SOLVING AND REFINING DIFFICULT STRUCTURES BY THE PROGRAM PACKAGE JANA2006

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The solution and refinement of crystal structures play a crucial role is characterization of new materials in very different branches of science dealing with crystalline solids. New experimental techniques, namely using of area detectors, make the procedure for describing of crystal structures much easier and more effective. Thus a standard crystal structure can be measured, solved and refined [1] within several hours. However, many other compounds cannot be characterized as standard ones as they are affected by twinning, modulations and disorder of different kinds. These effects not only complicate structure solution but in many cases they are strongly correlated with physical and chemical properties of the investigated materials. This fact is the main driving force for developing of the program package Jana2006 [2].

The system can be used to solve and refine regular, modulated and composite crystal structures against powder or single crystal data collected with X-ray laboratory diffractometers, synchrotron or neutron sources. The latest version allows so called joint-refinement in which several data sets can be combined together.

Probably the most common problem in crystal structure analysis is twinning in crystals. Several different examples including multiphase systems will be presented to show how to handle data and how to solve and refine such structures.

Modulated and composite crystals represent another category of complicated structures. The frequency of their occurrence has considerably grown up with increasing usage of modern CCD diffractometers, which give full information about the large portion of diffraction space. Thus detection of satellite reflections is simple as well as their integration, which is an option of the data processing software supplied with most diffractometers. Thus the way from experiment to the solution process is relatively straightforward.

The superspace theory, as developed by de Wolff, Janssen and Janner [3], is used to make a symmetry analysis which leads to the most probable superspace group. Recently developed technique called "charge flipping" [4] allows for *ab initio* solution of aperiodic structures and opens the field for non-specialists.

Modulations in the crystal, affecting various structural parameters (site occupancies, atomic positions and also atomic displacement parameters), can be usually described as a combination of continuous harmonic functions. There are cases, however, when the modulation has a discontinuous character, which cannot be sufficiently described with the continuous harmonic model. Discontinuity in modulation can be recognized already in the diffraction pattern by presence of strong higher order satellites and needs for efficient description special modulation functions of crenel or saw-tooth shape [5]. These functions have been introduced in Jana program and recently a new algorithm has been developed that facilitates their application.

The lecture will be made as an introduction to the Jana2006 workshop.

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