ESRF	**Experiment title:** Phase transitions and crystal structures of Bi-Te intermetallic compounds at high pressure	**Experiment number**: HS-4718
Beamline: ID09a	**Date of experiment**: from: 21 February 2013 to: 24 February 2013	**Date of report**: Sept. 2013
Shifts: 9	**Local contact(s)**: M. Hanfland	*Received at ESRF:*

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Report

We were awarded three days of beamtime on station ID09a to study the phase transitions and crystal structures of BiTe, Bi_2Te and Bi_4Te_3 under high pressure up to ~20 GPa by x-ray diffraction. These materials are members of the Bi_xTe_y family of intermetallic compounds, several of which are good thermoelectric materials [1, 2]. Bi_3Te_4 is also known to become superconducting at high pressure [3]. Earlier high-pressure x-ray diffraction studies on Bi_2Te_3 and Bi_3Te_4 indicated the existence of phases with complex crystal structures in the pressure range to 20 GPa, but the structural details of most of these phases remained unsolved. The high-pressure behaviour of the other members of the Bi_xTe_y family was completely unexplored.

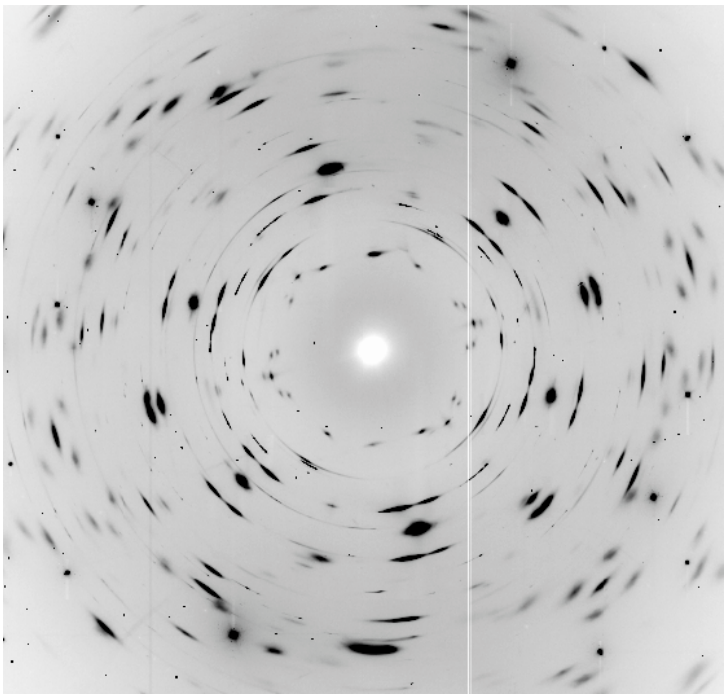

Figure 1: Diffraction pattern (±33° oscillation) of a quasi-single-crystal of Bi_4Te_3 at 10 GPa.

We collected a combination of powder and single crystal x-ray diffraction data of BiTe, Bi_2Te and Bi_4Te_3 up to pressures of 15–25 GPa. All samples were loaded in diamond anvil cells for pressure generation with either helium or nitrogen as the pressure-transmitting medium. Numerous phases were observed at high pressure, and the analysis of the diffraction data and the determination of the crystal structures are in progress. All three samples eventually adopted a disordered bcc alloy structure at the highest pressures, as reported previously for Bi_2Te_3 and Bi_4Te_3 [4, 3].

As expected, the diffraction patterns of several of the intermediate phases turned out to be rather complicated. Some of these complex-structured phases appear to have monoclinic lattices with at least one long axis of ~20 Å, and indexing of the corresponding diffraction patterns is challenging. Therefore, we attempted to pick quasi-single-crystal grains from the polycrystalline as-grown samples so as to collect

quasi-single-crystal diffraction data to aid the determination of the crystal lattices. This was successful in the case of Bi_4Te_3, the material that becomes superconducting above ~6 GPa [3]. During the beamtime, we succeeded in finding a high-quality single-crystal grain of this compound in the polycrystalline as-grown material, in loading it into a high-pressure cell with nitrogen as the pressure-transmitting medium, and in collecting diffraction data to 10 GPa. While the sample quality deteriorated significantly at the first pressure-induced phase transition, the diffraction pattern of the sample grain in the high-pressure phase at 10 GPa is still single-crystal-like (Fig. 1), and in comparison to the corresponding powder diffraction pattern, it provides significant additional information. Attempts to collect single-crystal data also for BiTe and Bi_2Te started promising, but we were unable to complete this within the allocated beamtime.

Overall, the experiment was successful, and by collecting high-pressure x-ray diffraction data from BiTe, Bi_2Te and Bi_4Te_3, we achieved the principal aims stated in the proposal: (i) to explore the possibility of pressure-induced structural complexity in these compounds, as part of the search for better-performing thermoelectrics, and (ii) to collect the data necessary to determine in detail the evolution of the structural parameters as a function of pressure so as to provide a basis for understanding the physical properties of these compounds at high pressure.

Please note that some specific results have been omitted from this publicly available report.

References

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