

08.4-33 HIGH-PRESSURE CRYSTAL STRUCTURE OF QUARTZ UP TO 102 KBAR. By J. Glinnemann & H. Schulz, Max-Planck-Institut für Festkörperforschung, Stuttgart, FRG

To test a new type of high-pressure cell for single-crystal work on four-circle diffractometers (Malinowski et al., Z. Krist. (1982) 159, 93), quartz was chosen to allow a comparison with recent work (D'Amour et al., Acta Cryst. (1979) B35, 550; Levien et al., American Mineral. (1980) 65, 920). An improved version of this cell is presented at this meeting by Dieterich et al. X-ray intensities were collected with a Philips PW1100 at 1 atm and 40, 72, and 102 kbar. Experimental conditions chosen were: $M\alpha_1$ -radiation, $\max(\sin\theta/\lambda) = 0.9\text{\AA}^{-1}$, stepscans. Half a sphere of reciprocal space with $-10 \leq h, k \leq 10$, $1 \leq l \leq 10$ was collected. The number of measured reflections laid between 1100 and 1300, i.e. nearly 90% of all non-Friedel reflections. Averaging yielded 240-280 unique observations with about 20% of the $I's \leq 3\sigma I$. The structural changes observed with increasing pressure include:

- (1) A decrease in the Si-O-Si angle from 143.8(1) to 130.3(1)°;
- (2) One Si-O bond distance remains constant at 1.614(1)Å, whereas the other one decreases from 1.605(1) to 1.600(1)Å. This results in only a minor decrease of the mean Si-O length from 1.610(1) to 1.607(1)Å.
- (3) The two inter-tetrahedral O-O distances decrease from 3.414(2) to 2.947(2)Å and from 3.345(2) to 2.793(2)Å.

Because of (2) the small but significant decrease in the average Si-O bond length observed by Levien et al. at pressures up to 61.4 kbar is again open for discussion.

08.4-34 AVERAGE STRUCTURE AND FAULTED SEQUENCES IN ORIENTITE. By M. Mellini, S. Merlino and M. Pasero, Dipartimento di Scienze della Terra, Università di Pisa, Pisa, Italy.

Orientite is a manganese silicate, closely related to ardennite (Moore et al., 1979). A structure model for the mineral was obtained starting from the sursassite-pumpellyite structural relationships (Mellini et al., 1983). Sursassite, $Mn_2Al_2[(OH)_3(SiO_4)_3(Si_2O_7)]$ and pumpellyite, $Ca_2Al_3[(OH)_3(SiO_4)_3(Si_2O_7)]$, apart from the chemical differences, are built up by identical structural layers, differently stacked. The distinctive feature in the two structures is that whereas in pumpellyite similar silicate groups (ortho-ortho and diortho-diortho) face each other, in sursassite different silicate groups face each other on both sides of the aluminum octahedral rows.

Similarly, ardennite, $Mn_4(Al,Mg)_6[(OH)_6AsO_4(SiO_4)_2(Si_3O_{10})]$, is characterized by trisilicate and orthosilicate groups, with different groups facing each other. By analogy to the sursassite-pumpellyite case, a different modification can be envisaged, with similar groups facing each other. This model was now refined to $R = 0.08$ for orientite from Cuba ($a = 9.044$, $b = 6.091$, $c = 19.031$ Å, $Pcmm$) and resulted in the ideal crystal chemical formula $Ca_4Mn_3[(OH)_6(SiO_4)_3(Si_3O_{10})]$.

As for sursassite, disorder phenomena are now observed also in orientite, which is characterized by disordered alternation of orientite-like and ardennite-like domains. Such faults can explain the difficulties found during the X-ray structure analysis and also the previous assumption of a C-centered cell for orientite: the C-cell is due to the average structure, resulting from the two domains.

08.4-35 ISOTOPIC VARIATIONS IN DIAMOND IN RELATION TO CATHODOLUMINESCENCE

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Using a Q-switched neodymium-YAG laser delivering 1.3mJ focussed with a 25mm objective such as is used for the production of heat sinks, diamond plates approximately 5mm in diameter and 0.4mm thick have been dissected to provide specimens for carbon isotope analyses at known locations within a single stone (Nature; 303 (1983) 793).

This technique has now been further developed, and subsequent experiments have shown significant variations on a sub-millimetre scale, ranging from regular progressions compatible with isotope fractionation calculations to very erratic variations whose cause is unknown.

It has also been found that the complexity of these isotopic variations appears to correspond to the complexity of the observed cathodoluminescence patterns. Diamonds generally contain measurable quantities of nitrogen, and most cathodoluminescence is thought to be associated with nitrogen in various states of aggregation.

We have recently been able to make systematic measurements of nitrogen isotope ratios and nitrogen concentrations in addition to the carbon isotope determinations at selected locations within individual diamonds, and available results will be presented.

Isotopic variations associated with the two main types of diamond will also be illustrated.

08.4-36 REINVESTIGATION OF LILLIANITE STRUCTURE WITH SYNCHROTRON RADIATION

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The lillianite structure ($3PbS \cdot Bi_2S_3$) is well known as being composed of polysynthetic cell twinning, across (311) of the galena structure, of galena slab (Takéuchi and Takagi, Proc. Japan Acad. 10(1974), 843). The cell parameters and the space group are: $a = 13.535(3)$, $b = 20.451(5)$, $c = 4.104(1)$ Å and $Bm\bar{m}$. The unit cell contains four formula units (Takagi and Takéuchi, Acta Cryst. B28(1972), 649). According to the structure already determined based on film intensity data ($M\alpha_1$), there are three kinds of metal sites named M(1), M(2) and M(3). Although the M(1) and M(2) sites were thought to be occupied by Pb and Bi at random, while M(3) by only Pb, the accurate occupancy in each metal site has been left unknown. Making use of the synchrotron radiation at Photon Factory, we may now directly study the atomic occupancy at each atomic site of lillianite by selecting suitable X-ray wave length.

The specimen employed for the present study was almost cubic in form with dimension of approximately 0.09mm in edge and contains a small amount of Ag detected by electron microprobe analysis. Synchrotron radiation was monochromated by pyrolytic graphite(002) to provide X-ray beam with the wave length of 0.96Å. The value of wave length was selected just longer than both L-III-absorption edges of Pb ($\lambda = 0.95029$ Å) and Bi ($\lambda = 0.92336$ Å) atoms. It results in the fact that a linear absorption coefficient (μ) can be made into small value of 339cm^{-1} (cf. 866cm^{-1} for $M\alpha_1$). Under the condition, the anomalous dispersion terms of both Pb and Bi atoms were estimated as $f'(Pb) = -14.5$ and $f'(Bi) = -12.3$ respectively, but imaginary terms (f'') were neglected at this stage of analysis compared with large values of real term ($f' + f''$). The difference between