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# Pressure-volume measurements by using diamond-anvil cells and an image processing system

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Diamond-anvil cells combined with a video image processing system provide a simple tool for measuring pressure-volume relations of small solid samples under high pressure. The feasibility of the technique is demonstrated for glassy Se and crystalline InSb. Accuracy evaluated for linear-scale measurements is approximately  $\pm 0.5\%$ .

High-pressure compressibility studies give useful information on the nature of atomic bonding forces in solids. In addition, investigations of volume changes accompanying pressure-induced phase transitions are of importance for understanding physical properties of matter. However, pressure-volume data are scarce, since experimental techniques available for the measurements have been limited so far.<sup>1,2</sup> For example, the piston-displacement and ultrasonic methods need large samples of about  $1\text{ cm}^3$  for inspection. The x-ray diffraction method can be applied only for crystalline materials, and we have had no techniques to measure pressure-volume relations of small and/or glassy solids subjected to hydrostatic pressure.

Diamond-anvil cells have been widely used for generating hydrostatic pressure up to  $\sim 100$  kbar.<sup>3</sup> We report in the present note a technique for pressure-volume measurements by using the cell with a video-image processing system. For demonstrating the feasibility of the technique, we present some results obtained for glassy Se and crystalline InSb.

A diamond-anvil cell (Toshiba Tangaroi) with gaskets made of Inconel having holes of  $300\text{ }\mu\text{m}$  in diameter and the mixture of methanol-ethanol were used for generating hydrostatic pressure up to  $\sim 100$  kbar. The pressure was calibrated by using photoluminescence spectra from ruby powders excited by a 4880-A beam from an Ar-ion laser (a review of these techniques is given in Ref. 3). Glassy samples of Se were prepared by vacuum evaporation and single-crystalline InSb disks were obtained by polishing the ingot. Shapes of the samples were not regular, and typically  $20\text{ }\mu\text{m}$  in thickness and  $100\text{ }\mu\text{m}$  in width.

The linear dimension of these specimens under hydrostatic pressure in the cell could be directly measured by using an optical microscope attached with an objective lens of  $10\times$  and a microscale, if the shape of the sample was smooth and regular. The accuracy of the measurement for length is about  $\pm 1\text{ }\mu\text{m}$ , which corresponds  $\sim \pm 1\%$ . This provides a simple method to measure pressure-volume relations. (Besson *et al.* have tried a similar technique by taking photographs, however the details are not known.<sup>4</sup>)

A more accurate method is to measure the two-dimensional extent of samples by using an image-analysis technique.<sup>5</sup> For these measurements, magnified views of the samples were converted to electric signals by using a high-precision vidicon camera (Hamamatsu C1000), and the images were stored in a digital image memory having  $128\times 128$

sampling points and 64 gray levels. Then, the image data were fed into a computer, and transformed to binary forms by setting a certain threshold level, and finally areas of the images were counted. A typical number of the area was about 5000 picture elements. The results obtained by this analysis is the area  $S$  of samples, and the linear dimension  $L$  as well as the volume  $V$  can be evaluated as  $L = S^{1/2}$  and  $V = S^{3/2}$ , if we may assume isotropic changes of sample shapes.

Results for glassy Se and crystalline InSb are shown in Fig. 1. As shown in Fig. 1(a), the volume of Se is compressed continuously with increasing pressure up to 100 kbar. When depressurizing the sample, hysteresis effects appear if the pressure is higher than  $\sim 20$  kbar, but when pressure is decreased to 1 atm the volume always relaxes to its original value. This may be due to the fact that the glass-transition temperature of Se at 1 atm is around room temperature.<sup>6</sup> The compressibility at 1 atm calculated from the result is  $1.1\times 10^{-2}/\text{kbar}$ , nearly the same with the results obtained with various techniques,  $\sim 1\times 10^{-2}/\text{kbar}$ .<sup>7</sup> Further, the pressure-length relation is consistent with the previous result.<sup>7</sup> The result shown in Fig. 1(b) for InSb agrees with a theoretical calculation.<sup>8</sup> The sample showed a marked volume transition of  $\sim 18\%$  at  $\sim 30$  kbar, which has been discussed by Soma<sup>8</sup> as the transition from the zinc-blend to  $\beta$ -Sn structure. When the sample is depressurized, the  $\beta$ -Sn structure reverts to the original one at 8 kbar. We see in the

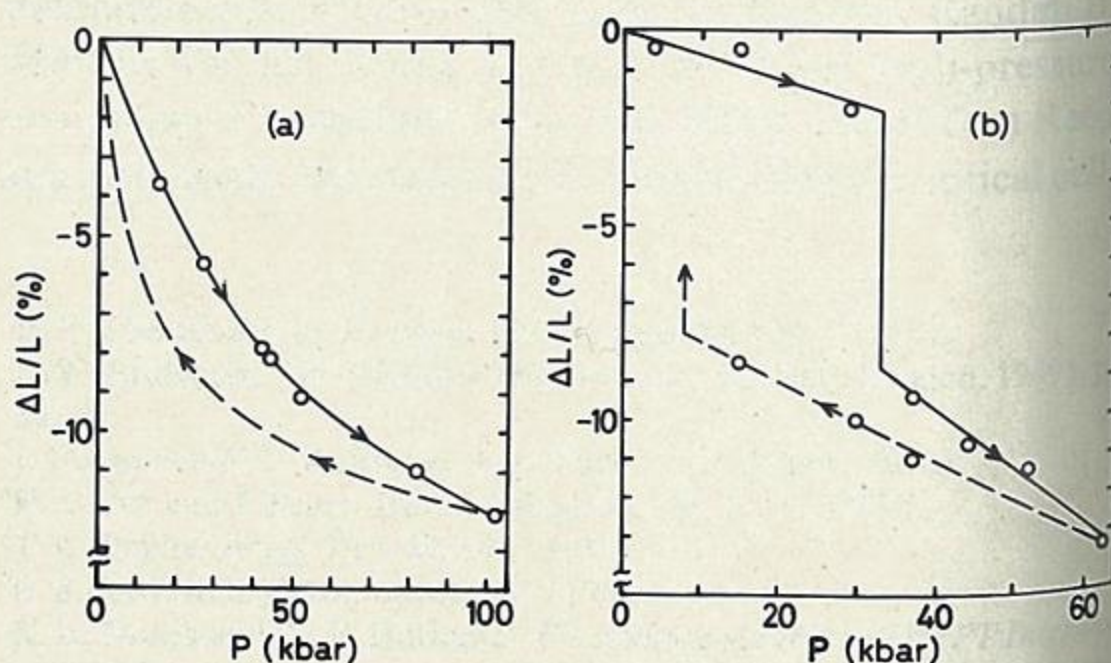


FIG. 1. Pressure-length ( $P$ - $L$ ) relations for (a) glassy Se and (b) crystalline InSb.

figure that, in the  $\beta$ -Sn structure, gradients of the pressure-length curves obtained when increasing and decreasing pressure are different from each other. It seemed that the single-crystalline InSb transformed to polycrystalline forms at  $\sim 30$  kbar, and generated voids located at crystallite boundaries. These voids may contribute to the difference in length. At the phase-transition points, the shape of the sample changed nonsimilarly, and therefore the absolute magnitudes of discontinuity may contain larger errors in length.

In summary, we have developed a useful technique for measuring changes in length and area of small solid samples subjected to hydrostatic pressure. The accuracy of the measurements evaluated for length is approximately  $\pm 0.5\%$ . This is mainly attributable to the image processing system, and can be improved by using a more stable system. The

technique may be applied to investigate pressure-volume relations at high and low temperatures. Transparent samples may also be measured by using a phase-contrast microscope.

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<sup>1</sup>C. A. Swenson, *Physics at High Pressure in Solid State Physics*, Vol. 11, edited by F. Seitz and D. Turnbull (Academic, New York, 1960), p. 41.

<sup>2</sup>J. W. Brasch, *Rev. Sci. Instrum.* **51**, 1358 (1980).

<sup>3</sup>A. Jayaraman, *Rev. Mod. Phys.* **55**, 65 (1983).

<sup>4</sup>J. M. Besson, J. Cernogora, and R. Zallen, *Phys. Rev.* **B22**, 3866 (1980).

<sup>5</sup>A. Rosenfeld, *Picture Processing by Computer* (Academic, New York, 1969).

<sup>6</sup>K. Tanaka, *Phys. Rev. B* **30**, 4549 (1984).

<sup>7</sup>E. Litov and O. L. Anderson, *Phys. Rev. B* **18**, 5705 (1978).

<sup>8</sup>T. Soma, *Philos. Mag. B* **41**, 113 (1980).