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Determination of crystal size distribution by two-dimensional X-ray diffraction

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Polycrystalline materials properties and behaviour are ultimately determined by their crystallinity, phase composition and microstructure (i.e., crystal size, preferential orientation). Two-dimensional (2D) diffraction patterns collected with an area detector (i.e., CDD), available in modern X-ray diffractometers, contain detailed information about all these important material characteristics. Furthermore, recent advances in detector technologies permits the collection of high resolution diffraction patterns in which the microstructure of the material can be directly imaged. If the size of beam relative to the crystal size in the sample is adequately choosen, the diffraction pattern produced will have spotty rings in which the spots are the diffracted images of individual grains. The resolution of the image is mainly dependent on the characteristics of the X-ray beam (i.e., diameter, angular divergence), which can be modulated by X-ray optics, sample to detector distance, the pixel size of the detector and the sharpness of the point spread function. From these patterns, the crystal size distribution of different crystalline phases present in the sample can be independently determined using specialized software capable of extracting and combining the information contained in these patterns. This technique is applicable to materials with crystal sizes ranging from submicron to mm sizes and is complementary to techniques based on peak profile analyses (i.e., Scherrer method) which are applicable only to nanocrystalline materials. Finally, given the high sensitivity of current detectors, crystal size evolution can be followed in real-time to study important transformation processes such as crystallization, annealing, etc. The use of 2D X-ray diffraction as applied to microstructure characterization will be illustrated through several examples.

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