

# Shock-recovery studies on InSb single crystals up to 24 GPa

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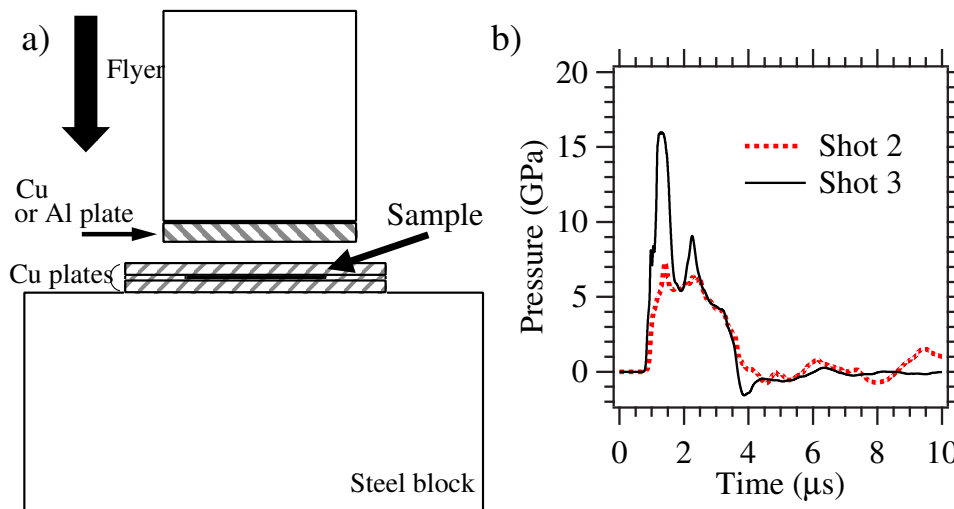
**Abstract.** A series of shock-recovery experiments on InSb single crystals along the (100) or (111) axes up to 24 GPa were performed using flyer plate impact. The structures of recovered samples were characterized by X-ray diffraction (XRD) analysis. According to calculated peak pressures and temperatures, and phase diagram for InSb, the sample could undergo phase transitions from zinc-blende structure to high-pressure phases. However, the XRD trace of each sample corresponded to powder pattern of InSb with zinc-blende structure. The XRD trace of each sample revealed the absence of additional constituents including metastable phases and high-pressure phases of InSb except for samples shocked around 16 GPa. At 16 GPa, in addition to zinc-blende structure, additional peaks were obtained. One of these peaks may correspond to the *Cmcm* or *Immm* phase of InSb, and the other peaks were not identified.

## 1. Introduction

The mechanical behaviors and structural phase transformations of III–V binary compounds under high pressures are of both fundamental interest and technical importance for designing electronic and optical devices. High-pressure and high-temperature states of InSb have been intensively investigated because of its technological importance, its low melting temperature, and low transition pressure [1]. Although InSb has been investigated at high pressures since the 1960s, its phase diagram is still the subject of debate [1–6]. Nelmes and co-workers showed that the long-accepted pressure–temperature ( $P$ – $T$ ) diagram was substantially incorrect [3, 4]. Mezouar *et al.* revised the  $P$  –  $T$  diagram of InSb [5]. More recently, the  $P$ – $T$  diagram has been extended to a pressure of 20 GPa and to a temperature of 1570 K and local structures of liquid InSb at a high pressure and a high temperature have been investigated [7].

Shock experiments have revealed the behavior of several materials under extreme conditions of strain rate, temperature, and pressure [8]. A phase transition to either a metastable or amorphous state can be expected because of extreme conditions achieved during shock compression [9, 10]. For instance, metastable high-pressure phase of CdS [11] and KCl [12] have been observed. Metastable phases caused by shock compression using a pulse laser have been recovered [13–15]. Shock compression studies on III–V and II–VI binary semiconductor have been reported [11, 16–19]. However, few studies have investigated shock loading of InSb. The shock-induced phase transition along the (111)-axis of InSb was investigated and the application of shear stress was found to facilitate the phase transition under shock loading [20, 21]. However, the structure of shock-loaded InSb crystals has not been analyzed.





**Figure 1.** (a) Schematic diagram of loading configuration with gas-gun shock-compression fixture. (b) Example of simulated pressure profile for shocked InSb.

In this study, we conducted shock compression on InSb single crystals along the (100) or (111)-axis and characterized the structures of recovered samples by X-ray diffraction (XRD) analysis. Recovered samples revealed that high-pressure phases of InSb produced by shock compression remained after pressure release.

## 2. Experiment

The samples used were InSb(100) and InSb(111) wafers of 450  $\mu$ m thickness. A small piece was cut from a wafer and used as a sample. A flyer plate impacted an InSb single crystal. An approximately 1.5-mm-thick Cu or Al flyer plate was mounted on a 30-mm-diameter projectile and accelerated using a single-stage powder-propellant gun. The flyer velocity was measured by the magnet flyer method, which involves embedding a magnet in the flyer. The flyer velocity was varied between 0.4 and 1.1 km/s. The sample was placed between two square copper plates (thickness: 1.0 mm; side length: 40 mm) and was bonded to a mild steel holder and a momentum trap [22]. Only a few samples that were stuck to the copper plates were recovered because the copper plates generally broke during shock loading at 23 GPa. Figure 1(a) shows a schematic diagram of the experimental arrangement.

The shock pressure and temperature were estimated by computer simulation using a two-dimensional hydrocode, ANSYS®AUTODYN®-2D. The polynomial equation of states was applied to the relation between the pressure and the volume of InSb [4, 6]. In the calculation, we assumed that  $\rho_0\gamma_0=\rho\gamma=\text{constant}$ , where  $\rho$  is the density and  $\gamma$  is the Grüneisen parameter. We also assume that the simulation parameters such as the heat capacity, the thermal conductivity, the shear modulus, and the bulk modulus remain constant at their values at 300 K [6,23–25]. Table 1 lists the experimental conditions and calculation results. Figure 1(b) shows the simulated pressure profiles for shots 2 and 3.

The crystal structures of recovered samples were analyzed by XRD using a Rigaku RINT-2200 diffractometer with Cu  $K\alpha$  radiation ( $\lambda = 1.5406 \text{ \AA}$ ). All measurements were performed at room temperature.

**Table 1.** Experimental conditions and simulation results for shock compression of InSb.

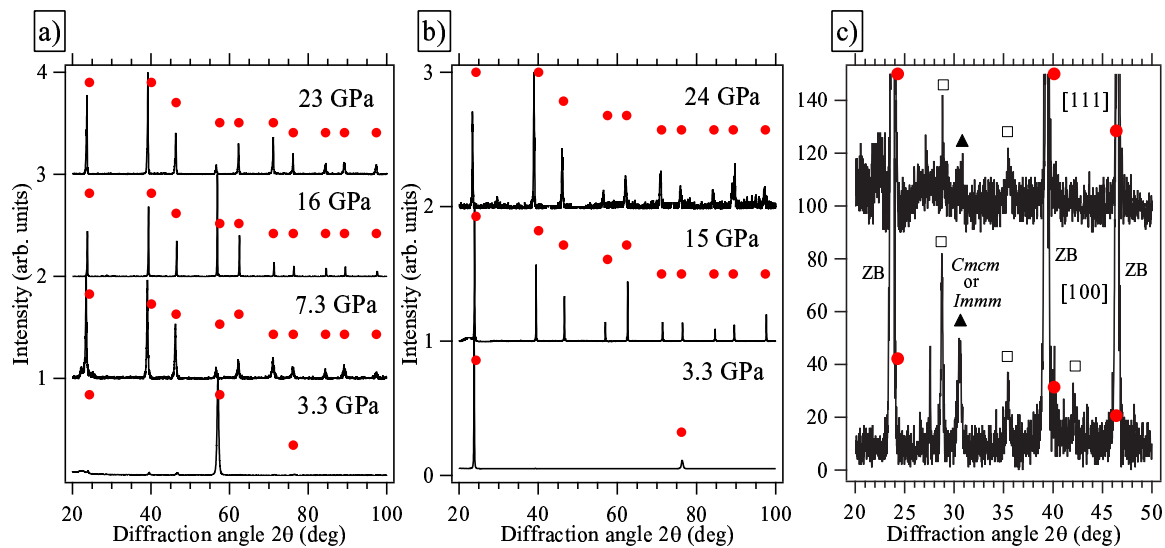
Shot	Sample	Flyer Material	Flyer vlocity (km/s)	Calculated pressure (GPa)	Calculated temperature (°C)
1	[100]	Al	0.40	3.3	99
2	[100]	Al	0.92	7.3	320
3	[100]	Cu	0.80	16	550
4	[100]	Cu	1.1	23	780
5	[111]	Al	0.41	3.3	99
6	[111]	Cu	0.78	15	520
7	[111]	Cu	1.1	24	840

### 3. Results and discussion

Figure 2 shows XRD spectra of shock-recovered InSb crystals. For both series of shocked InSb, the XRD spectra are consistent with a powder XRD pattern corresponding to the zinc-blende structure of InSb. Although the XRD spectra of samples shocked at 3.3 GPa exhibit a high degree of preferred orientation, it contains other reflections. These results are similar to those for Si [22] and Ta [26]. They indicate that many grains are produced independently of the shock-loading direction. Although some material remains a single crystal at extreme shock compressions up to and over a Mbar [27], polycrystalline InSb were formed at a pressure of 3.3 GPa. The difference in the degree of preferred orientation between (100) and (111) samples shocked at 3.3 GPa may be caused by the difference in Hugoniot elastic limit for these orientation. The grain formation indicated in the XRD spectrum is conjectured to be the result of plastic deformation and a phase transition occurring simultaneously with deformation.

Figure 2(c) shows XRD spectra of samples shocked around 16 GPa. In addition to peaks corresponding to InSb with the zinc-blende structure, a peak at  $2\theta=30.5^\circ$  is observed. This diffraction peak originates from the (200) reflection of InSb with the *Cmcm* or *Immm* structure. The appearance of this additional peak indicates that shock-induced phase transitions from the zinc-blende InSb occurred and some high-pressure phases (at least the *Cmcm* or *Immm* phase) were quenched. Although the shock-induced state has a very short period, high-pressure phases of Si produced by laser-driven shock wave were quenched [14, 15]. Other additional peaks were observed at  $2\theta=28.8, 35.4,$  and  $42.2^\circ$ ; these peaks could not be identified. The d-spacing of these peaks are 3.10 Å, 2.53 Å, and 2.14 Å, respectively. They may correspond to metastable phases produced by shock compression. In particular, the peak at  $2\theta=35.4^\circ$  can be explained as InSb with a hexagonal structure, which has been observed at a pressure of 17.5 GPa [6]. These other peaks may be associated with a shock-induced metastable phase that was not obtained in a static experiment [11] or with a metastable phase that is possibly produced under rapid decompression [28].

Figure 3 shows the calculated pressures and temperatures for the present experimental conditions plotted on the phase diagram of InSb obtained by experiments [5, 7]. To compare the results, simulations were also conducted for the conditions used in previous studies [20, 21]. As mentioned above, the simulations were conducted with disregard to variation in thermodynamic variable due to rise of pressure and temperature and latent heat due to phase transitions [22]. Although all the present experimental conditions correspond to high-pressure phases of InSb, only samples shocked around 16 GPa show evidence of the shock-induced phase transition. We speculate that the period of high-pressure phases and the temperature rise are long enough



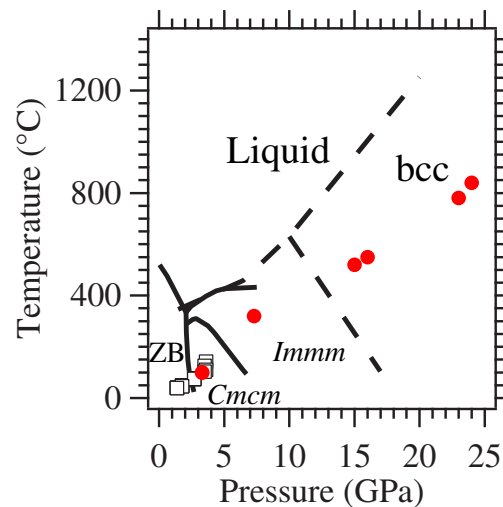
**Figure 2.** XRD spectra of InSb crystals after shock compression along (a) (100) and (b) (111) axes. (c) XRD spectra of InSb shocked around 16 GPa. Filled circles (“ZB”) denote diffraction peaks from the zinc-blende phase. The triangles denote diffraction peaks from the *Cmcm* or *Immm* phase. The squares denote diffraction peaks from unidentified phases.

for the phases to grow sufficiently to remain after shock unloading. On the other hand, the recovered samples shocked around 23 GPa only gives the zinc blende structure. One of the possible explanations is that the amount of recovered sample that was shocked around 23 GPa may be too little to definitively demonstrate shock-induced phase transitions. The other is the effect of post-shock temperature due to residual heat. In other words, even though high-pressure phases and/or metastable phases of InSb were formed by shock loading at 23 GPa, these phases were transformed to the zinc blende structure. According to the simulations, the calculated residual temperature of the recovered sample shocked around 23 GPa was around 550 °C, which is too high to survive for the high-pressure phases and metastable phases.

It is not possible to determine whether the *Cmcm* or *Immm* phase is produced because only a single XRD peak was observed. The unidentified peaks in the XRD spectra of samples shocked around 16 GPa may indicate the existence of unknown phase transition pathways. To clearly determine the shock-induced phase transitions of InSb, it is necessary to characterize the shocked samples by electron microscopy and Raman spectroscopy [29] or in-situ XRD analysis [30, 31, 32].

Recently, structural phase transitions of III–V semiconductors caused by non-hydrostatic pressure induced by mechanical impact have been reported [33]. Undefined phases have been detected from recovered samples. Although the estimated pressure for the mechanical impact was much lower than our shock pressures, high-pressure phase or unknown metastable phase can be quenched. Uniaxial compression is induced by a plate impact, while severe shear deformation may be applied by the mechanical impact. Duration of high pressure state achieved by shock compression will be shorter than that of the mechanical impact. The shear deformation may play a key role in bringing about phase transitions.

It is suggested that the behavior of single-crystalline sample under shock loading differs from that of polycrystalline sample [27]. The structural phase transition may occur with facility if polycrystalline sample is used. However, if a polycrystalline sample is used for a shock compression experiment, residual heat induced by shock compression may affect stability of metastable and/or high-pressure phases.



**Figure 3.** Phase diagram of InSb obtained by computer simulation and the  $P - T$  conditions used in this study (solid circles) and previous studies [20, 21] (squares). The phase boundaries indicated by the solid lines are from reference [5] and the dashed lines denote the phase boundaries determined in reference [7].

#### 4. Conclusion

Shock-loaded InSb single crystals were characterized and evidences of shock-induced phase transitions were obtained. All recovered samples contain many grains with the zinc-blende crystal structure. A diffraction peak corresponding to the  $Cmcm$  or  $Immm$  phase was observed in XRD spectra of samples shocked around 16 GPa. Unidentified peaks in XRD spectra may indicate the existence of unknown phase transition pathways.

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