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

MS.68.4

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 $\lambda/2$ 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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Keywords: neutron_diffraction, data_analysis, laue_method

## MS.68.5

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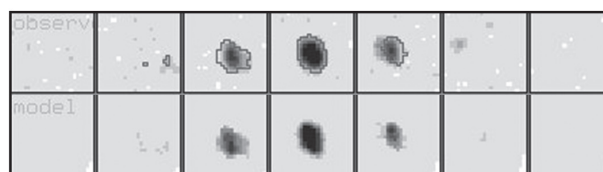
A detailed analysis of the data quality obtained by EVAL15 is given. The analysis comprises data sets of (metal)-organic compounds with a large dynamic range of reflected intensities, crystals with packing disorder and high resolution data. We use several quality indicators based on the statistical analysis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

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Keywords: data_quality, EVAL15, intensity_integration

MS.69.1

Modelling thermal scattering and solving structures using Z-contrast imaging

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Thermal scattering (due to phonon excitation) plays an important role in transmission electron microscopy and convergent-beam electron diffraction patterns. It makes the dominant contribution to Z-contrast (high-angle annular dark field) measurements in scanning transmission electron microscopy. Recent experimental advances have put Z-contrast imaging on an absolute scale [1] and structures can be solved and the atoms in a column counted to an accuracy of ±1 atom [2]. The results in Ref. [2] relied on simulations of thermal scattering using the frozen phonon model. Within this semi-classical model "the electron sees a snapshot of the atom frozen midvibration" [3]. Each electron "sees" a different configuration, and the contributions of different electrons are summed incoherently in the detector plane. Furthermore the frozen phonon model does not contain within its conceptual framework the momentum or energy transfer one would normally associate with inelastic scattering (in this case phonon excitation).

The correct model for thermal scattering is based on many-body quantum mechanics, as expressed by the equations of Yoshioka [3], with phonon excitation treated as a quantum excitation of the crystal during which the incident electron is inelastically scattered. We use an approximation similar to the Born-Oppenheimer-type approximation used in molecular physics for the many body wave function to derive a model for electron diffraction and imaging which can explicitly calculate the elastic component and the many inelastic components of the scattered electron wave. Our model predicts the scattered probability distributions for a single electron, including multiple elastic and inelastic phonon scattering to all orders. This is an advantage over other approaches based on the Yoshioka formalism in which, to allow for tractable calculations, the single inelastic scattering approximation is made.

Our model leads to a scattering intensity which is numerically similar to that calculated using the frozen phonon model and we provide a perspective on why this is the case, albeit that the two models have quite different conceptual underpinnings. Therefore our model underpins the integrity of the recent progress in the use of quantitative Z-contrast imaging to solve structures.

We will present several examples of how (crystalline) structures can be solved using quantitative Z-contrast imaging.

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Keywords: modelling, thermal, scattering

MS.69.2

SrTiO$_3$ surface structures from diffraction & DFT: Homologous Series & Glasses

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Understanding the structure of oxide structures has proved difficult. Part of this is the technical direct methods of how to handle datasets with relatively large numbers of missing reflections (up to 50%), where the missing reflections may be strongest ones but overlap with bulk reflections which are orders of magnitude larger. A second issue is how to solve a structure when the exact number of atoms involved is not known. Lastly, there is the question of how to validate a structure, which in most cases has involved using Density Functional calculations where there can be very severe errors in the energies due to inaccuracies in the exchange-correlation potentials which is a particularly hard problem for oxides.

A very recent advance has been the realization that oxide surfaces are not that different from inorganic bulk oxides. In particular, it has turned out that the classic method of Bond Valence Sums in fact works remarkably well, and can have predictive power in clarifying what are reasonable or unreasonable direct methods solutions [1], [2]. This can be combined with better methods of calculating the energetics of oxide surfaces using more advanced methods with hybrid exact-exchange functionals (e.g. [3] and references therein) which are rather important as this more correctly represents the ionicity of the system; simpler functionals make oxides much too covalent.

Combining these, we have started to make some inroads into oxide surface structures. One specific case is the determination of a full homologous series of nx1 reconstructions on the SrTiO$_3$ (110) surface [1], in essence a monolayer equivalent of homologous series in bulk oxides. A second is the determination of a complicated ($\sqrt{13} \times \sqrt{13}$) R33.7° reconstruction on the SrTiO$_3$ (001) surface [4] which is a prototype structure of a series of glass-like tilings, again very similar to bulk glasses at the monolayer level. In all cases the bond valence sums at the surface are good, and the DFT energetics indicate that these are low-energy stable structures.

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Keywords: Surfaces, DFT, TED

MS.69.3

JANA2006 as a unique tool to refine nuclear and/or magnetic structures using ToF data

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Powgen represents a departure from previous designs for a time-of-flight powder diffractometer at a spallation neutron source and may be considered as a third-generation design. The geometric design of the instrument allows for all detected scattered neutrons to be focused onto a single diffraction profile yielding high count rate while preserving good resolution $\Delta d/d = 0.0015$ at a $d = 1$ Å. The settings differ in the center wavelength, λ_{CW}, of neutron band chosen, each band being of ~ 1 Å width with Powgen chopper system operating at 60Hz. Full diffraction profiles need to be fitted using a peak shape based on convolution of back-to-back exponentials with a pseudo-Voigt. The use of a cold cryogenic moderator (and to lesser extent supermirror guide) yields significant differences in the d-space dependencies of the exponential rise (α) and decay (β) terms as well as the correction needed for reflection position, compared to the standard dependency functions modeled in GSAS [2]. Therefore, alternate functions for α, β, and