American Mineralogist, Volume 100, pages 1899–1911, 2015

Grain size measurement from two-dimensional micro-X-ray diffraction: Laboratory application of a radial integration technique

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Abstract

Two-dimensional X-ray diffraction data contain information about not only the type of mineral phases present in an assemblage, but also the textural or grain size relationships between minerals in a sample. For minerals within a certain grain size range, ~0.1 to 100 μm, the appearance and characteristics of a Debye ring can reveal the mean grain size of a sample. In this contribution, using mineral and rock samples of known grain size ranges, we investigate the applicability of calculating the grain size of a material using a two-dimensional X-ray diffraction crystallite size analysis method for micrometer-sized materials. A radial integration technique was used to derive the number of grains contributing to produce diffraction spots in the Debye ring. Monomineralic pyroxene and magnetite samples of known grain size ranges were analyzed, and the calculated grain size was observed to broadly correlate with the sample size except at the upper and lower extremes. To evaluate the technique on broader geological materials, polymineralic basalt samples with known grain size ranges were analyzed, and the calculated grain sizes did not correlate with the size of the rock fragments, but did correlate closely with the size of the individual mineral grains. Using a Bruker D8 Discover X-ray diffractometer with a 300 μm nominal incident beam diameter, the effectiveness of the applied method appeared limited to the grain size range of ~15–63 μm for monomineralic samples. The method is further limited by several complicating factors and assumptions, including the requirement for the crystallite size to correlate with the sample grain size. The effective range of this method will vary with different instrumental and experimental conditions. When applying this method to calculate the grain size of geological materials, the calculated result should be interpreted as a minimum estimate of the grain size.

Keywords: Micro-X-ray diffraction, two-dimensional X-ray diffraction, grain size, crystallite size, χ-profile, γ-profile