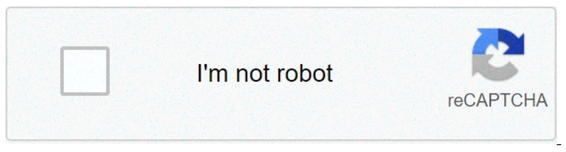

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Nacl x ray diffraction peaks

You are using an out of date browser. It may not display this or other websites correctly.You should upgrade or use an alternative browser. In my x-ray crystallography data for a single crystal of NaCl using x-rays generated by Cu k alpha (154 pm), there are three peaks identified at angles of 14.155, 31.475 and 53.5 degrees. According to fabo02/solid/xray.pdf, these peaks must correspond to only even values of h, k and l (as there is quite a lot of background noise) How can I then identify which peak corresponds to which layers? Answers and Replies Lord Jestocost Why not following the “recipes” given in fabo02/solid/xray.pdf. As you are using Cu K-alpha X-ray radiation, you can use equation (8) to determine Sqrt(h2 + k2 + l2) for the three measured angles in order to roughly check whether there is something in common. As you are measuring on a NaCl single crystal, the measured peaks must belong to a certain set of planes. Why not following the “recipes” given in fabo02/solid/xray.pdf. As you are using Cu K-alpha X-ray radiation, you can use equation (8) to determine Sqrt(h2 + k2 + l2) for the three measured angles in order to roughly check whether there is something in common. As you are measuring on a NaCl single crystal, the measured peaks must belong to a certain set of planes. Many thanks for your reply. I have find the values of h, k, l as being closest to (2, 0, 0), (4, 0, 0), (6, 0, 0) as my parallel planes. Could I use to justify that the structure of the crystal is face-centered cubic? Lord Jestocost Could I use to justify that the structure of the crystal is face-centered cubic? I don’t think so. Appropriate aligned crystals with other crystal structures could give rise to a peak pattern which could be assigned in the same way. I don’t think so. Appropriate aligned crystals with other crystal structures could give rise to a peak pattern which could be assigned in the same way. Can I safely say that the planes identified are indeed the correct ones, even though the plane of the cut of the crystal is unknown? Also, can I use the method in to calculate the relative intensities, will the formula work for single crystals as well? Lord Jestocost Can I safely say that the planes identified are indeed the correct ones, even though the plane of the cut of the crystal is unknown? As you are measuring on a NaCl single crystal, the planes identified seem to be the correct ones. I am, however, surprised about the measured theta values of 14.155, 31.475 and 53.5 degrees which indicate a severe misalignment of your diffractometer. The correct ones should be 15.85° (200), 33.11° (400) and 55.02° (600) when assuming a lattice parameter of 0.5640 nm. As you are measuring on a NaCl single crystal, the planes identified seem to be the correct ones. I am, however, surprised about the measured theta values of 14.155, 31.475 and 53.5 degrees which indicate a severe misalignment of your diffractometer. The correct ones should be 15.85° (200), 33.11° (400) and 55.02° (600) when assuming a lattice parameter of 0.5640 nm. Yes, unfortunately the equipment we were given was not well aligned and so an offset had to be taken into account. Should I perhaps identify the planes using the ratio of the sin^2(measured angle 1) / sin^2(measured angle 2 - of next peak) = (h1^2 + k1^2 + l1^2)/ n^2(h2^2 + k2^2 + l2^2)? Would this work? Lord Jestocost The best way would be to correct the peak positions to a first approximation by applying a simple zero point shift. Sodium chloride, also known as salt or halite, is an ionic compound with the chemical formula NaCl, representing a 1:1 ratio of sodium and chloride ions. With molar masses of 22.99 and 35.45 g/mol respectively, 100 g of NaCl contain 39.34 g Na and 60.66 g Cl. The salient features of its structure are: Chloride ions are ccp type of arrangement, i.e., it contains chloride ions at the corners and at the center of each face of the cube. Sodium ions are so located that there are six chloride ions around it. This equivalent to saying that sodium ions occupy all the octahedral sites. As there is only one octahedral site for every chloride ion, the stoichiometry is 1 : 1. It is obvious from the diagram that each chloride ion is surrounded by six sodium ions which are disposed towards the corners of a regular octahedron. We may say that cations and anions are present in equivalent positions and the structure has 6 : 6 coordination. The structure of sodium chloride consists of eight ions a unit cell, four are Na+ ions and the other four are Cl– ions. In this structure, each corner ion is shared between eight unit cells, each ion a face of the cell by two cells, each ion on a edge by four cells and the ion inside the cell belongs entirely to that unit cell. So the positions of the ions are the followings (with Na at the center of axis) : Na+ (0 0 0) (1/2 1/2 0) (1/2 0 1/2) (0 1/2 1/2) Cl– (0 1/2 0) (1/2 0 0) (0 0 1/2) (1/2 1/2 1/2) With this information we can compute the Structure Factor Fhkl, assuming the scattering factors of Na and Cl are expressed by fNa and fCl, respectively. The structure factors determines the amplitude and the phase of the diffracted beams. The intensity (measured value) is linked to the squared module of the Structure Factor. Ihkl ∝ | Fhkl|2 Definition of For a perfect crystal the lattice gives the reciprocal lattice, which determines the positions (angles) of diffracted beams, and the basis gives the structure factor which determines the amplitude and phase of the diffracted beams for the (hkl) crystal plane. where the sum is over all atoms in the unit cell, are the positional coordinates of the jth atom, and is the scattering factor of the jth atom. The coordinates have the directions and dimensions of the lattice vectors . That is, (0,0,0) is at the lattice point, the origin of position in the unit cell; (1,0,0) is at the next lattice point along and (1/2, 1/2, 1/2) is at the body center of the unit cell. defines a reciprocal lattice point at which corresponds to the real-space plane defined by the Miller indices. Face-centered cubic (FCC) The FCC lattice is a Bravais lattice, and its Fourier transform is a body-centered cubic lattice. However to obtain without this shortcut, consider an FCC crystal with one atom at each lattice point as a primitive or simple cubic with a basis of 4 atoms, at the origin (0, 0, 0) and at the three adjacent face centers, (1/2,1/2,0), (0,1/2,1/2) and (1/2,0,1/2). Equation becomes with the result The most intense diffraction peak from a material that crystallizes in the FCC structure is typically the (111). Films of FCC materials like gold tend to grow in a (111) orientation with a triangular surface symmetry. A zero diffracted intensity for a group of diffracted beams (here, of mixed parity) is called a systematic absence. NaCl Strucure NaCl is composed by the Na+ FCC unit cell plus the displace Cl– FCC unit cell, thus the calculation of is similar to the pure FCC case and becomes the following : Fhkl = [1 + (-1)h+k + (-1)k+l + (-1)h+l] [fNa + (-1)hfCl] that can be tabulated for each hkl triplet : hkl Fhkl 100 0 110 0 111 4(fNa -fCl) 200 4(fNa +fCl) 210 0 211 0 220 4(fNa +fCl) 300 0 221 0 310 0 311 4(fNa -fCl) 222 4(fNa +fCl) It can be noted that the main peaks are the reflections from 200 and 220 planes, whilst the reflection from the plane 111 is present but it is weak because it arises from the scattering difference from the two types of ion. Most of the alkali halides, alkaline earth oxides, and sulphides exhibit this type of structure. XRD Patterns The first test performed was the complete 10° to 120° scan of the crystal, using the non-filtered tube emission, configured at 30 KV and 80 µA. The result is shown in the graph below, in which the Bragg reflections for the Kα and Kβ lines of the copper are present. The reflections were highlighted for the order n = 1, n = 2 and n = 3. For the crystallographic analysis of sodium chloride we used the Nickel filter to have monochromatic emission in correspondence only to the Kα line of the copper at 0.1542 nm. We examined the NaCl crystal in the two orientations allowed by our protractor, corresponding to the crystal orientations (200) and (020). The images below show the setup of the measurements and the Bragg peaks obtained, both at about 31° 30′. Having obtained the same results, within the experimental error, is the demonstration that we are dealing with a crystal of cubic structure. By rotating by 45° the crystal, together with the crystal holder, with respect to the movable arm, we sought the presence of reflections corresponding to the planes (nn0), the graph below shows the result obtained with the Bragg peak corresponding to the plane (220). Further experimental investigations can be made by diffraction from powders or with the Laue technique having a small thickness single crystal available, but at the moment we have to settle for what we have already obtained and proceed to the “resolution” of the crystal with the available data. “Solving” the Crystal Structure From the checks made, described above, it has been established that the sodium chloride crystal has a cubic structure. The variants of the cubic lattice are the followings : Primitive cubic Body-centered cubic (bcc) Face-centered cubic (fcc) Diamond (or Zincblende) each of these is characterized by a specific pattern of reflections. We verify that the experimentally obtained Bragg peaks are compatible with the fcc structure. Meanwhile for a cubic crystal with lattice constant a, the distance d between adjacent lattice planes (hkl) is computed as follows: By combining the Bragg condition for the reflections with the above written equation we get : From which we deduce that, for the diffraction peaks, the ratio remains constant and this allows to verify the type of cubic structure and to determine the lattice constant a, if the wavelength λ of the X-rays is known. For the measured Bragg reflections we can compile the following table for which we hypothesize an fcc structure, using the following formulas: λ = 0.1542 nm d = λ / 2senθ a = d x √(h2+k2+l2) hkl 2θ d(nm) h2+k2+l2 √(h2+k2+l2) a(nm) 200 31.50 0.284 4 2 0.568 220 45.83 0.198 8 2.828 0.560 The values of a obtained are congruent with each other, a sign of the correctness of the initial hypotheses. They are also close to the correct value which turns out to be 0.564 nm. Laue Diffractogram The image below shows the Laue diagram of a NaCl (100) single crystal with a face-center cubic crystal lattice (fcc). If the diffraction pattern is rotated by 90° around the direction of the primary beam, it is again brought to coincidence. Since the primary beam impinges perpendicularly on the (100)-plane of the NaCl crystal, the crystal direction [100] is a fourfold axis of symmetry. The intensity of the reflections depends on the reflecting crystal surface as well as on the spectral intensity distribution of the X-rays. If you liked this post you can share it on the “social” Facebook, Twitter or LinkedIn with the buttons below. This way you can help us! Thank you ! Donation If you like this site and if you want to contribute to the development of the activities you can make a donation, thank you ! Related