MS-02.03.36 DIRECT METHODS FOR POWDER DIFFRACTION. By Henrik Sverdrup, Jouk Jauane, Paval Capkova, Wieslaw Lasocho, David Rafaia and Reuc Pech, Laboratory for Crystallography, University of Amsterdam, Nieuwe Achtergracht 166, Amsterdam.

The structure determination from powder diffraction data by an 'ab initio' method like Direct Methods is greatly facilitated when as many unique intensities as possible are known accurately. However, powder patterns generally show a range in intensities from 1 to 1000, whereas single-crystal data are found in a much wider range. This overlap causes complications and, moreover, texture or preferred orientation prevents the intensities to be correct. Our program POWElM employs for the reconstruction of intensities in the last step a procedure in the first step the complete powder diagram is used in a Rietveld-Win procedure applied to the complete diagram (J. Appl. Cryst. 1992, 25, 231), which proves to be an improvement over Pawley's method (J. Appl. Cryst. 1981, 14, 357). By minimizing the least-squares sum of the intensities and background exactly, the fitting become independent of the initial intensity and background values. Our results show that the intensities refined with this new technique deviate less from the true intensity values than those from the original Pawley fitting. Any curve-fitting procedure will never be able to unravel two peaks at the same or nearly the same position. In order to avoid this problem, several relations from Direct Methods have been employed to predict which overlapping reflections are strong and which are weak. This information and the sum intensity of overlapping peaks, which is determined correctly by the fitting procedure, can be used to redistribute the intensities in each case, the overlapping peaks (J. Appl. Cryst. 1992, 25, 231). In this way we get a vast number of intensities that are determined accurately, each of them getting a reliability-index for its accuracy, which is being used in the successive phasing process (Z. Kristall, in press). At present fine-tuning of the Direct method is performed in theoretical and practical work is carried out to include texture correction in the program system. The texture measurements are done with a prototype of a diffractometer, developed in the Philips Research Laboratories (Adv. X-Ray Anal. Vol. 31 (1988), Kluwer, A. C.) Also, structure determinations using the method will be presented.

PS-02.03.07 ELECTRON DISTRIBUTION IN GERMANIUM BY MAXIMUM ENTROPY METHOD. By H. Nishihara, S. Ishihara, S. Nishimura and K. Fukagawa, Department of Materials Science, University of Tsukuba, Ibaraki, Japan.

A detailed electron density distribution in germanium was examined from powder X-ray diffraction data. Ultrafine particles of Ge with average particle size less than 1000 were synthesized by a 'hydrogen plasma-thermal' reaction method (K. Nishihara, S. Ishihara, S. Nishinasa and M. Kaneko, Jpn. J. Appl. Phys. 1982, 21, 1202-1209). Diffraction intensities were measured by a step scanning with the sampling interval of 0.02° in 2θ and accumulation time was 10 seconds for each step. The scan range in 2θ was from 40° to 165° of CuKα radiation with tube voltage and current of 50kV and 200mA. Intensities were estimated by the computer program WPDR (Typo, J. Appl. Phys. 1981, 440-441). The integrated intensities were converted to the structure factors by the ordinary least-squares refinement. By this analysis we obtained the values of 11 independent structure factors and one combined structure factor which was a pair of 303 and 511. The electron density distribution map was drawn by the maximum entropy method (M. Sakata and M. Sato, Acta Cryst. 1989, A45, 243-267). A special feature for a diamond structure reappears in the map for (110) plane. However, the bonding electrons were not clearly observed because of the limited number of reflections measured. In order to improve the map, an experiment using synchrotron radiation is in progress. Results will be presented at the meeting.

PS-02.03.08 CHARGE-DENSITY ANALYSIS BY MEANS OF GAMMA-RAY DIFFRACTION: NiF2, FeF2. By A. Palmier and W. Jauch, Hahn-Meitner-Institut Berlin, Germany.

NiF2 and FeF2: Measurements of data sets up to 1.3 Å-1 at 295 and 15 K, contrast to MnF2, combination with neutron diffraction from the identical samples gives no indication for a polarization of the F-atoms in the antiferromagnetic phase: the population of 5d-orbitals, derived from multipole-models agrees with the expected crystal-field splitting in an octahedral environment.

StTlO3: Data collection up to 1.6 Å-1 at room temperature, at 5 K above the 167 K structural transition and at about 50 K below the corresponding peaks (J. Appl. Cryst. 1992, 25, 231). In this way we get a vast number of intensities that are determined accurately, each of them getting a reliability-index for its accuracy, which is being used in the successive phasing process (Z. Kristall, in press). At present fine-tuning of the Direct method is performed in the theoretical and practical work is carried out to include texture correction in the program system. The texture measurements are done with a prototype of a diffractometer, developed in the Philips Research Laboratories (Adv. X-Ray Anal. Vol. 31 (1988), Kluwer, A. C.) Also, structure determinations using the method will be presented.

PS-02.03.09 DIRECT INVESTIGATION OF ATOMIC VIBRATIONS OF Beryl BY THE MAXIMUM ENTROPY METHOD. By M. Ihara, T. Sakai, Department of Applied Physics, Nagoya University, Nagoya, 464-01 Japan, S. Kuma, Department of Physics, Science University of Tokyo, Tokyo, Chiba 278 Japan and F.K. Larsen, Department of Chemistry, Aarhus University, DK-8000 Aarhus C, Denmark.

The Maximum Entropy Method (MEM) applied to neutron diffraction data directly yields the nuclear density distribution, which is equivalent to the thermal smearing function. The MEM procedure thus enables us to form maps showing the atomic thermal vibrational features in real space without using a structural model. The purpose of this study is to examine the capability of the MEM for the direct investigation of thermal vibrations and its choice as a test example.

Mean-square atomic displacement and antisymmetric atomic vibrations in Be at room temperature have been determined from short-range neutron data by conventional analysis (G. Larsen, F.K. Lehmann, T. N. and M. H. O. 1988, Acta Cryst. A45, 139-162). The same data were treated by the MEM in the present study.

The resulting density distribution at the pressed 'nuclear site in the basal plane, shown in Fig. 1(a) and 1(b), respectively. In Fig.1(a) a characteristic triangular feature is clearly visible. We interpret this as being caused by 3rd order inharmonic vibrations in the basal plane. In Fig.1(b) the nuclear density shows significant deviation from the expected oblate shape from harmonic vibrations. It interpret this feature as indicating a significant quartic contribution to the potential.