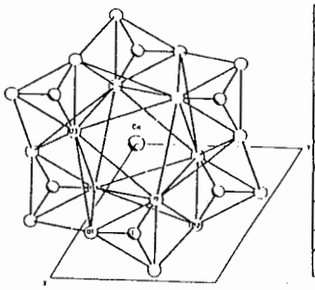

Fig. 1

Fig. 2

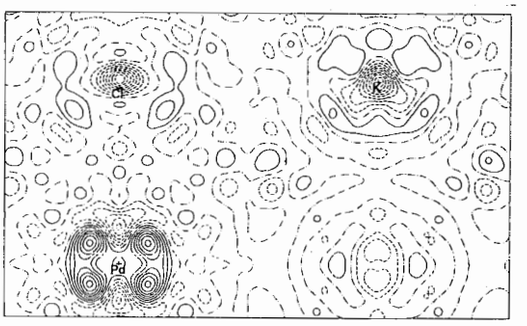

Figure 2: $\Delta\rho$ in [110] plane of K_2PdCl_6. Borders 6.6Åx 4.0Å, contours 0.2eÅ^{-3}.

PS-14.02.16 ACCURATE ELECTRON DENSITY IMAGING OF K_2MH_6 COMPOUNDS. By J. R. Hester, Crystallography Centre, University of Western Australia, Nedlands 6009, Australia. Accurate electron densities of K_2SiF_6 and K_2PdCl_6 have been obtained using synchrotron radiation, providing detailed information on the atom-atom interactions in these structures. X-ray data was collected from crystals on conventional sources and at the Photon Factory synchrotron. Although the electron densities for the two compounds are topographically similar, they contain unexpected features. In the K_2SiF_6 maps (Fig 1) the density is depleted within the SiF_6 moiety. The density near the K is strongly depleted along the K-Si vectors. The density is also depleted along the non-bonded K-K and F-F vectors. Electrons accumulate in regions close to the K atom and in a broad interatomic "sea", delineated by the zero contour in Fig 1. The $\Delta\rho$ topography near K is similar in the room temperature K_2PdCl_6 map. If the anomalous features around K in each case are due to anharmonic thermal vibrations, these should diminish at low temperatures. A low temperature K_2SiF_6 data collection, while quite noisy, shows evidence of the persistence of this feature, as also does the relevant section (Fig 2) based on low temperature K_2PdCl_6 data of Takazawa, H., Ohba, S. & Saito, Y.[(1988) *Acta Cryst.*, B**44**, 580-585]. The K electron density indicates that exchange effects between second-nearest neighbours are important in this structure. Overlap of the atoms in the SiF_6 moiety forces electrons out of the volume surrounding the nuclei into interatomic regions. The effects of K-K and Cl-Cl overlap are predictably weaker in the less tightly packed K_2PdCl_6 structure. A calculation of atomic charges based on projection of $\Delta\rho$ onto atomic density basis functions partitions the two difference densities quite differently, so that the signs of the K_2PdCl_6 charges agree with their formal values whereas the signs of the K_2SiF_6 charges are reversed. This is mainly due to the changes in atomic radii partitioning the interatomic sea differently. It is clear that the same physical processes are responsible for both electron distributions.

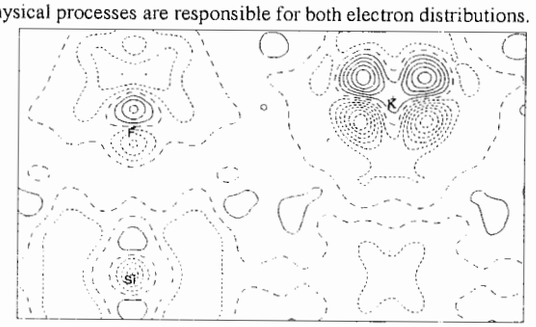

Figure 1: $\Delta\rho$ in [110] plane of K_2SiF_6. Borders 5.6Åx 3.3Å, contours 0.1eÅ^{-3}. Negative - dotted, positive - solid, zero - dashed.

14.03 - X-ray Physics, Diffraction and Absorption

PS-14.03.01 X-RAY POLARIZATION PHENOMENA IN PERFECT AND IMPERFECT CRYSTALS.
By H.R.Höche, C.Eisenschmidt, H. Höfer and W.Leitenberger, Fachbereich Physik, Martin-Luther-University Halle, Germany

It can be seen in highly resolved X-ray diffraction experiments that the polarization state of linearly polarized X-radiation is changed.
The polarization state is not changed by BRAGG-case diffraction in perfect crystals if the polarization plane is parallel or perpendicular to the diffraction plane. In all other relations between diffraction plane and polarization plane the polarization of the diffracted beam is dependent on the angular position in the interference range (rocking curve).A systematical polarization mixing was found in experiments with a Si crystal containing a high density of dislocations which were produced by a high temperature deformation.
The polarization state is unchanged also by LAUE-case diffraction in perfect crystals if the polarization plane of the incident beam is parallel or perpendicular to the diffraction plane. In all other relations of these two planes four X-ray waves are excited inside the perfect crystal. In general the diffracted beam is elliptically polarized, it is caused by the different absorption coefficients (BORRMANN-effect). The polarization state depends on the crystal thickness and on the angular position in the interference range (BORRMANN-fan). The experiments were done with synchrotron radiation at HASYLAB (DESY Hamburg). The experimental results are in good agreement with the dynamical theory of X-ray diffraction. There are a phase difference between the σ – and π – polarized waves in the transmitted beam also. Especially in the centre of the BORRMANN-fan this phase difference can be used for the construction of a polarization optical phase shifter. Theoretical and first experimental results will be presented in detail.

This work was supported by the BMFT under the project Nr. 05 5NHAAB 0.

PS-14.03.02 HIGH RESOLUTION POWDER DIFFRACTOMETRY WITH A BENT PERFECT CRYSTAL MONOCHROMATORS.
By P. Mikula*, M. Vrána, P. Lukáš, Nucl. Phys. Inst., 250 68 Řež, Czech Republic, V. Wagner, PTB, 3300 Braunschweig, Germany and B. Alefeld, KFA, 5170 Jülich, Germany.

We are presenting a highly efficient diffraction arrangement with a cylindrically bent perfect crystal monochromator suitable particular-

ly for the investigation of individual powder lines and their neighbourhood. The arrangement fully employs focusing in real space and the possibility of a control of strong correlation between the scattering angle φ and the neutron wavelength in the momentum space without any use of Soller collimators (Mikula, Wagner, Lukáš & Scherm, 1992; Lukáš, Vrána, Mikula, & Kulda, 1992). In this way, with a properly adjusted monochromator bending radius, the reflected neutrons corresponding to a chosen powder diffraction line at an angle φ are quasi-parallel in the scattering plane. Then the maximum flux of neutrons registered by a detector corresponds to the minimum FWHM of their angular distribution. To avoid a rather large angular uncertainty given by the divergence of a Soller collimator placed in front of the detector, for determining the position of the peak and its profile a spatially high resolution position sensitive detector (PSD) may be advantageously used. The FWHM of about 10^{-3} rad, and the high luminosity of the diffraction geometry strengthened by the use of PSD predict such an instrument for the employment in high resolution powder diffraction experiments e.g. for the investigation of line broadening in the case of plastically deformed crystals, the analysis of peak shifts (with a precision better than 10^{-4} rad) for the studies of residual stresses (Kulda, Mikula, Lukáš & Kocsis, 1992), time and temperature phase studies and particular inelastic scattering investigations. The luminosity and resolution of the diffraction arrangement which uses the bent perfect crystal monochromator will be compared with the arrangement using a conventional mosaic monochromator.

Kulda, J., Mikula, P., Lukáš, P. & Kocsis, M. (1992). *Physica* B **180 & 181**, 1041-1043.
Lukáš, P., Vrána, M., Mikula, P. & Kulda, J. In *Proceedings of SPIE's 1992 International Symposium on Optical Applied Science and Engineering*, 22-24 July, San Diego, Conference *Neutron Optical Devices and Applications*, editted by C.F. Majkrzak & J.L. Wood, **1738**, p. 438-446.
Mikula, P., Wagner, V., Lukáš, P. & Scherm, R. (1992). *Physica* B **180 & 181**, 981-983.

PS-14.03.03 COMPARISON OF THE STATISTICAL DYNAMICAL THEORIES WITH MEASUREMENT OF INTEGRATED INTENSITY. By T. Takama, Department of Applied Physics, Faculty of Engineering, Hokkaido University, Japan.

Since N. Kato (NK) developed the statistical dynamical theory of diffraction (Acta Cryst. A36, 1980, 763-778), much effort has been devoted to improving and testing it. It is expressed in terms of the static Debye-Waller factor E and two correlation lengths for the phase factor τ and the wave-field amplitude Γ. Recently, the theory was reformulated by Becker and Al Haddad (BA) (Acta Cryst. A48, 1992, 121-134). The main difference exists in treating Γ which strongly affects the incoherent scattering. NK suggested that Γ is close to the extinction distance. On the other hand, BA showed theoretically that Γ should be of the same order of magnitude as τ. The two theories were tested (Schneider, Bouchard, Graf & Nagasawa, Acta Cryst. A48, 1992, 804-819) experimentally by comparing them with the integrating reflection power of γ-ray measured for heat-treated Si as a function of crystal thickness. They concluded that the BA theory predicts excellently their data and the E parameter alone characterizes substantially the crystal imperfection.

In the present study, an attempt was made to verify which theory describes better the diffraction from specimens with various degree of imperfection. In order to introduce the randomly distributed micro defects, the parallel-sided Cz-Si crystals were heated systematically at 1223 K for different duration from 25 to 145 hr. The integrated intensities on the Laue case were successively measured for four reflections as a function of X-ray wavelength by the energy-dispersive diffraction method. The measured profiles showed remarkably the increase in the intensity as well as the decrease in both the period and the amplitude of the Pendellösung beats with increasing duration. The NK theory described the profiles very well assuming Γ to be constant for a reflection of a crystal (Takama , Harima & Sato, Acta Cryst. A46, 1990, C412). The BA theory taking account of the parameter E alone also fitted the profiles for relatively short heating duration as concluded by Schneider et al. For the specimens heated for longer duration, however, the comparison showed a poor agreement although three parameters were taken into consideration. This means that the NK theory with constant Γ is better to interpret the diffraction in the present development stage of the theory.

PS-14.03.04 ANGULAR MEASUREMENTS WITH X-RAY INTERFEROMETRY. By P.Becker*, J.Stümpel and D.Windisch, Physikalisch-Technische Bundesanstalt, W-3300 Braunschweig, Germany.

The angular dependence of the reflectivity in a skew-symmetric X-ray interferometer with the axis of rotation between two pairs of the reflecting wafers has been investigated experimentally and theoretically. Rapid oscillations with a periodicity of almost two milliseconds of angle have been observed. The period depends on the geometry and the lattice spacing of the silicon crystal. The amplitudes of the angular oscillations are strongly affected by "Pendellösung" interference phenomena. As an example of applications an optical autocollimator is calibrated by the X-ray interferometer.

A triple-Laue X-ray interferometer capable of angular measurements in the range below a second of angle was first proposed and put into operation by Becker & Bonse (1974). They used two pairs of lamellae in a skew-symmetric arrangement with an axis of rotation between them and measured the intensity oscillating in the outgoing X-ray beams taking into account the alignment parameters between the separate parts of the interferometer. They found as a result of the geometrical treatment, that the periodicity of the oscillations depends on the angle of rotation and the lamellae distance in one subunit, lamellae thickness and the lattice spacing. Their calculations, however, did not include information about the angular range and amplitude of the oscillations in the reflectivity of the interferometer.