ESRF	**Experiment title:** Incommensurate and Liquid Tellurium at High Temperatures and Pressures	**Experiment number**: HS-2748
Beamline: ID09	**Date of experiment**: from: 13/06/2005 to: 17/06/205	**Date of report**: 25/02/07
Shifts: 9	**Local contact(s)**: M. Hanfland	*Received at ESRF:*

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Report: Recently, we have shown that the structure of the high-pressure phase Te-III, stable between 7 and 27GPa, is not orthorhombic as previously claimed, but monoclinic with an incommensurate structure containing surprisingly large modulations of the atomic positions [1]. This structure type was previously unknown in the elements, and Te-III, Se-IV and S-III (in which we have found the same incommensurate structure) are three of only five known simply modulated elemental structures. The known phase diagram of tellurium shows Te-III existing up to the melting curve, and the incommensurate nature of the structure will then have important implications for studies of liquid-Te (l-Te), where the structures of the crystalline phases have been essential in interpreting diffraction data from the liquid phases. The very different structure for Te-III thus opens the way to a reinterpretation of the structures of l-Te at high pressure, in particular those involved in the liquid-liquid phase transition reported at 4-5GPa.

In this proposal we asked for 3 days of beam time on ID09 to follow the structure of Te-III at high temperatures, (i) to determine the T-dependence of the incommensurate wavevector and atomic modulations, and (ii) to monitor the changes in structure, coordination and density that accompany the onset of melting of Te-III at ~800K.

Powder diffraction data were collected from 3 different samples over a wide range pf P and T up to 21GPa and 743 K, as illustrated in Fig. 1. We found the incommensurate Te-III phase to be stable up to at least 743K, and determined the P-T dependence of the incommensurate wave vector q, as shown in Fig. 2. q is clearly strongly dependent on both P and T. As reported previously, q initially decreases on compression at room temperature before reaching a minimum and then increasing again. Similar behaviour is observed on compression at higher temperatures. q is also strongly temperature dependent, and decreases with increasing temperature, with the change being largest at lower pressures.

In addition to determining the P-T dependence of the incommensurate Te-III, we found that the rhombohedral β-Po phase of Te, Te-IV, previously reported to exist at room temperature, is stable only above 315 K (Fig. 3). The revised phase diagram for Te to 35GPa and 900K, including the newly-observed Te-IV phase, is shown in Fig. 4. Attempts to study liquid-Te were, unfortunately, unsuccessful. Although we were able to reach the P-T conditions of the liquid state (see Fig. 1), this phase reacted very rapidly with the metallic gaskets used in the pressure cells making data analysis impossible.

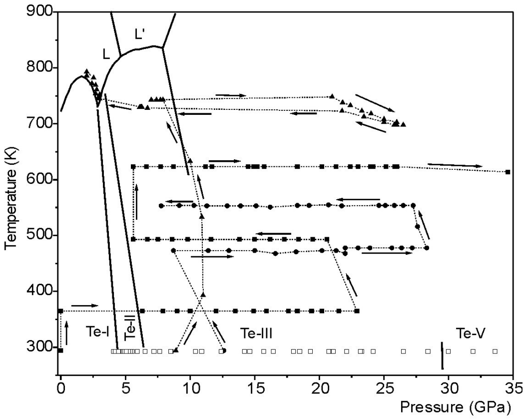

Fig 1: The reported *P*-*T* phase diagram of Te, showing the *P*-*T* paths taken by the three samples.

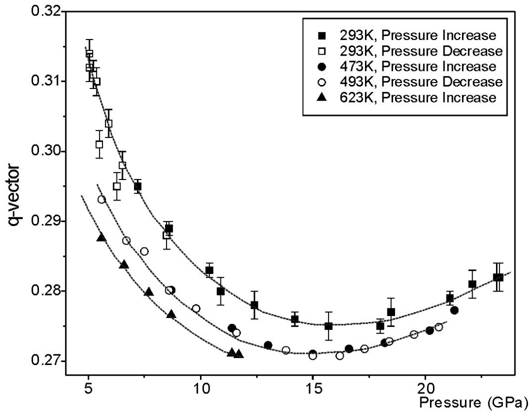

Fig 2: P dependence of the incommensurate wave vector q in Te-III at room temperature, 473/493 K, and at 623 K.

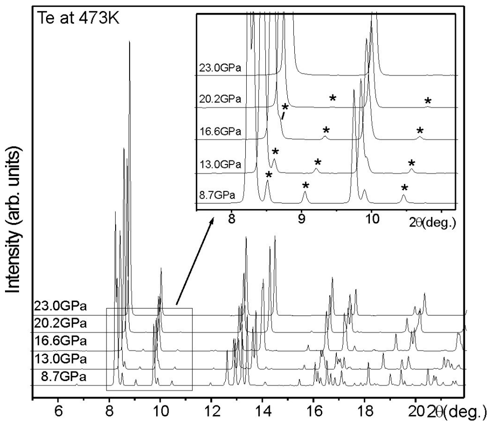

Fig 3: Diffraction profiles collected from Te as a function of pressure at 473 K, showing the transition from monoclinic Te-III to rhombohedral Te-IV between 22.0 and 23.0 GPa.

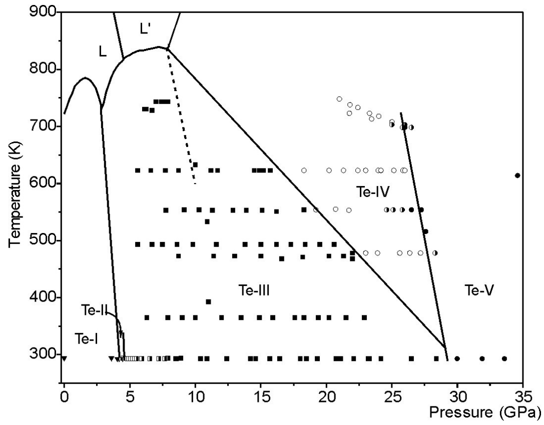

Fig 4. Revised phase diagram of Te to 35 GPa and 900K.

The results of this experiment were published in Physical Review B in November 2006, as detailed below.

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Phase transitions in tellurium at high pressure and temperature

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The structural phase transitions in tellurium have been studied up to 21 GPa and 743 K using powder-diffraction techniques. We find the incommensurate Te-III phase to be stable up to at least 743 K, and have determined the *P*-*T* dependence of the incommensurate wave vector q. We also find that the rhombohedral β-Po phase of Te, Te-IV, previously reported to exist at room temperature, is stable only above 315 K.

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References:

[1] C. Hejny and M. I. McMahon, Phys. Rev. Lett. **91**, 215502 (2003).