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APPARENT LONG SPACINGS FROM CLAY-WATER GELS, GLASSES, AND CRYSTALLINE MATERIALS DUE TO TOTAL REFLECTION OF X-RAYS: A COMMENT

Key Words—Diffractometer alignment, Long spacings, Reflection, X-ray powder diffraction.

In a recent article, Brindley and Simonton (1984) claimed that they were able to measure long spacings in glass, mica, clay minerals, and clay-water gels with an X-ray powder diffraction apparatus due to total reflection of X-rays under low angles. The spacings were observed by raising the sample somewhat above the axis of the diffractometer. After a displacement of 0.86 mm, the recorded pattern showed a broad peak at about 1°2θ, and with further displacement, the peak maximum moved toward higher angles. Extrapolation of the measured 2θ angles gave a value for the original aligned position, interpreted as representing the real spacing. We will here demonstrate that these authors in fact screened the incident beam with the sample and, thus, measured only artifacts.

Figure 1 shows the X-ray paths in the diffractometer used by Brindley and Simonton, fitted with a ¼° fixed divergence slit which has an opening of 0.30 mm (Philips Application Laboratories, Almelo, the Netherlands, personal communication). Assuming that a broad-focus Philips tube is applied, focus depth is 2 mm. If the tube is mounted at an angle of 6°, as usual, a 0.2-mm virtual focus height can be calculated. We assume that the sample holder, which has a 4-cm long cavity, was about 5 cm long.

At 0°2θ, with the sample holder in normal position (position A), the incident beam coming from the upper half of the focus will theoretically reach the receiving slit at S_0. The lowest part can be recorded up to S_1, which is at 0.36°2θ. In practice, however, the incident beam is still recorded at higher angles, about 1.5 times larger (S_2) due to X-rays produced somewhat outside the given focus (F_2).

Again at 0°2θ, but raising the sample holder, the incident beam will be completely screened by a displacement of 0.057 mm (position B). The entire focus can be detected again if the diffractometer is moved to angles larger than 0.03–0.04°2θ. The left side of the sample holder will reach line F_1S_1 after a displacement of 0.49 mm. Following this displacement, the incident beam will no longer irradiate the receiving slit during the scan. In practice, the sample holder must be raised to line F_2S_2 in order to screen the beam completely, because of the diffuseness of the focus.

The diffraction patterns of Brindley and Simonton (1984, figure 2) demonstrate that the incident beam was already partly interrupted during the experiments when the samples were raised to 0.70 mm, because the patterns show a break (at 1°2θ). By raising the sample further, 0.86 mm or more, the incident beam was no longer measured at the lowest angles; that is, the entire beam had been screened.

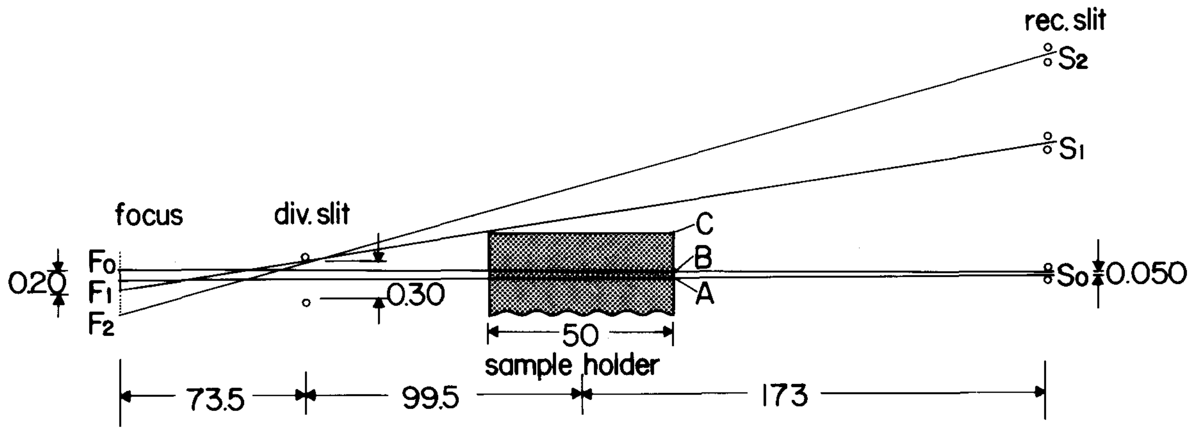

Figure 1. Schematic view of X-ray paths in the diffractometer used by Brindley and Simonton (1984). A, B, and C indicate different positions of the top of the displaced sample holder. Distances are in millimeters.

At higher angles only scattering was recorded, decreasing with increasing angle as is described by the Lorentz-polarization factor. The result was a diffraction peak measured at an angle which depended on both the size of the screened part of the incident beam and the disorientation of the diffractometer caused by the displacement of the sample. This phenomenon is nicely illustrated by Brindley and Simonton's Figure 2 where, starting from a displacement of 0.86 mm, each diffraction curve is formed by cutting off the lowest-angle part of the previous curve.

We conclude that Brindley and Simonton (1984) measured neither long spacings caused by total reflection nor a displacement of them, but rather artificial peaks due to misalignment of the diffractometer. It is therefore not surprising that they recorded very similar results for different types of materials.

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REFERENCE

Brindley, G. W. and Simonton, T. C. (1984) Apparent long spacings from clay-water gels, glasses, and crystalline materials due to total reflectance of X-rays: *Clays & Clay Minerals* **32**, 235–237.

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APPARENT LONG SPACINGS FROM CLAY-WATER GELS, GLASSES, AND CRYSTALLINE MATERIALS DUE TO TOTAL REFLECTION OF X-RAYS: REPLY TO COMMENT

Key Words—Diffractometer alignment, Long spacings, Reflection, X-ray powder diffraction.

van der Gaast and Jansen (1985) made some interesting comments as to the cause of apparent long spacings from clay-water gels and other materials; however, they misunderstood the intent of the note by Brindley and Simonton (1984). In no way did we attempt to measure true long spacings by total reflection of X-rays. The purpose of the note was to warn the reader that apparent long spacings, or spurious peaks, on the order of 150 Å *could* be observed *if* the surface of gel smears, single mica flakes, and other materials was raised sufficiently above the axis of the goniometer. Moreover, the ramifications of sample displacement above the goniometer axis were discussed as they related to coefficient of variation calculations for interstratified clay minerals.

We were aware of the fact that the incident X-ray beam was screened by the elevated sample and illustrated the effect in Figure 2. The extrapolation of data in Figure 1 was given as evidence to support the theory that the apparent long spacings may be due to reflection of X-rays which occurs between 0.33° and 0.50°2θ.

The only valid point of contention that van der Gaast and Jansen posed was that the apparent long spacings observed may not have been due to reflection of X-rays, but instead were artifacts produced by the screening of the incident beam. However, this is a minor point. To reiterate, the purpose of the Brindley and Simonton (1984) note was to warn investigators that displacement of the sample surface above the goniometer axis could give spurious peaks or shift true crystalline reflections from their correct positions.

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REFERENCES

Brindley, G. W. and Simonton, T. C. (1984) Apparent long spacings from clay-water gels, glasses, and crystalline materials due to total reflection of X-rays: *Clays & Clay Minerals* **32**, 235–237.

van der Gaast, S. J. and Jansen, J. H. F. (1985) Apparent long spacings from clay-water gels, glasses, and crystalline materials due to total reflection of X-rays: a comment: *Clays & Clay Minerals* **33**, 471–472.

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