering factors or form factors Atoms diffract or scatter X-rays because an incident X-ray beam, which can be described as an electromagnetic wave with an oscillating electric field, sets each electron of an atom into vibration. A vibrating charge such as an electron emits radiation which is in phase or coherent with the incident X-ray beam. The electrons therefore act as secondary point sources of X-rays. Coherent scattering may be likened to an elastic collision between the wave and the electron: the wave is deflected by the electron without loss of energy and, therefore, without change of wavelength. The intensity of the radiation scattered coherently by 'point source' electrons is given by the Thomson equation: $IP \propto 1 (1 + \cos^2 2\theta) / 2$ (5.5) where IP is the scattered intensity at any point, P, and 2θ is the angle between the directions of the incident beam and the diffracted beam that passes through P. From this equation, the scattered beams are most intense when parallel or antiparallel to the incident beam and are weakest when at 90° to the incident beam. The Thomson equation is also known as the polarisation factor and is one of the standard angular correction factors that must be applied when processing intensity data for use in structure determination. At this point, it is worth mentioning that X-rays can interact with electrons in a different way to give Compton scattering. Compton scattering is an inelastic collision since the X-rays lose some of their energy Solid State Chemistry and its Applications, Second Edition, Student Edition 250 on impact and the scattered X-rays are therefore of longer wavelength. They are also no longer in-phase with the incident X-rays; nor are they in-phase with each other. Compton scattering is caused by interaction between X-rays and more loosely held valence electrons; it is an important effect with light elements and can have a deleterious effect on the powder patterns of organic materials such as polymers. A close similarity exists between Compton scattering and the generation of white radiation in an X-ray tube; both are examples of incoherent scattering that are sources of background radiation in XRD experiments. The X-rays scattered by an atom are the result of the waves scattered by each electron in the atom. For this purpose, electrons may be regarded as particles that momentarily occupy different positions in an atom and interference occurs between their scattered waves. For scattering in the direction of the incident beam, Fig. 5.19(a), beams 1 and 2, all electrons scatter in-phase irrespective of their position and the scattered intensity is the sum of the individual intensities. The scattering factor, or form factor, f , of an atom is proportional to its atomic number, Z , or, more strictly, to the number of electrons possessed by that atom. For scattering at some angle 2θ to the direction of the incident beam, a phase difference, corresponding to the distance XY, exists between beams 1 and 2. This phase difference is usually less than one wavelength λ for Cu K α X-rays because distances between electrons within an atom are short and (i.e. XY

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