Letter

High-pressure X-ray diffraction study of ThOS and UOSe by synchrotron radiation

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Abstract

High-pressure X-ray diffraction studies were performed on ThOS up to 43.3 GPa and on UOSe up to 47.5 GPa, at room temperature, using a diamond anvil cell and synchrotron radiation. The tetragonal structure ($P4/nmm$) of these compounds was retained over the whole pressure range. The bulk modulus B_0 and its pressure derivative B_0' were determined for each compound.

ThOS and UOSe have a PbFCl-type structure (tetragonal) at room temperature and atmospheric pressure. They belong to the space group $P4/nmm$ with two molecules per unit cell. Values for the lattice constant at ambient pressure are

TABLE 1

Lattice parameters of ThOS and UOSe at ambient pressure (from ref. 1)

ThOS			UOSe		
a (pm)	c (pm)	c/a	a (pm)	c (pm)	c/a
396.3	674.6	1.702	390.8	699.6	1.790

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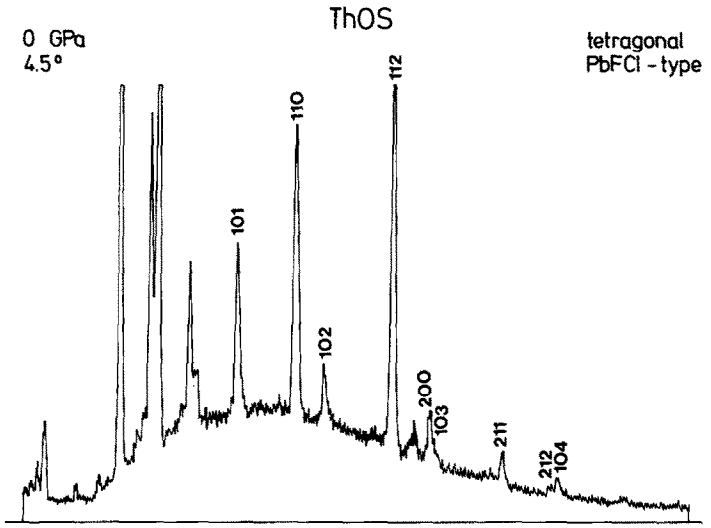

Fig. 1. Diffraction spectrum at ambient pressure of ThOS.

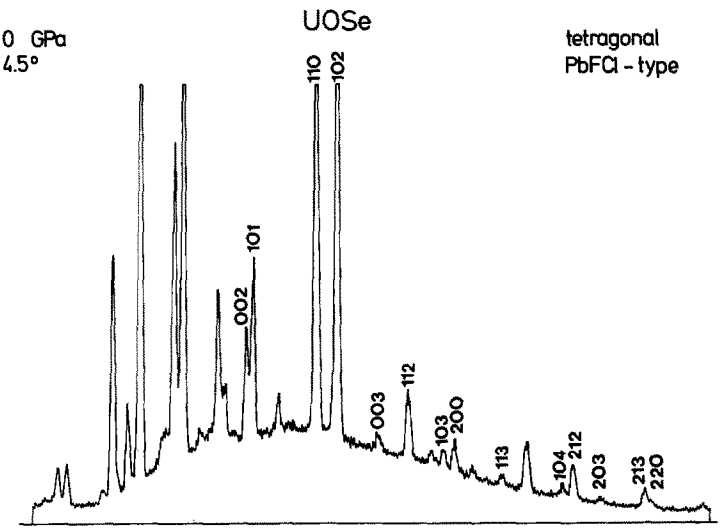

Fig. 2. Diffraction spectrum at ambient pressure of UOSe.

presented in Table 1 [1]. The present work is a study of the compressibility and crystal structure of ThOS and UOSe in a pressure range up to about 47 GPa. To the best of our knowledge these are the first AnOX compounds studied by X-ray diffraction under pressure.

ThOS was obtained by the reaction of thorium (99.8% pure) and sulphur (99.99% pure) in the presence of oxygen. UOSe was synthesized from pressed powder mixtures of UO_2 and USe_2 (the former prepared from uranyl acetate pro

analysi, the latter from 99.7% pure uranium turnings and 99.99% pure selenium reacted in silica near 1000 °C) annealed for three weeks at 950 °C in a silica ampoule.

High pressure studies were performed at room temperature with a Syassen–Holzapfel diamond anvil cell using synchrotron radiation at Hasylab-Desy. The electron energy of the storage ring was 3.7 GeV. Our powder samples and ruby chips were loaded in a hole 0.17 mm in diameter of an inconel gasket. The applied pressure was measured via the wavelength shift of the ruby fluorescence excited by an argon laser [2]. Hydrostatic conditions were approached by adding silicone oil as a pressure transmitting medium.

In our experiments only one Bragg angle was fixed. The exact value of this angle was determined from the diffraction spectrum of an NaCl standard sample. For each pressure step, lattice parameters were calculated from the diffraction spectra.

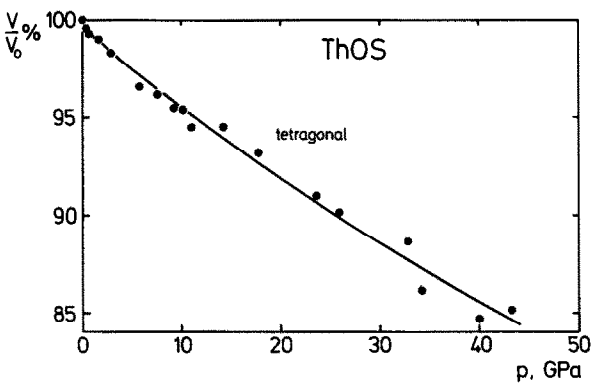

Fig. 3. Equation of state (relative volume *vs.* pressure) for ThOS.

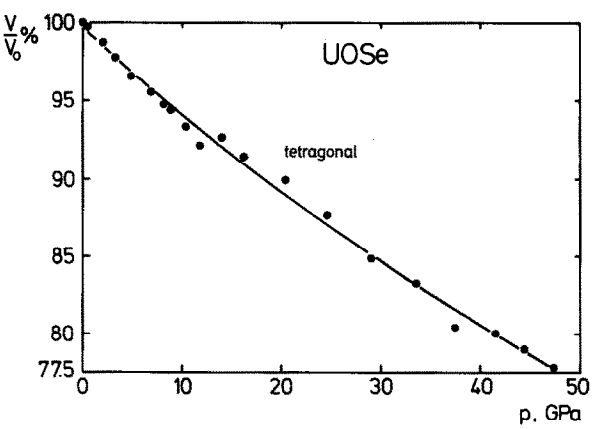

Fig. 4. Equation of state (relative volume *vs.* pressure) for UOSe.

TABLE 2

Isothermal bulk moduli of ThOS and UOSe

Sample	B_0 (GPa)		
	Birch	Murnaghan	Average
ThOS	201	202	201.5
UOSe	153	155	154

TABLE 3

Pressure derivative of the bulk moduli of ThOS and UOSe

Sample	B_0'		
	Birch	Murnaghan	Average
ThOS	3.1	2.9	3.0
UOSe	2.1	1.5	1.8

ThOS was studied up to 43.3 GPa in 18 steps of increasing pressure. UOSe was studied up to 47.5 GPa in 20 steps of increasing pressure. No structural phase transition was observed over the whole pressure range. Figures 1 and 2 show diffraction spectra of ThOS and UOSe respectively at ambient pressure.

The relative volumes of both compounds are plotted *vs.* pressure in Figs. 3 and 4. They are based on the lattice parameters refined by the program LCR-2 [3]. V_0 is based on the lattice parameters determined in the diamond anvil cell at ambient pressure, the piston being removed from the cell. Table 2 shows the bulk modulus of ThOS and UOSe, and Table 3 its pressure derivative. These values were calculated by fitting the pressure–volume data to the Murnaghan and Birch equations [4, 5].

References

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