

# X-ray diffraction study of sulfur laser-heated at 11 GPa

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## 1. Introduction

Since the pioneering work of Tammann [1], sulfur has long been studied at high pressure, the dramatic findings being obviously the consecutive structural phase transitions, superconductivity, and amorphization.

The amorphization of sulfur was detected by x-ray diffraction as well as by Raman scattering and, there exist substantial controversies among the pertinent results; between the amorphization pressures detected by x-ray diffraction and Raman scattering, for example. Besides, within the Raman scattering experiments, the situation is quite complicated by occurrence of photo-induced change which is caused by laser beam for exciting the Raman spectra.

Apparently at least four different photo-induced phases have been reported under pressures up to 40 GPa. The assignment of the phases has been achieved simply by comparing the Raman scattering data with those of known phases without any direct structural analysis by x-ray diffraction, as exemplified by the assignment of high-pressure high-temperature (hpht) phase to  $S_6$  [2]. The present work was undertaken to study a photo-induced phase of sulfur which had been laser-heated at about 11 GPa.

## 2. Experimental

Commercially obtained sulfur ( $\alpha$ - $S_8$ ) with purity 99.999% was loaded with a gasketed diamond-anvil cell together with chips of ruby to monitor the pressure by shift of the fluorescence line. A mixture of methanol, ethanol, and water (16:3:1 in volume ratio) was employed as a pressure-transmitting medium.

At a pressure of 10.5 GPa, the sample was exposed to a pulse laser beam from a Nd:YAG source. The power density was about  $3.6 \times 10^7$  W/cm<sup>2</sup> which was sufficient to photo-induce the hpht phase.

The sample laser-heated and still under pressure (hereafter designated as HPLH) was

x-rayed for 30 min at BL 10XU. For reference, sulfur pressurized to 12 GPa (HPRT) was also x-rayed.

## 3. Results and discussion

Figure 1 shows x-ray diffraction pattern of sulfur laser heated at 10.5 GPa. The pattern is basically a mixture of HPLH and HPRT phases. After subtracting reflections of HPRT, peaks of HPLH were derived. These are indicated by asterisks in Fig. 1.

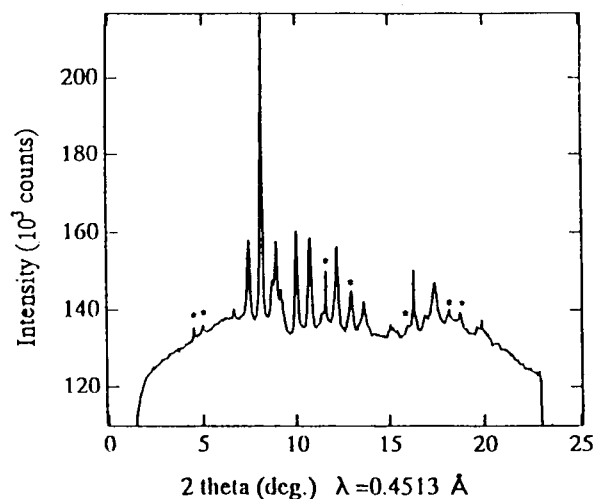


Fig. 1. X-ray diffraction pattern of sulfur after being laser-heated at 10.5 GPa.

The leftmost peak in Fig. 1 has a  $d$  spacing of 5.71 Å, and this is larger than the largest value in the  $d$  spacing of  $S_6$ , 5.40 Å [3]. Likewise, other peaks, being taken into account of the contraction caused by pressurization, cannot be interpreted by  $S_6$ . Other likely candidates are not in accord with the present result, suggesting an appearance of a new phase.

## References

- [1] G.Tammann, *Ann.Phys.* **3**, 178 (1900).
- [2] W.Häfner *et al.*, *High Press.Res.* **6**, 57 (1990).
- [3] JCPDS File No. 13-144.