PS11.10.02 PENDELLÖSUNG EXPERIMENT OF SILICON AT LOW TEMPERATURES. T. Eto, H. Naruoka, S. Nagao, Z. Lu, Y. Soejima, A. Okazaki, Department of Phisics, Kyushu University, Fukuoka 812-81,Japan

In connection with anomalous behavior at low temperature of the silicon lattice spacing in particular that of (444), the structure factors of 111, 220 and 004 have been examined as a function of temperature. It is found that the structure factor shows no anomaly in a temperature range 17-300 K, while the Debye-Waller factor shows anisotropic temperature dependence. Measurements were made by means of X-ray diffraction according to the Pendellösung fringes method applied to specimens in a form of a thin plate. Descriptions are given of the experimental technique to keep the specimen at low temperatures free from strains that are mainly due to the difference of thermal expansion between the specimen and its holder. The characterization of the long-range strain field is given on the basis of the method in the literature. It is also found that strains introduced at low temperatures can be relieved at the lowest temperature; this phenomenon of a kind of aging is consistent with the observation in the previous experiment on the lattice spacing by means of high-angle double-crystal X-ray diffractometry and the Bond method. The procedure of the analysis of the fringe pattern is also examined, and the precision of the results is discussed. Finally, values of the structure factor at room temperature are compared with those in the literature, and a discussion is given of the accuracy of them.

PS11.10.03 STRUCTURAL RELATIONSHIPS IN KH_2PO_4-TYPE COMPOUNDS. Jürgen Glinnemann, Matthias Becker, Theo Hahn, Gernot Heger. Institut für Kristallographie, RWTH Aachen, D-52056 Aachen, Germany.

The large number of seemingly unrelated crystal structures of compounds with formula type $A(H,D)_2XO_4$ with A = Li, Na, K, Rb, Cs, Tl, NH_4 and X = P, As can be grouped into five structure families in the sense of Megaw (1973). Each family can be derived from one of the following five basic structure types: NaCl, CsCl, NaTl, PtS, and a shear variant of CsCl with coordination number 7. These structure types form the head entries for each family tree. This derivation consists of the following procedure:

- Degradation of the point symmetry and increase of the unit cells of the ideal AB structures by stepwise application of group-subgroup relations;
- replacement of the B atoms by oriented XO_4 tetrahedra;
- insertion of positions for the H and D atoms.

This procedure permits to place all known crystal structures of the $A(H,D)_2XO_4$ compounds into a hierarchical order.

The distribution of the individual compounds and polymorphs over and within the families is rather heterogeneous. The behaviour of KD_2PO_4 and of $Rb(H,D)_2PO_4$ is exceptional as their polymorphs occur in two different families: KD_2PO_4 occurs in the families derived from NaTl and NaCl, the rubidium salts in the families derived from NaTl and the shear variant of CsCl.

The five family trees will be presented and their geometrical relations discussed.

Megaw, H. D. (1973). *Crystal structures: a working approach*, ch. 12, pp. 282-340. Philadelphia: Saunders.

PS11.10.04 STRESS RELAXATION IN SILICON AT LOW TEMPERATURES. A. Kohno, Z. Lu, Y. Soejima, A. Okazaki, Department of Physics, Kyushu University, Fukuoka 812-81, Japan

The effect of uniaxial external stresses on the lattice spacing of silicon (444) has been examined in a temperature range 25-305 K by means of high-angle double-crystal X-ray diffractometry (HADOX); the relative precision of the lattice spacing $\Delta\, d/d$ is $\pm 3\times10^{-6}$. It is found that the effect is not at all consistent with the theory of elasticity. The stress brings about a change in d that is 10^3 times as large as that expected from the values of elastic compliance. The effect is anisotropic, and most remarkable when the stress is applied along $[1\bar{1}0]$; the stress of 5.4 kPa, only 1/20 of atmospheric pressure, results in $\Delta d/d \sim 4\times10^{-5}$. The effect is just significant, that is, $\Delta d/d \sim 1\times10^{-5}$, when the stress is along [111]; it is intermediate for the stress along $[11\bar{2}]$. Previous X-ray diffraction experiments by HADOX and the Bond method showed that d of (444) is not a one-to-one function of temperature; a variety of d vs temperature curves depending on stress, thermal history etc. were observed. It was shown that the temperature where d shows a minimum, or the minimum value of d can be the index of the d vs T relation. This is more clearly observed in the present d vs T results with the stress as a parameter. This means that the anomaly so far observed in d vs T relation in silicon is directly related to the negative thermal expansion. It is also found that the response to the external stress is very slow; the relaxation time is of the order of one hour. The behaviour is similar to that characteristic to viscoelastics. This explains the anomalies observed in the ultrasonic attenuation.

PS11.10.05 LATTICE SYMMETRY OF SILICON AT LOW TEMPERATURES. K. Munakata, Z. Lu, A. Kohno, Y. Soejima, A. Okazaki, Department of Physics, Kyushu University, Fukuoka 812-81, Japan

By means of X-ray diffraction according to the Bond method coupled with a quadruple-crystal monochromator, the lattice spacing of perfect crystals of silicon has been measured. It is found that the lattice symmetry at low temperatures is not cubic. In general, the lattice symmetry slightly distorted from cubic can be identified from the splitting of {hhh} and {h00}. If the symmetry is trigonal, monoclinic or triclinic, the data of {hhh} will be enough to specify the symmetry. It turned out that this is the case for silicon at low temperatures. Measurements were made in a temperature range 20-300 K, and repeated over three or more thermal cycles, on cooling and heating. In the previous experiment, while the temperature dependence of $d(440)$, the lattice spacing of (440), was normal, $d(444)$ showed a variety of temperature dependences depending on specimen and its thermal history. One specimen showed a normal temperature dependence of $d(440)$ and anomalous of $d(444)$, indicating a break of the cubic symmetry. In the present experiment, a specimen showed normal behaviour of $d(444)$ above 120 K. However, below this temperature, *i.e.* in the region of negative thermal expansion, it showed slightly anomalous behaviour; in this temperature region, a set of four {444} spacings that are equivalent in the cubic lattice were examined simultaneously. While in the first thermal cycle the four values of $d\{444\}$ were all different, in the third cycle the values were separated into two: one for 444 and three for the other three, namely 444, 444 and 444. The separation of the two sets is marginally significant in terms of the precision of the data. Only a trigonal lattice symmetry is consistent with this. The results suggested an effect of a kind of aging that was also observed in the Pendellösung experiment of silicon at low temperatures.