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New developments in structure analysis of powders using preferred orientation. Jürgen Grässlin[a], Lynne B. McCusker[a], Christian Baerlocher[a], Fabia Gozzo[b], Bernd Schmitt[b], Luca Lutterotti[c] , [a]*Laboratory of Crystallography, ETH Zurich, Switzerland,* [b]*Swiss Light Source, PSI, Villigen, Switzerland,* [c]*Department of Materials Engineering and Industrial Technology, University of Trento, Italy*
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In recent years, the reflection overlap problem, which is the primary hindrance to structure determination from powder diffraction data, has been addressed by exploiting preferred orientation. The changes in the diffraction pattern intensities as a function of sample orientation (tilt χ and rotation ϕ) that are observed for a textured sample in an X-ray beam, can be used to resolve the relative intensities of reflections that overlap in a conventional powder diffraction pattern. It has been shown that this principle can indeed be exploited to obtain more single-crystal-like data from a polycrystalline material [1]. The availability of a new version of the 1-dimensional Si-microstrip detector Mythen II on the Materials Science Beamline at SLS [2] and new features in the data analysis software *Maud* [3] have made it possible to improve the quality of extracted reflection intensities significantly. Textured samples of a zirconium phosphate framework material (ZrPOF-pyr) with a known crystal structure [4] and of an ammonium niobium silicate with an unknown structure were measured recently using the new experimental setup. The measurements consist of two steps. First, 302 orientations are measured quickly to determine the orientation of the crystallites in the sample. Then a few selected orientations are measured with better counting statistics for the intensity extraction. Data analysis using *Maud* also involves two steps. First, the intensities of a few non-overlapping reflections as a function of sample orientation (302 patterns) are used to generate pole figures (PFs) for these reflections and to determine the orientation distribution function (ODF). The ODF is then used to calculate the pole figure values for all reflections in the pattern, and these are applied in a joint refinement of the patterns collected with better counting statistics to obtain a single set of single-crystal-like reflection intensities. Analysis of the ZrPOF-pyr data shows that indeed more reliable intensities can be derived for reflections that overlap in a conventional powder diffraction pattern. However, the few orientations corresponding to main zone axes (e.g. *h00, 0k0, 00l, hh0, h0h, 0kk*), which were measured longer for intensity extraction, do not contain sufficient information to resolve all overlapping reflections. Consequently, a new strategy for selecting an optimized set of sample orientations for longer data collection has been developed. All PFs are calculated from the ODF, and from these, difference PFs are calculated for overlapping reflections. Those orientations that represent maxima in the PFs (i.e. above a given threshold value) and maximum differences for overlapping reflections (texture contrast) are selected. This new approach will be tested during our next experimental sessions in May and June 2010.

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[3]

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