Oral presentation

Absolute intensities & minimal required number of crystals in PXRD

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Powder X-ray diffraction is a widely used technique, but almost always using relative intensities. Even when extracting the absolute content of a specific crystalline component in a complex sample, typically a reference is used [1]. For neutron powder diffraction, some attempts for absolute intensity measurements have been made [2]. Modern photon-counting detectors facilitate the use of absolute intensities and thus we have tested how accurately we can calculate and measure powder X-ray diffraction on an absolute scale for both the Bragg-Brentano (see figure 1) and capillary geometries, and taking proper account of absorption, sample size, preferred orientation, etc. We used a laboratory instrument for this.

For simple test samples, in particular corundum Al_2O_3 and diamond, we find excellent agreement with a deviation that can be as small as 10%. This shows that classical diffraction theory works very well and does not point to the need for a new theory [3].

We have further tested the limits of how many independent crystals are needed to constitute a proper powder. For a normal sample in the Bragg-Brentano geometry, the number of probed crystals can be several million, but here we tested the use of a very small and exact number of crystals, down to 10 crystals. As expected, we find that the number of required crystals depends strongly on the geometry, where a stationary Bregg-Brentano geometry requires many more crystals than a spinning capillary.

The calculations of the absolute intensities and of required number of crystals are facilitated by using a convenient form of the differential cross section:

$$\frac{d\sigma}{d\Omega} = r_e^2 \frac{V}{V_u} P |F_{hkl}|^2 u(\boldsymbol{Q}), \quad (1)$$

making use of a normalized profile function $u(\boldsymbol{Q})$ for a specific reflection, with $\boldsymbol{Q}$ the momentum transfer.

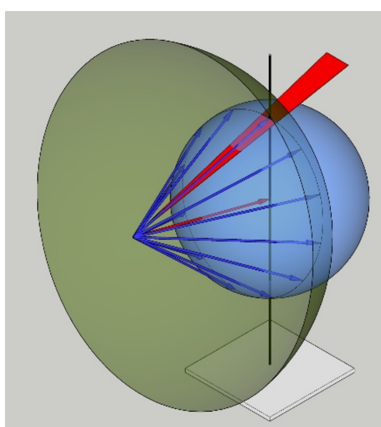

Figure 1. Schematic of the Bragg-Brentano geometry, showing the cross section of the Ewald sphere (green) with the sphere of a specific reflection (blue), leading to a cone of diffracted X-rays (Debye ring). Typically, a small fraction (read beam) is detected.

[1] Hubbard C.R., Evans E.H & Smith D.K. (1976). J. Appl. Cryst. ***9****,169.*

[2] Merisalo M., Paakkari T. (1975), J. Appl. Cryst. ***8****, 522.*

[3] Fewster, P.F., (2014) Acta Cryst. ***A70****, 257.*