Structure of Chalcogens under High Pressure

※Kiyofumi Nagata/3752, Masaharu Takumi/3749, Toru Ueda/3846, Akihisa Hirata/3843

Department of Applied Physics, Faculty of Science, Fukuoka University

In this beam time, powder X-ray diffraction patterns of trigonal selenium (Se) and α-monoclinic $Se_{0.96}Te_{0.04}$ have been measured using a DAC at pressure up to 54 GPa and 33 GPa, respectively. The purpose of the former experiment is to investigate whether or not Rietveld analysis of diffraction data obtained from such a highly parallel X-ray beam at BL10XU is available for the structural analysis. The purpose of the latter experiment is to investigate an effect of tellurium (Te) content on the sequence of the phase transition in α-monoclinic type $Se_{1-x}Te_x$ mixture. All diffraction patterns were measured with X-ray beam of 0.4373 A wavelength, which was collimated to 50 μm square.

1. Trigonal Se

Good powder diffraction pattern has been obtained and Rietveld analysis has been carried out. The resultant crystal data of trigonal Se near 13 GPa are summarized in Table together with previously obtained results. The S/N of the diffraction data obtained at SPring-8 is so high that the reliability factor R_F on the refinement converges to the smallest value. Though the present lattice parameter of c-axis is a little different from that obtained from the diffraction data measured at BL18C of Photon Factory (PF), other crystal parameters, atomic position parameter (u), inter- (R) and intramolecular (r) bond distances, and bond angle (θ) obtained from both diffraction data measured at SPring-8 and PF agree well within an experimental error. This shows that Rietveld analysis of the diffraction data obtained from X-ray source at BL10XU is very useful for the structural analysis.

Table. Crystal parameters of trigonal Se near 13 GPa obtained by Rietveld analysis of the diffraction data measured with various X-ray sources.

X-ray source	SPring-8	PF(BL18C)	Mo$K\alpha$
Pressure	12.9 GPa	12.4 GPa	12.7 GPa
λ	0.4373 A	0.6199 A	0.7107 A
R_{wp}	2.66	4.34	4.36
R_F	1.24	1.53	2.49
a	3.6739(16)	3.6843(12)	3.6376(38)
c	5.1569(16)	5.1395(14)	5.1048(57)
u	0.2692(16)	0.2739(36)	0.2635(89)
r	2.427(7)	2.447(16)	2.377(28)
R	2.913(5)	2.900(11)	2.902(19)
θ	104.6(3)	103.6(6)	105.6(19)

2. α-monoclinic $Se_{0.96}Te_{0.04}$

The crystal structure of α-monoclinic $Se_{0.96}Te_{0.04}$ is almost the same as that of α-monoclinic pure Se consisting of Se_8 ring molecules. However, because of 4 at.% Te content, the band gap is lower than that of α-monoclinic pure Se by 0.1 eV and the color is red-black. Figure shows the diffraction patterns of α-monoclinic $Se_{0.96}Te_{0.04}$ at various pressures. The crystal undergoes phase transitions to a new metallic phase[1] at 9 GPa and to orthorhombic phase at 23 GPa. The second transition is a little sluggish. The phase transition starts at 23 GPa and finishes at 30 GPa. The sequence of the phase transition is similar to that reported previously in α-monoclinic pure Se.[1] However, the first and the second phase transition pressures are lower than those of α-monoclinic pure Se by 3 and 10 GPa, respectively. Pressure range where the new metallic phase exists in α-monoclinic $Se_{0.96}Te_{0.04}$ is narrower than that in pure Se by 7 GPa, suggesting that the new metallic phase becomes to be unstable with Te content.

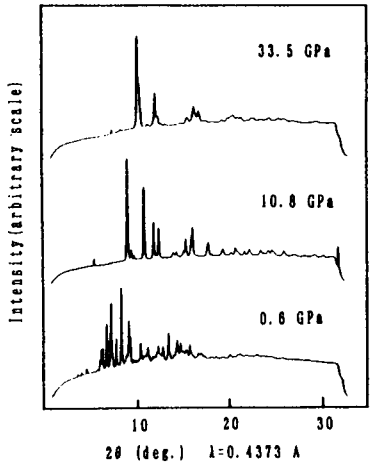

Figure. The diffraction patterns of α-monoclinic $Se_{0.96}Te_{0.04}$ at various pressures.

References

1)Y. Akahama, M. Kobayashi and H. Kawamura: Phys. Rev. B 56 (1997) 5027.