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Phase transitions in shock compressed bismuth identified using single photon energy dispersive X-ray diffraction (SPEDX)

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Abstract. We present evidence for phase transitions in shock-compressed bismuth using the SPEDX x-ray diffraction technique. Experiments were performed on the Vulcan laser at the Central Laser Facility, RAL, Didcot, UK. We observed diffraction from the (110) bcc peak of Bi-V, and from its calculated lattice parameter the pressure was determined to be approximately 17 GPa. Upon further compression (higher laser intensities), no further diffraction from solid phases was observed. Shock melting of bismuth is thought to occur between 18 and 27 GPa. Diffraction results at lower pressures as a function of delay time are also presented.

1. Introduction

Dynamic compression of materials using high-power lasers allows access to extreme P-T states that lie well beyond the current limits of diamond anvil cell techniques. Laser facilities such as the National Ignition Facility are capable of compressing samples to 10s of megabars (> 1 TPa), whilst ensuring that the sample remains sufficiently cool to investigate solid-solid phase transitions [1]. However, collecting X-ray diffraction data from such samples is difficult, as the very high laser intensities used to produce the nanosecond-duration plasma X-ray source create a hostile environment in which the X-ray background from the drive lasers can eclipse any diffraction signal from the sample [2]. Obtaining X-ray diffraction from dynamically-compressed samples is therefore challenging at even modest pressures. Single photon energy dispersive X-ray diffraction (SPEDX) is a relatively new diffraction technique that uses CCD cameras in single photon counting mode to directly record the energy of X-rays diffracted from a laser-compressed sample [3]. Two X-ray CCDs record the diffraction at different Bragg angles, with each camera covering a wide range of k-space, allowing for recording of a significant number of Bragg peaks arising from the sample's crystal structure.

Recent shock experiments on bismuth, combined with *in situ* synchrotron X-ray diffraction, have revealed complex structural dynamics and phase transitions upon shock release [4]. After

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peak compression to the Bi-V phase (body centered cubic; bcc) at $\sim$ 10 GPa, the pressure in the sample then released to ambient pressure via three successive phase transitions – Bi-V (bcc) $\rightarrow$Bi-III (host-guest) $\rightarrow$ Bi-II $\rightarrow$ Bi-I – within 30 ns. More recently, we showed that on shock release from pressures above 11 GPa Bi-V melts within 3 ns [5].

Here we present evidence for phase transitions in shock-compressed bismuth using the SPEDX diffraction technique. Experiments were performed on the Vulcan laser at the Central Laser Facility, RAL, Didcot, UK. We observe diffraction from the (110) bcc peak of Bi-V, and from the measured lattice parameter the sample's pressure was determined to be $\sim$ 17 GPa. Upon further compression (higher laser intensities) no diffraction from crystalline Bi was observed, consistent with the incipient melting thought to occur between 18 and 27 GPa [6]. Also presented and discussed are lower-pressure diffraction data on compressed Bi to $<$ 10 GPa.

2. Experimental methods

The experiment was carried out at the TAW, Vulcan facility (Central Laser Facility, Rutherford Appleton Laboratory, Didcot, UK). Two laser beam pathways were used to either drive the sample target package (with 1 or 2 drive beams) or to generate a broadband X-ray spectrum from a backlighter target foil (Fig. 1). The drive lasers delivered up to 200 J of 527 nm light onto target using a flat top laser pulse with pulse length of 6 ns, which shock-compressed samples to pressures of $\sim$ 20 GPa and below. The drive spot was 3.2 mm in diameter, generating intensities of $\sim$ 10^{11}-10^{12} W/cm^2. Similar laser intensities were shown to shock compress Bi to peak pressures of $\sim$ 14 GPa using the same ablating material [5]. Hydrocode simulations in Gorman *et al.* revealed a steady shock in 15 μm of Bi with a 20 ns pulse length. The flat top laser pulse of 6 ns used in these experiments ensures a steady shock within the diffracting layers of the sample (since X-ray diffraction here is collected in reflection geometry).

Six beams were used to drive the x-ray backlighter, delivering up to 600 J of 1053 nm light in 1 ns with a spot size of $<$ 1.0 mm and intensities of $\sim$ 10^{14} W/cm^2.

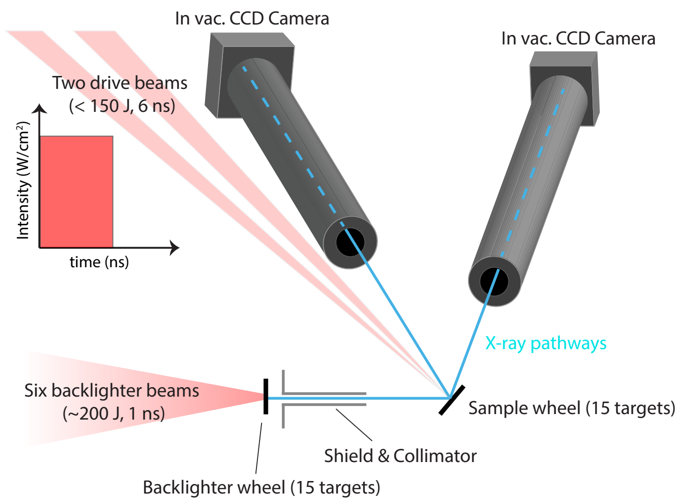

Figure 1. Experimental setup within the TAW target chamber.

Samples consisted of 50 μm Bi metal foil coated with 20 μm of CH-N polyimide ablator with a 100 nm Al flash coating. Backlighter targets (used to generate a quasi-white light source between 3-9 keV) were mixed-metal coatings, produced by the CLF target fabrication group.

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Both sample and backlighter targets were mounted on a multi-target wheel that was remotely controlled to rotate and align new targets after each shot, a technique developed to increase the shot rate during each experimental shift.

Two in-vacuum X-ray CCD cameras were used to collect scattered X-rays at two different Bragg angles. The experimental setup is shown in Fig. 1. The two cameras were placed approximately 50 cm from the sample wheel and were shielded with thick Al tubes to reduce the background noise. Plastic filtering was also added to the end of the Al tubes to preferentially reduce the number of background photons near 3 keV that were generated from the mid-Z backlighter samples. Both cameras were water-cooled *via* an external chiller to minimize the number of dark counts recorded by the CCDs. The X-rays were collimated using a Mo collimator with Pb shielding, thereby ensuring that only an X-ray spot size of ~ 0.5 mm^2 was incident upon the driven area of the sample.

The two drive laser beams were timed with respect to each other such that the samples could be studied before, during and after the shock wave reached the bismuth sample. This allowed us to collect ambient diffraction from undriven targets at early times, diffraction from driven samples at the peak compression, and observe possible phases on shock release from the peak state, such as liquid-Bi or Bi-III / Bi-II, as observed by Hu *et al.* in their synchrotron-based study [4].

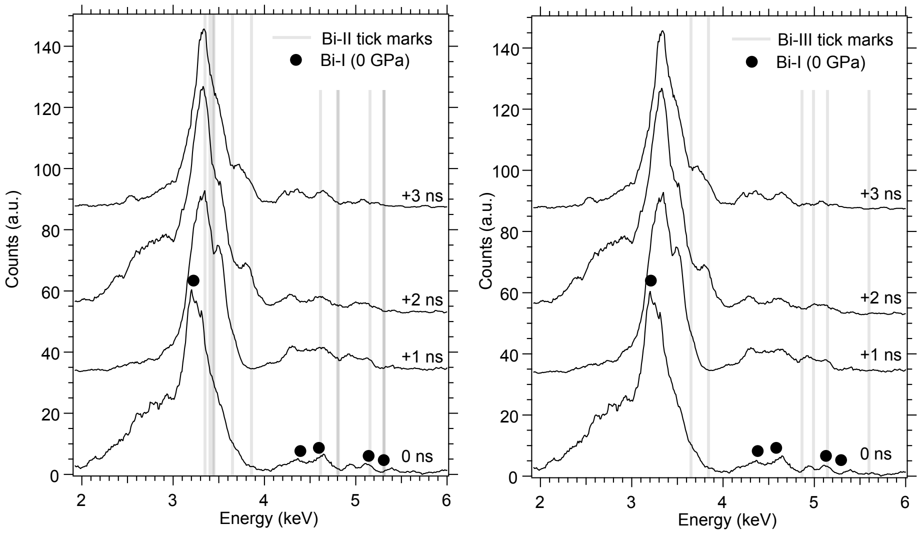

Figure 2. SPEDX diffraction data collected at low pressure P < 5 GPa with tick marks (grey shaded lines) of the Bi-II (left) and Bi-III (right) crystal structures. Filled circles identify the ambient Bi-I diffraction peaks.

3. Results

We first investigated shock-compressed Bi at low pressures (P < 10 GPa) by using laser intensities of $\sim 10^{11}$ W/cm^2. From a previous experiment on Bi we were able to generate a pressure versus laser-intensity calibration for the same target configuration (pressure-intensity calibrated to P < 20 GPa) [5]. Figure 2 shows diffraction data obtained just before the shock entered the Bi (0 ns profile) and then three further diffraction profiles obtained at increasing time delays. These low-pressure data reveal new diffraction peaks that cannot be fitted with either the compressed Bi-I or Bi-V crystal structures. It is possible that the peaks arise from Bi-II, only stable between 2.55 and 2.7 GPa, and the vertical grey lines shown in (Fig. 2 left) show the expected Bi-II peak positions at $\sim$ 2.7 GPa [7]. Whilst there is a reasonable agreement with the observed peaks at +1 ns (Fig. 2 left), at +2 ns a new peak appears at $\sim$ 3.75 keV that does not fit very well to the Bi-II structure. Fits to the Bi-III crystal structure are shown in Fig. 2 right. Both the Bi-II and Bi-III crystal structures have some agreement to the position of this peak (Bi-II peak within 0.6 keV, Bi-III within 0.3 keV), but it should be noted that the Bi-II peak (-201) has a calculated intensity that is significantly weaker than the strong Bi-III (310) peak at this energy.

When the laser intensity was increased to beyond $\sim 10^{12}$ W/cm^2 heavy filtering of the CCD cameras was required in order to reduce the large drive noise at 3-4 keV (Fig. 3). For data collected using such drive laser intensities, the total number of counts in the signal was significantly reduced and some weak fluorescence peaks were also observed. The position of these fluorescence peaks on the CCD cameras are independent of the Bragg angle (the peaks are present at the same energy on both cameras) and we can therefore easily distinguish them from the diffracted Bi photons. Figure 3 shows an example of data collected from a driven target that shows evidence of the high-pressure Bi-V phase. The fluorescence peaks arising from the backlighter foils are identified in Fig. 3 with filled circles above them. By knowing the Bragg angle of each detector (obtained using ambient diffraction from Ta/Bi and calibrating the detector angles), the peak position of the Bi-V (110) reflection ($\sim$ 4.03 keV at 35°) can be used to determine the lattice parameter for the driven sample, which was found to be 3.68 Å. From the known equation of state of Bi [8], this gives an estimated sample pressure of $\sim$ 17 GPa. All of the remaining peaks can be fitted to the ambient Bi-I phase arising from uncompressed material ahead of the shock wave (rectangular tick marks in main figure).

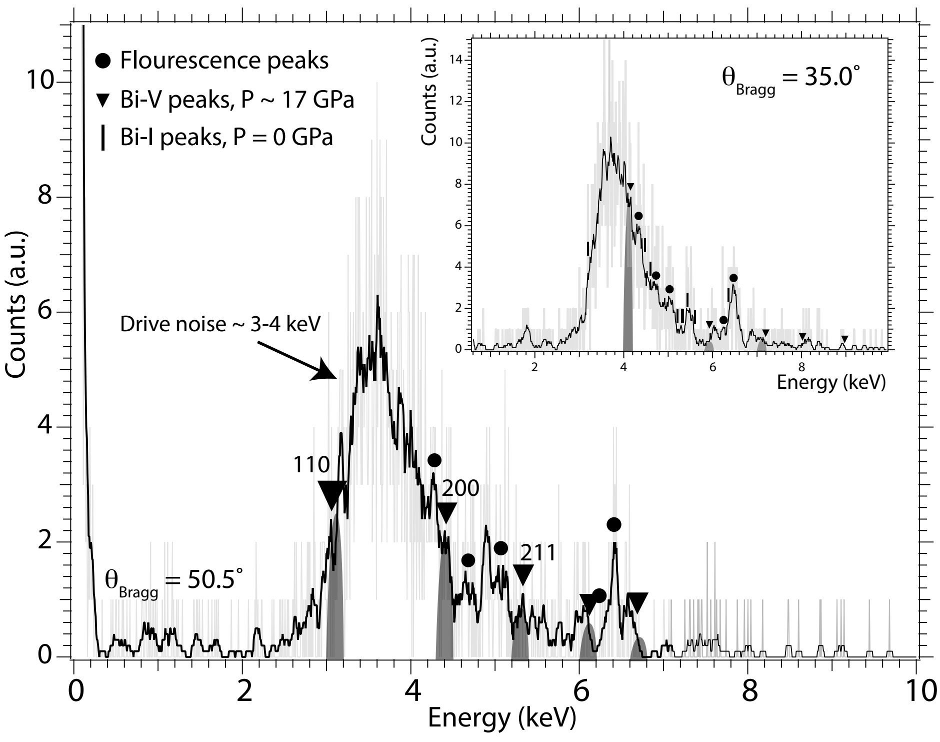

Figure 3. Diffraction data collected from the two CCD cameras at different Bragg angles. Triangles indicate the location of Bi-V peaks at a pressure of 17 GPa. Filled circles identify peaks that are present at the same energy on both cameras, and which are therefore fluorescence peaks from the backlighter foil.

Recent experiments carried out at the LCLS x-ray free electron laser (XFEL) indicate that melting of the bismuth should also be observed on shock release from pressures above 11 GPa [5]. Unfortunately, the weak diffraction signal from the liquid is difficult to extract in the current experiment as it is overwhelmed by diffraction from several high-pressures phases. In some later shots in the campaign, we used higher laser energies than those where the Bi-V phase was observed and found that we were no longer able to observe any Bragg peaks from the Bi-V phase. This could be an indication that shock melting had occurred (previously reported to occur between 18 and 27 GPa [6]) as the Hugoniot crosses the melting curve. However, the drive noise was significantly higher for these data collections and the expected liquid diffraction peak is overlapped by the drive noise. In several cases the detectors were also unable to operate in single photon counting mode, since excessive numbers of noisy pixels from the drive saturated the CCD camera, overwhelming the diffraction signal from the liquid.

4. Conclusions

X-ray diffraction studies of bismuth under shock compression have been carried out and reveal phase transitions at low pressure ($P < 10$ GPa) and observe the high-pressure Bi-V phase at $P \sim 17$ GPa, the highest pressure that Bi-V has been observed on the shock Hugoniot. It is not yet clear as to what structure/structures the samples transform to at low pressures, and further work is planned to investigate the lower-pressure phase transitions using different detector angles. This would both improve our ability to distinguish Bi-II and Bi-III peaks, and move key Bragg peaks away from the region of drive noise for the highest energy drives. We will plan to investigate using the SPEDX technique in a transmission geometry, using thinner sample targets, thereby completely removing the drive noise signal present on the CCD camera, and enabling the weak liquid diffraction signal to be observed.

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