Title: Indexing of KI and $SrTiO_3$ X-ray powder diffraction patterns

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Abstract

In X-ray diffraction, Bragg's law connects measurable angles of diffraction to planar spacings within crystal structures. Only certain combinations of planar spacings are allowed depending on lattice type. This study tests the applicability of Bragg's law to potassium iodide (KI) and strontium titanate ($SrTiO_3$) using powder X-ray diffraction. KI indexed to a face-centered cubic lattice, while $SrTiO_3$ indexed to a simple cubic lattice. These lattices agree with the literature listed structures of rock salt and perovskite, respectively.

Introduction

X-ray diffraction (XRD) is a common technique used to determine the crystalline structure of a wide range of materials. When subjected to monochromatic X-ray beams (λ), the periodic planes (d) within a crystal will produce strong intensity diffracted beams if certain orientation criteria are met [1]. If the crystal is oriented such that the incident angle (θ_B) of the beam satisfies Bragg's law (Eq. 1) for a set of periodic planes in the crystal, then a diffracted beam will leave the crystal at the same angle (θ_B) [2].

$$(1) \quad n\lambda = 2d \sin (\theta_B)$$

In powder XRD, the orientation requirements are relaxed as a fully powdered sample should contain every crystal orientation. The simultaneous satisfaction of Bragg's law for each set of planes within a crystal produces all possible diffracted beams (Fig. 1).
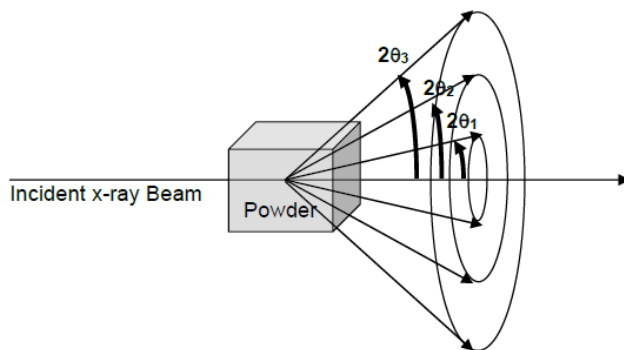

Figure 1 - Simultaneous generation of diffracted X-ray beams, each with a unique angle (2θ) in a powder sample. Adapted from Hobbs [2].

The set of angles of these diffracted beams are characteristic of each material and can be used to identify the material. Additionally, set of angles can be calculated back into a set of planar spacings using Eq. 1. Assuming a cubic lattice parameter (a_o), the planar spacings can indexed

the respective {hkl} family of planes using Eq. 2. Lattice type can then be determined using the selection rules of allowed planes [1,3].

$$(2)\ d_{hkl} = \frac{a_o}{\sqrt{h^2+k^2+l^2}}$$

In this experiment, the powder diffraction patterns of potassium iodide (KI) and strontium titanate (SrTiO$_3$) were collected and indexed to test Bragg's law and the cubic lattice type selection rules.

Methods

Lab grade purity potassium iodide (KI) and strontium titanate (SrTiO3) were powdered using a mortar and pestle and epoxied into aluminum sample holders. X-Ray diffraction (XRD) data was collected at angles from 2 - 120° generated over 10 minutes for each sample using an ARL Equinox 100 benchtop X-Ray diffractometer operating at 40kV and 0.9 mA with a Cu K-α radiation source (λ= 1. 5406 Å) and. Peaks were identified using Match! software and indexed using lattice parameters from Wyckoff (1963). The indices were then used to confirm the Bravais Lattice selection rules.

Results

Application of Bragg's law to all indexed peaks yields a d^2/a_o^2 within 0.2 of a whole number. The deviation is consistent smaller at low angles and greater at higher angles (Table 1,2). Powdered KI exhibits fourteen strong peaks between 2θ = 10-100° (Fig. 2). Using a lattice parameter of 7.066 Å, the first indexed peak is (111) at 21.87° (Table 1). All indexed peaks for KI follow the face centered cubic selection rule of *h, k, l* being either all even or all odd. Powdered SrTiO$_3$ exhibits eight strong peaks between 2θ = 10-100° (Fig. 3). The SrTiO$_3$ pattern also contains a weak peak at 22.79°. Using a lattice parameter of 3.899 Å, the first weak indexed peak is (100) at 22.79° while the first strong indexed peak is (110) at 32.45° (Table 2). All indexed peaks for SrTiO$_3$ follow the selection rules of simple cubic of *h, k, l* being any value.
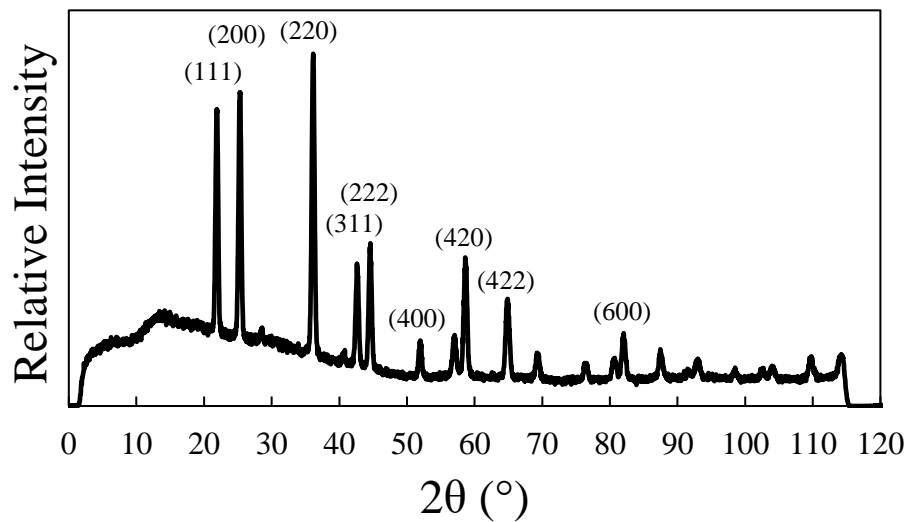
Figure 2 - X-ray diffraction pattern of powdered KI. Data was collected with Cu Kα radiation (λ =1.5046 Å). Strong peaks have been indexed. Full calculations can be found in Table 1.

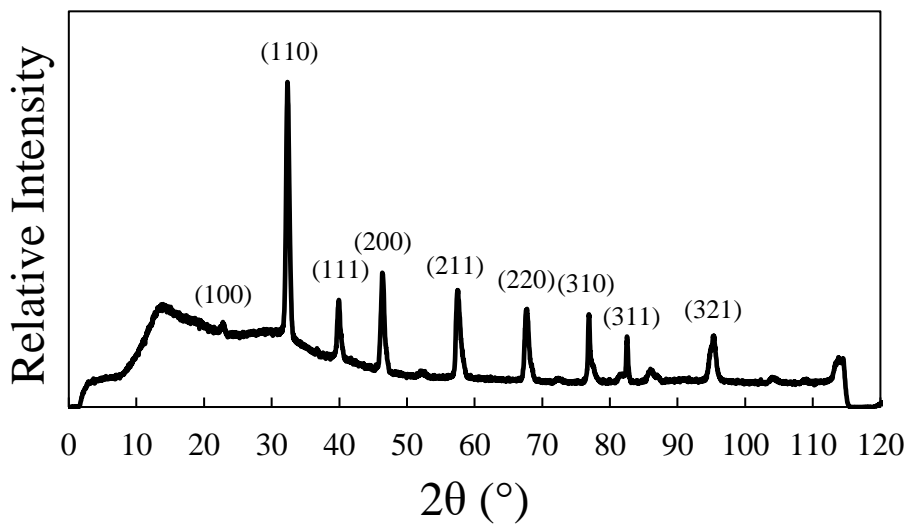*Figure 3 - X-ray diffraction pattern of powdered SrTiO₃. Data was collected with Cu Kα radiation (λ =1.5046 Å). Strong peaks have been indexed. Full calculations can be found in Table 2.*

Table 1 – Indexed X-ray diffraction peaks for powdered KI. Data was collected using Cu Kα radiation (λ =1.5046 Å). Indexing calculations were performed with a lattice parameter of a_o = 7.066 Å.

2θ (°)	d_{hkl} (Å)	a_o^2/d^2	(h k l)
21.94	4.05	3.05	(111)
25.44	3.50	4.08	(200)
35.90	2.50	7.99	(220)
42.36	2.13	10.98	(311)
44.63	2.03	12.13	(222)
52.08	1.76	16.21	(400)
56.96	1.62	19.14	(331)
58.45	1.58	20.05	(420)
65.21	1.43	24.42	(422)
69.64	1.35	27.44	(511)
76.12	1.25	31.95	(440)
87.76	1.11	40.45	(620)
93.18	1.06	44.44	(622)

Table 2 – Indexed X-ray diffraction peaks for powdered SrTiO₃. Data was collected using Cu Kα radiation (λ =1.5046 Å). Indexing calculations were performed with a lattice parameter of a_o = 3.899 Å.

2θ (°)	d_{hkl} (Å)	a_o^2/d^2	(h k l)
22.79	3.81	1.05	(100)
32.45	2.69	2.10	(110)
39.83	2.22	3.08	(111)
46.50	1.95	4.00	(200)
57.38	1.60	5.94	(211)
67.65	1.38	7.98	(220)
77.02	1.24	9.89	(310)
82.65	1.17	11.11	(311)
95.44	1.04	14.06	(321)

Discussion

The results of d^2/a_o^2 nearly equaling a whole number across a wide range of angles suggests Bragg's law holds for the X-ray diffraction of cubic systems. The consistently increasing deviation from whole numbers at higher angles suggests a systematic error proportional to the diffraction angle. The additional minor peaks seen in both patterns, although more prominent in the KI pattern, can be attributed to diffraction of the aluminum sample hold. Likewise, the broad amorphous hump from 2-50° is consistent with the poorly crystalline epoxy used to bind both powders.

KI is face-centered cubic based on the selection rules of *h, k, l* being either all even or all odd. KI cannot be body-centered cubic as the (111) peak violates the *h+k+l* being even. The lowest 2θ angle being (111) is also consistent with the closest packed plane for a face-centered cubic system. Literature states KI exhibits a rock salt structure, a two atom basis on an FCC lattice, which agrees with our lattice determination [3].

SrTiO₃ is simple cubic due to process of elimination as the (100) peak violates both the face-centered cubic selection rule where *h, k, l* are all odd or all even and the body-centered cubic selection rule where *h+k+l* is even. The lowest 2θ angle being (100) is also consistent with the closest packed plane for a simple cubic system. The weakness of the (100) peak is due to the closeness in scattering factor of the Sr and Ti atoms, causing the cubic lattice to behave more like a body-centered cubic lattice at low angles. However, even without the weak (100) peak, the

presence of both the (110) and (111) peak verify the simple cubic nature of $SrTiO_3$ as those two strong peaks are incompatible in any other cubic symmetry. Our simple cubic classification of $SrTiO_3$ agrees with the literature listed perovskite structure [3].

Conclusion

The simplicity of Bragg's law combined with the broad accessibility of X-ray powder diffraction provides a powerful tool for the determination of lattice systems. This study not only confirms the applicability of Bragg's law to cubic crystal systems, but also corroborates the literature values for the KI and $SrTiO_3$ crystal structures. Additionally, the data suggest there may be a systematic error associated with X-ray diffraction that warrants further study.

References

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