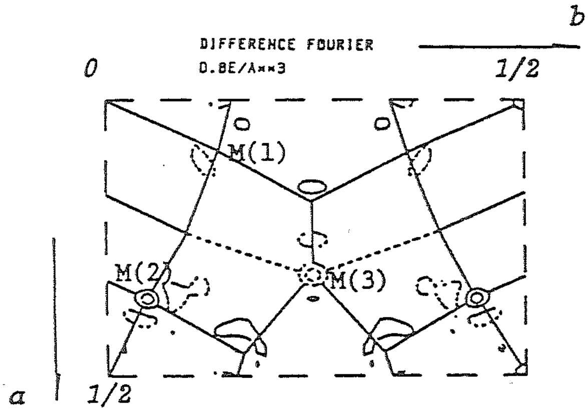
scattering factor of Pb and Bi atom at $\sin\theta/\lambda = 0.0$ is enlarged to 3.2. A set of 237 intensity of *hk0* reflections(including 37 standard reflections) was collected with the ω-2θ step scan mode, using a four-circle diffractometer set up at the BL-10A station. After correction of dead time of detector system, Lorentz factor and absorption, all the intensities were normalized by those of standard reflections measured every ten reflections during data collection.

Several cycles of least-square refinement gave R-factor 0.06 under the condition of referring to the already determined structure as the starting model and employing the averaged scattering factors of Pb and Bi for both metal atoms. The difference among weights of metal sites in d-Fourier map seems to reveal features on the site occupancy of each metal site. A more quantitative study of the site occupancy is now in progress.



08.4–37 FORMATION OF α-Al_2O_3 BY DEHYDRATION OF DIASPORE. By M. Ohmasa[1], J. Ryuta[1], K. Terada[1], N. Yamada[1], S. Sueno[1], H. Nakazawa[2], K. Tsukimura[3] and S. Sasaki[4], 1: Univ. of Tsukuba, 2: Nat. Inst. for Research in Inorganic Materials, 3: Geol. Survey, 4: Nat. Lab. for High Energy Physics, Japan.

Diaspore(α-AlOOH) transforms to α-Al_2O_3 at about 500°C and the change is expressed as follows: $2\alpha\text{-AlOOH} \rightarrow \alpha\text{-}Al_2O_3 + H_2O$. Lima-de-Faria (Z. Kristallogr. (1963) 119, 176) studied the dehydration and found that a topotactic relation exists between diaspore and α-Al_2O_3 and reflections of the latter are accompanied by satellites. He concluded that the satellites could be ascribed to a modulation of amplitude, but recently his conclusion was opposed (Watari, J. Sol. Stat. Chem. (1979) 29, 417). The present studies have been undertaken to check the validity of both results and to clarify the mechanism of the dehydration.

Good single crystals from Shokozan, Hiroshima, Japan were selected for experiments. The process of the dehydration was observed by two different methods. One of them was a quenching method; that is, the specimen were heated at definite temperatures for various periods and then quenched in air. The specimens were tested by X-ray diffraction. The temperatures selected were 450°C, 500°C and 550°C, and the period ranged from 15 minutes to 24 hours. The second was a direct observation of the change at elevated temperatures by X-ray diffraction with SR beam in Nat. Lab. for High Energy Physics.

The unit cell of diaspore is orthorhombic Pbnm with dimensions a=4.425, b=9.380, c=2.844Å and that of α-Al_2O_3 is rhombohedral $R\bar{3}c$ with a= 4.781 and c=13.032Å. Topotaxy was confirmed between diaspore and α-Al_2O_3: [100]s//[001]p and [001]s//[$1\bar{1}0$]p where s and p indicate the starting material and the product, respectively. The transformation progresses gradually. The satellites come up in the early stage and their feature changes slightly when the samples are heated at different temperatures. Streaks are observed between satellites and main reflections. Their intensity is affected by the temperature and the period of heating. The following features are also noted: (1) the satellites are observed along the c* of α-Al_2O_3 and the period of modulation derived is about three times of the c, and (2) satellites of higher order are not observed and the intensity of outer satellites are slightly stronger than the inner one. Since satellites were also noticed by direct observation with SR beam, the modulation should not be formed by quenching. The above features ascribe the modulation to a periodic distribution of Al-deficient layers along the c. The spots of the products, being broad, become sharper when the specimens are heated longer and with higher temperature. This suggests that nucleation and growth play important roles in the formation of α-Al_2O_3.

08.4–38 PHASE TRANSITION AND ORDERING IN LEUCITE. By T. Grögel, H. Boysen and F. Frey, Institut f. Kristallographie d. Universität, München, FRG.

Leucite ($KAlSi_2O_6$) is known to undergo a phase transition from a cubic high temperature form (Ia3d) to a tetragonal low temperature form ($I4_1/a$). Quoted transition temperatures T_c range from ~870 K to ~950 K, where single crystal X-ray investigations revealed only one transition point (Peacor, Z.Krist.(1967) 127, 213), while DTA measurements seem to indicate an additional intermediate phase (Faust, Schweiz.Min.Pet.Mit.(1963) 43,165). The transition is mainly connected with a rearrangement of the $(Si,Al)O_4$-terahedra, whereas a suggested Si-Al ordering on crystallographical different positions could not be detected with X-rays (Mazzi,Galli,Gottardi, Am.Mineral.(1967) 61,108). To clarify this latter point a neutron powder study (taking advantage of the larger Si/Al-scattering lengths ratio for neutrons and the absence of any twinning problems being serious in single crystals) on natural leucite has been performed at 5 different temperatures between room temperature and 1073 K together with supporting single crystal X-ray measurements. Using the Rietveld refinement program (final structural parameters will be given) the neutron data clearly revealed a partial Si-Al ordering of the starting material at r.t., which becomes complete after heat treatment at higher temperatures and totally disordered again in the cubic phase (requested by symmetry). Moreover, the results indicate