for intermediate rocks against the observations, it can be shown that intermediate rocks are not excluded by their observations. In addition, as the authors themselves have emphasized, the necessity of allowing for background corrections, which are very uncertain and which account for 90 percent of the measured values, makes it somewhat difficult to draw firm conclusions from this pioneering effort.

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Pressure-Induced Phase of Sulfur-Selenium

Abstract. Crystals of a fibrous phase of sulfur-selenium obtained at 20 kilobars and 280°C are trigonal, the most probable space groups being $P3_1$ and $P3_2$, with a = 7.85, c = 4.62 ± 0.01 Å. The unit cell contains nine atoms, and the measured density of 3.20 g/cm³ implies five sulfur and four Se atoms. The structure contains mixed atom helices of 1.54 Å pitch and 0.91 Å average radius.

In a continuing investigation of group VI A elements a new pressure-induced sulfur-selenium phase has been found. The phase is fibrous but is not isostructural with the fibrous sulfur phase (II) (*1*). In fact, we have also found that some selenium does dissolve in the fibrous sulfur phase.

Starting materials were pure (99.999+ percent) Se and S (American Smelting and Refining Company). A one-to-one mixture (atom percent) was put into a fused silica tube, evacuated, and sealed. The mixture was melted and kept at 250°C for 2 hours and annealed at 80°C for 110 hours. It was then removed from the tube and ground and mixed thoroughly in an attempt to insure homogenization. Some of this material was then packed into tantalum containers and subjected to pressure and heating in furnaces and piston cylinder devices similar to those described by others (*1*, ref. 1). The fibrous S-Se phase reported here was prepared in a furnace (2.54 cm diameter) at 20 kb. The temperature was raised to 550°C and held there for 10 minutes; the temperature was then reduced to 280°C and maintained there for 56 hours. The resulting material was not homogeneous but that part of the sample in the bottom portion of the sample capsule was red-brown, crystalline, and fibrous. A measurement of the density of isolated crystals of the fibrous form by the flotation technique gave 3.20 g/cm³.

An apparently single crystal of the fibrous sulfur-selenium was aligned along the fiber axis with oscillation photography; CuKα radiation was used, and Weissenberg photographs were taken. Lattice constants were determined from Buerger precession camera photographs (MoKα radiation). The diffraction symmetry of all the photographs is $6/m$, the only systematic absences being those reflections ($00l$) for which l is not equal to $6n$. The lattice constants of the particular crystal photographed are: $a = 7.85$, and $c = 4.62 \pm 0.01$ Å. Hexagonal selenium has $a = 4.355$, $c = 4.949$ Å. The sublattice obtained by a 30° rotation from the unit cell of the sulfur-selenium phase has lattice constants $a = 4.53$ Å and $c = 4.62$ Å. It appears then that the sulfur-selenium unit cell must contain nine atoms. A cell content of five S and four Se atoms gives an x-ray density of 3.20 g/cm³ equal to the measured density.

Any space group giving diffraction symmetry $6/m$ satisfying the conditions for these helices must contain screw axes. Further, because of the length of the c-axis, the helices in the sulfur-selenium phase must have three atoms per turn as in hexagonal selenium itself. No hexagonal space group giving diffraction symmetry $6/m$ can satisfy the requirements for this structure. Thus it appears that the $6/m$ is only an apparent diffraction symmetry; the more probable diffraction symmetry is $\bar{3}$. When crystals with this symmetry are 120° rotation-twinned, they give the apparent symmetry observed. This is analogous to the case of selenium itself (*2*) in which the twinning of crystals with diffraction symmetry $\bar{3}m$ leads to apparent symmetry $6/mmm$.

Thus the most probable space groups to which the fibrous $S_{0.555}Se_{0.444}$ belongs are $P3_1$ or $P3_2$. It is possible also that the two enantiomorphs are cocrystallizing in the twinned crystals.

Thus far the preliminary refinement of the x and y parameters with the use of the Busing-Martin-Levy (*3*) program (modified for use on the IBM 360 computer) and only the $hk0$ intensity data (for which there is no overlapping of nonequivalent reflections) indicates that the helix radius is close to 0.91 Å; the pitch, given by $c/3$ is 1.54 Å. This implies an average S-Se distance (*4*) of 2.20 Å as compared with a calculated one of 2.18 Å based on a value of 2.34 Å for an Se-Se distance and 2.05 Å for an S-S distance.

For Se, the pitch and radius of the helix are 1.65 and 0.95 Å, respectively (*4*). Thus the larger a-axis of the subcell (see above) implies poorer packing efficiency of the sulfur-selenium phase than of the hexagonal Se phase.

Spacings were calculated with the lattice constants determined from the Buerger precession camera photographs. It is seen in Table 1 that the calculated spacings compare well with those measured on an x-ray powder photograph of the material. All nonequivalent sets of indices are given.

There appears to be a range of solid solutions having the same fibrous structure, but the limits have not yet been determined. The new phase is not nearly as stable as the fibrous sulfur phase, in which case a specimen 15 months