in the range between 15 and 250°C reveals the occurrence of at least two partially dehydrated phases. From the evaluation of the collected patterns it can be concluded that the principal crystallographic differences between these two materials and the kanemite structure are due to a pronounced decrease of the lattice parameter perpendicular to the silicate layers (direction [010]).

An ab-initio structure determination by simulated annealing of the first dehydration product of kanemite with nominal composition $NaSi_2O_4(OH)*H_2O$ was successful. Whereas the silicate layers of the kanemite structure are retained almost unmodified, pronounced changes can be observed in the interlayer sheet containing the Na cations. The previously unknown phase adopts space group *Pbcn* as well. Lattice parameters at ambient conditions are as follows: a=4.888, b=15.332, c=7.203Å.

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Keywords: hydration, transition, silicate

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Data reduction of area detector measurements

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Time of flight Laue neutron diffraction a powerful tool to sample reciprocal space in a highly effective manner by collecting scattering data of a wide wavelength band simultaneously. Efficiency is further improved by highly pixelated large area detector coverage with excellent time resolution. This produces a wealth of data with every sample setting. Consequently large files of raw data need to be handled for data visualization, raw data corrections for incident spectrum variations, detector efficiency, background, sample effects and contributions. Furthermore, the software needs to be easy to use by new and experienced users alike. An increasingly important part of data collection is a strategy software that allows to tailor the measurement time and conditions to the sample symmetry and resolution requirements.

Examples of new and improved software developed and implemented at the TOPAZ single crystal diffractometer at the Oak Ridge National Laboratory will be presented. CrystalPlan helps define the measurement strategy and ISAW EV analyzes live neutron event data on the fly.

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Keywords: data reduction, tof neutron single crystal diffraction, area detectors

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Applications of area detectors to texture measurements

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The use of 2-dimensional detectors in Quantitative Texture Analysis (QTA) has been pointed out long ago mainly in order to reduce acquisition times, both at x-ray and neutron diffraction instruments [1], [2]. Indeed, the necessity to acquire several pole figures to correctly refine the orientation distribution functions makes indispensable their simultaneous acquisitions. Since typically 1000 pole figure points have to be measured, using point detectors creates very often acquisition times over several days, a dramatic drawback particularly at scarce neutron beam times. Historically, the development and use of linear, then curved position sensitive (CPS) detectors helped in reducing the acquisition to several hours [3], even at neutron steady state reactors [4]. Furthermore, it opened the way to the treatment of the whole pattern simultaneously including QTA information and more, a procedure nowadays called Combined Analysis [5]. CCD cameras and image plate systems further offer fast QTA analysis with no loss in the capability of full-profiling the patterns. Curvature of image plate detectors can be operated to create cylindrical solid angles for x-ray instruments, and at neutron lines, shaping parts of cylinders with individual detector plates or wires has been recently developed. For all these 2D-detectors, the aim to reduce the number of sample orientations to be measured has been at least partly achieved.

We will illustrate the main scheme used to construct pole figures and calculate ODFs from area patterns, giving some exemples of the use of Combined Analysis, which includes the determination of structure, phase and microstructure also. One of the last developments allowed by the use of Curved Area PSDs is the determination of Magnetic Quantitative Texture Analysis [6], which we will illustrate also.

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Keywords: texture analysis, area detectors

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Modern trends in area detectors for single-crystal neutron diffraction

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Neutron single-crystal diffraction is the tool of choice to determine the accurate positions of hydrogen atoms in solids and the arrangements of magnetic moments. In some cases, hydrogen positions can be inferred from the positions of the other atoms, but if hydrogen bonding occurs, this is not reliable. Since neutron fluxes are typically low compared to those available at synchrotron X-ray facilities, there has been a major effort to maximize the exploitation of the available scattering data. Much emphasis has been placed in particular on the provision of large area detector systems. The D19 diffractometer at the ILL which has been equipped with a 120º x 32º multiwire proportional counter since 2007 provides greatly improved data quality for crystallographic work in structural chemistry, physics and the biosciences.

Another slightly less precise single-crystal diffraction method is the Laue method where the crystal is illuminated by a broad spectrum of wavelengths, and a large area of neutron-sensitive image plates (VIVALDI at the ILL, KOALA at ANSTO) is used to measure

the diffraction spots. Recently a CCD-based Laue diffractometer, CYCLOPS at the ILL, has been constructed which should provide a unique possibility for real-time exploration of reciprocal space and rapid data collection through phase transitions due to its fast readout system. Finally the Very Intense Polarized neutron diffractometer (VIP) at the LLB which combines a large area detector and a hot polarized neutron beam shows an unprecedented efficiency in the measurement of spin densities. In this talk a review of neutron instruments with area detectors, the receipts of the data reduction and trends of their development will be given.

Keywords: neutron, diffraction , monocristal

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Accurate data analysis for the koala and VIVALDI neutron Laue diffractometers

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The Koala (ANSTO) and VIVALDI (ILL) instruments are designed for relatively fast data collections using a minimal sample volume. This is possible due to large image-plates with ~3π steradian coverage and the use of unfiltered "white" neutron beams from high flux reactors. The historical difficulty with these instruments has been the processing of the multi-wavelength diffraction data into accurate corrected intensities useable by structure refinement programs. The Laue1234 software suite has been developed to address this problem and to allow accurate structural refinements[1], [2] for the Koala instrument. The software has now been extended to address the problem of the complex wavelength distribution found at the VIVALDI instrument.

Laue1234 runs on a conventional PC using new software and modified versions of the existing neutron Laue diffraction suite: lauegen and its associated programs [3] and argonne_boxes [4]. The peak integration software argonne_boxes has been modified to extend its applicability to lower peak intensities and to rigorously handle the propagation of errors. The algorithms were validated using simulated and observed data which has lead to the discovery of a new statistical effect due to detector crosstalk [5].

The program Laue4 was written to correct and merge the multi-wavelength peak intensities. The software corrects the intensities for beam variations between exposures, the incident beam wavelength distribution, wavelength dependent and time dependent efficiency corrections, secondary extinction and sample absorption corrections, and to a limited extent the effect of λ/2 peak overlap. The majority of these corrections are performed using a least squares approach where parameters in the correction model are adjusted to minimize a measure related to the R_{merge} of equivalent reflections. A non-parametric approach is used for the particularly complex and changeable wavelength distribution of the VIVALDI instrument. This approach optimizes the distribution using the observed intensities of equivalent reflections against a measure of the local complexity of the distribution compared to *a priori* distributions.

Of equal importance to the actual corrections is the incorporation of intensity uncertainties due to the limitation of the correction models. Without reasonable estimates of these systematic uncertainties the merging of intensities is dominated by long wavelength data where the counting statistics are best but the systematic errors due to extinction and absorption are worst.

Examples from Koala and VIVALDI will be presented to illustrate the capability of the software for refining structures with R_1 factors in the 2 to 5% range.

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Diffraction data quality analysis of EVAL15 integration

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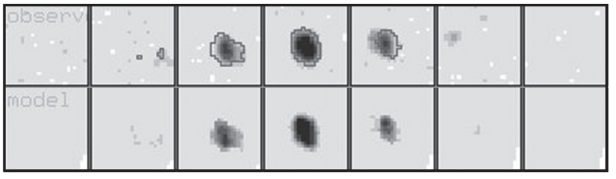
EVAL15 is a diffraction data integration method [1] that is implemented as part of the EVAL software package [2]. It simulates the complete diffraction process on the basis of only a few physical model parameters. The resultant reflection profiles are subsequently used in profile fitting of reflection intensities. This versatile method can cope with a range of complicated integration problems, e.g. of crystals with anisotropic mosaic spread or lattice distortion, overlapping reflections due to long axes or multiple lattices and aperiodic (incommensurate or composite) crystals. Moreover, the EVAL package can handle many types of goniometers, a large range of detectors, and various types of X-ray or neutron sources.

The general opinion on profile fitting is that the standard deviation of weak data is reduced, whereas for strong data it presents a disadvantage over summation integration. Methods that use ab initio generated models need a comprehensive description of the diffraction process. In this contribution we will describe the procedure for generating high quality profiles.



A detailed analysis of the data quality obtained by EVAL15 is given. The analysis comprises data sets of (metal)-organic compounds with a large dynamic range of reflected intensities, crystals with packing disorder and high resolution data. We use several quality indicators based on the statistical analysis of the data and on aspects of the refined structure.

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Keywords: data_quality, EVAL15, intensity_integration

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Modelling thermal scattering and solving structures using Z-contrast imaging

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