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

INTRODUCTION

Throughout the century-long history of X-ray diffraction, methods have been developed and applied to measure the size distribution of crystalline materials with two-dimensional X-ray diffraction (2D XRD) images by studying the characteristics of diffraction spots on the images and their relationship within a Debye ring. Deciphering the grain size relationships with the progression of smooth Debye rings to “spotty” rings, and finally to large diffraction spots as the grain sizes of micrometer-sized minerals increased was pursued in two manners: (1) qualitative description of the Debye ring characteristics of minerals of known grain sizes with broad qualitative application to other minerals; and (2) more quantitative attempts to measure parameters from 2D XRD images and calculate a given grain size with some accuracy.

The qualitative method of grain size identification can be seen in the study of Debye-Scherrer X-ray diffraction film characteristics by several authors (e.g., Azároff and Buerger 1958; Klug and Alexander 1974; Cullity 1978) who presented observations of the visual qualities of the Debye rings of samples with known grain size to which samples with unknown grain size could then be compared. These observations can be collectively summarized as follows: Below ~0.1 μm, Debye rings will display line broadening, and the lines will broaden with decreasing grain size until ~0.01 μm where the irradiated sample will begin a transition toward being X-ray amorphous. In the size range of 0.1 to 10 μm, a “perfect” powder X-ray diffraction pattern with thin, clearly discernible rings will exist, although there is not complete agreement on the exact transitions zones. Cullity (1978) stated that the transition from continuous rings without spots to spotty diffraction rings occurs between 1 and 10 μm, whereas Azároff and Buerger (1958) state that between 10 and 40 μm, the sample has clearly discernable diffraction rings consisting of very many spots that are closely spaced. Klug and Alexander (1974) place continuous rings at <5 μm and spotty rings at 15–50 μm for quartz. Beyond ~50 μm Debye rings become progressively more discontinuous, and by ~200 μm or larger only a few diffraction spots are scattered on the film. Hörz and Quaide (1973) give a summary of Debye ring characteristics pertaining to the grain block size in several minerals.

In the finer grain size range where the Debye rings begin to broaden in the transition toward becoming X-ray amorphous,