Poster Presentation

FAST RIETVELD METHOD ALGORITHM FOR IN-SITU XRD

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X-ray diffraction is an analytical technique used to identify and quantify phases in a crystalline material. In a diffractometer, an X-ray with constant wavelength is directed to the sample which diffracts the wave in many directions with intensities that depend on the structure of the crystal. The result of the experiment is a diffractogram that presents the intensity of the diffracted wave at each measured angle. Each phase of the material results in a different pattern on the diffractogram, in such way that it is possible to quantify the amount of each phase in the sample. The Rietveld refinement method is probably the most used algorithm to refine the structure of crystals and to quantify the amount of each phase. The Rietveld method is based on the solution of a least-squares problem where it is minimized the difference between the diffractogram peaks collected in an experiment and a mathematical model of the crystal. Since the intensity of the peaks is proportional to the quantity of each phase in the sample, the method present good results in phase quantification. In-situ XRD comprises an experiment where many measures are made from the sample in different instances of time, resulting in a sequence of diffractograms that show the changes in the sample with time. It can be used to study and understand chemical reactions that form and consume different crystal in time. For instance, when cement is hydrated, many crystalline materials are consumed and formed until the reaction finishes. In many applications, in-situ XRD produces and huge amount of data, since samples may be collected once per minute for a complete day (or several days) resulting in thousand of diffractograms, where each one usually is close to one megabyte of data, totalizing gigabytes of data. This large amount of data is difficult to save and transport, but much more difficult is to analyze and to extract the information from each diffractogram, and to obtain useful information from these set of diffractograms. This problem is becoming much more relevant with the new sources of light like Sirius in CNPEM, since a brighter light allows shorter samples and therefore many more samples for each day. Rietveld refinement technique is usually performed by specialized people and requires a long time to extract the information of each sample, since many parameters need to be estimated resolving a non-convex optimization problem. Even experienced users may spend from tens of minutes to hours to resolve a single sample. In this work we present a new Matlab Toolbox used to quantify phases of a crystal using the Rietveld Refinement Technique specialized to In-Situ Samples. It is observed that since two consecutive samples present similar diffractograms then the estimated parameters of each sample should be similar, and then the optimal solution of one optimization problem is a good initialization parameter for the next one, resulting in faster time to resolve a sequence of diffractograms. The algorithm is implemented in Matlab and uses parallel programming capabilities to improve performance.

Keywords: XRD, Rieteveld, Numerical Method