	Experiment title: Structures and Transitions in Metals at High Pressure	**Experiment number**: HS1128
Beamline: ID9	**Date of experiment**: from: 28/4/00 to: 29/4/00	**Date of report**: 29/8/00
Shifts: 3	**Local contact(s)**: M. Hanfland	*Received at ESRF:*

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Report:

Experiment HS1128 was originally scheduled for 12 shifts of beamtime on beamline ID9. However, the failure of vital experimental equipment immediately before the beamtime prevented the very high pressure experiments on Ca and Sr originally planned.. Only 3 shifts of the beamtime were therefore scheduled, and were used to make studies of elements with structures closely related those in the original proposal. HS1128 was resubmitted in the following proposal round and the beamtime has been rescheduled on ID30 for early 2001.

Recently, using angle-dispersive diffraction data collected at the SRS and ESRF synchrotrons, we have found that the high-pressure phases of Ba and Sr have an entirely new elemental structure type [1,2]. This new structure comprises a body-centred tetragonal `host' structure with channels along the c-axis (Figure 1). Contained in these channels are chains of atoms from one or more 'guest' structures that are incommensurate with the host along c. In barium we found that these guest structures could undergo structural phase transitions without any accompanying change in the host, and termed this an intra-phase transition. Subsequent studies at the ESRF suggested that strontium too has an intra-phase transition at 71GPa.

While the incommensurate host-guest structure was initially found in the group-II alkaline earth metals, the structure bears a striking resemblance to a (commensurate) tetragonal structure reported to be stable in the group-V elements Bi, Sr and As at high pressure. Diffraction studies at

SRS have now revealed that for all three elements, the incommensurate host-guest structure provides a considerably better fit to the diffraction data, and correctly calculates the known density of B-III.

However, in each element, the SRS diffraction patterns contain very weak extra reflections that cannot be accounted for with a host and (single) guest structure. The extremely high intensity available at the ESRF was used to collect high quality data from each of the host-guest phases with the aim of locating all of these extra reflections in order to determine whether they arise from a second guest phase.

Diffraction data were collected from each of the host-guest phases – Bi-III (stable between 2.5 and 7.7GPa), Sb-II III (stable between 8.5 and 28GPa) and As-III (stable between 48 and 97GPa). A Rietveld refinement of a diffraction profile from Sb-II is shown in Figure 1, with the extra reflections enlarged and indicated in the inset. The additional reflections in Bi-III are weaker, but in the same relative positions, suggesting that the same structure is responsible for the diffraction peaks in both Sb and Bi. The extra reflections in As are considerably weaker and, indeed, are only just visible. Those that are visible, however, are clearly in different positions to those observed in Sb and Bi, suggesting they arise from a different structure.

Attempts to determine the additional structures responsible for the extra reflections are still ongoing. However, it is clear that the solution of their structures will only be possible from the extremely high-quality data collected on ID9.

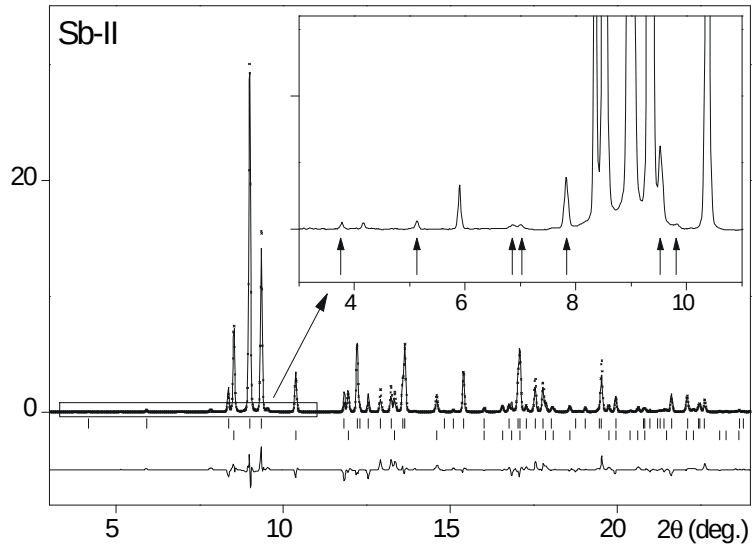

Figure1. Rietveld fit to the host-guest structure of Sb-II at 10.3GPa. Some of the low-angle additional reflections not accounted for by this structure are indicated in the inset.

References:

[1] R.J. Nelmes, D.R. Allan, M.I. McMahon and S.A. Belmonte, Phys. Rev. Lett. **83**, 4081 (1999).

[2] M. I. McMahon, T. Bovornratanaraks, D. R. Allan, S. A. Belmonte, and R. J. Nelmes, Phys. Rev. B **61**, 3135 (2000).

[3] O. Degtyareva, M.I. McMahon and R.J. Nelmes, Submitted to Physics Review (2000).

[4] H. Iwasaki and T. Kikegawa, Acta Cryst. B**53**, 353 (1997).