# Structure Analysis of Tetrahedral-Molecular Crystal and Amorphous at High Pressure

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In our beam time, we checked performance of the diffractometer installed in the BL10XU-downstream station and tested a Bragg Fresnel Lens which was thought of the most suitable focusing element for ultrahigh-pressure x-ray diffraction measurements. The remaining beam time was used to study phase transition in $SnI_4$ under pressure.

At atmospheric pressure $SnI_4$ crystallizes into a cubic lattice with space group $Pa\bar{3}$ and with eight molecules packing loosely in the unit cell. This phase had been believed to undergo gradual phase transition to the metallic amorphous phase above 10 GPa. Our recent SR x-ray diffraction study, however, showed that $SnI_4$ exhibited the structure sequence, crystalline phase(CP) I ($Pa\bar{3}$) – metallic CP II – amorphous – CP III with increasing pressure. According to this observation, CP II is stabilized at 7 GPa and its structural disordering starting at 15 GPa results in the amorphous phase. Therefore it is important to determine the crystal structure of CP II in understanding the amorphization process.

High-pressure x-ray powder diffraction measurements were carried out at BL10XU using a monochromatic x-ray tuned at 27.50 keV in combination with an imaging plate detector. We conducted two sets of experiments using a diamond anvil cell as a high-pressure device. In the first experiment, $SnI_4$ was compressed to 15 GPa in a pressure transmitting medium of 1:1 normalpentane-isopentane mixture. The CP I – CP II phase transition took place at 8 GPa at room temperature. Figure 1 shows a diffraction pattern measured at 9.6 GPa. Although about 40 % in volume fraction remains of CP I, newly appeared reflections of CP II can be clearly observed. Above 13 GPa all reflections became broader.

In the second experiment, no pressure medium was used. A sample was compressed to 10.3 GPa at room temperature. To accelerate the transformation from CP I to CP II, the sample was heated at about 500 ℃ for a few minutes by irradiating a collimated YAG-laser beam through a diamond. Figure 2 shows a diffraction pattern of the resulting phase measured at room temperature after heating. Examination of d-values indicates that this diffraction pattern contains several, new reflections from an unknown phase. As the pressure was decreased at room temperature, at 4 GPa the unknown phase transformed to CP I showing broad reflections, which became sharper after the sample was heated at 80 ℃ at atmospheric pressure.

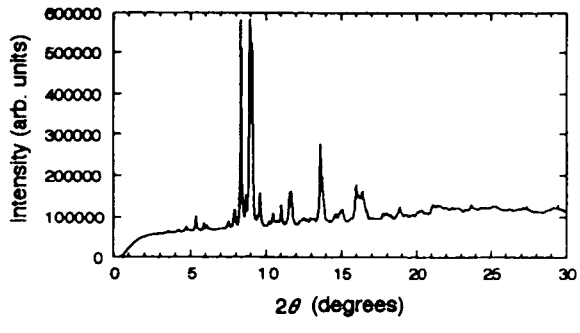


Figure 1. Diffraction pattern at 9.6 GPa.

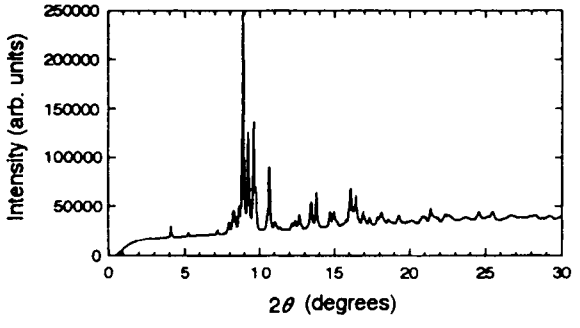


Figure 2. Diffraction pattern at 10.3 GPa after heat treatment at about 500 ℃.