ESRF	**Experiment title:** High pressure-high temperature study of elemental titanium	**Experiment number**: HC-839
Beamline: ID27	**Date of experiment**: from: 26/07/2013 to: 31/07/2013	**Date of report**: 24/09/2013
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Report:

Titanium is controversial in its phase transformations at high pressure. Several phases had been observed experimentally, which could have been favoured by non-hydrostatic compression. The aims of this project were: (i) establishment of a reference experimental transition sequence for Ti at ambient T; (ii) determination of the solid high pressure-high temperature phases; (iii) measurement of the Ti melting curve. For that purpose, we have combined isothermal quasi-hydrostatic and high temperature (laser-heating) measurements in a diamond anvil cell.

Cell name	Pressure medium	P range (GPa)	T range (K)	P gauge
CDMX19	He	49-109	300	Ruby
CDMX12	He	1-139	300	W
CDMX8	NaCl	1-15	300-2100	NaCl
CDMX15	KCl	30-75	300-3500	KCl, MgO
CDMX7	KCl	50-150	300-4000	KCl

Table 1: conditions of each experimental run.

Table 1 summarizes the conditions of the experimental runs performed during HC-839. Monochromatic X-rays were focused down to a 2 × 3 spot size, scattered off the samples, and collected on a MAR-CCD bidimensionnal detector. Laser heating was performed simultaneously with diffraction data collection, using two yttrium aluminum garnet (YAG) lasers which provide a maximum power of 200 W. The temperature was determined from spectral radiometry measurements. The pressure was estimated using pre-calibrated gauges: ruby or X-ray gauges (W, NaCl, KCl, MgO).
Our measurements made on cold compression in quasi-hydrostatic conditions (first two runs) confirmed the sequence of phase transformations already reported [1]; in particular, the appearance of γ-Ti and δ-Ti. These phases are not observed for other group IV metals (Zr and Hf). Laser-heating runs showed that the bcc structure of Ti (β-Ti) is stable below the melting line in a wide pressure range (0-150 GPa at least) (see

Figure 1). However, β-Ti is not observed at ambient temperature, either by cold compression or quench after heating, which contradicts theoretical predictions [2]. We did not confirm the observation of η-Ti [3] and ω-Ti is stable in a wide pressure range (2-116 GPa). On the transformation α-Ti -> ω-Ti, we have observed orientation relations which will help elucidating the mechanism of this martensitic phase transformation.

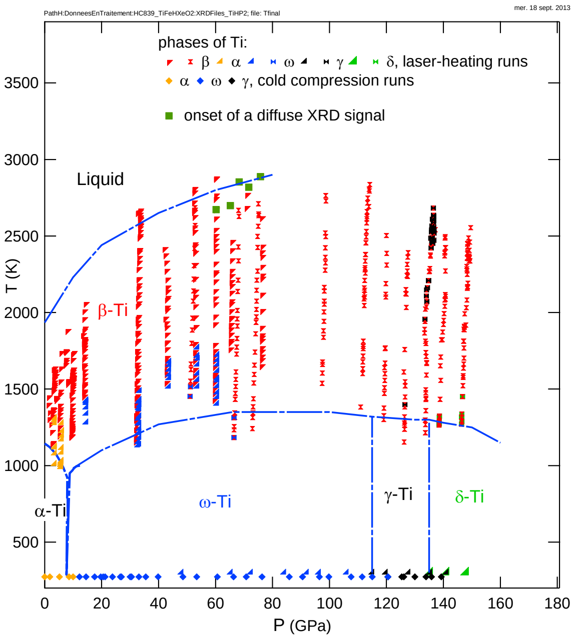

Figure 1: P-T conditions at which X-ray diffraction data points have been recorded for solid titanium. The temperature has been measured by pyrometry. The pressure has been measured using the pressure gauges specified in Table I. The symbols color indicates the phase of Ti identified by X-ray diffraction. For several data points, two phases have been observed on the same X-ray diffraction pattern which can be explained by temperature gradients within the sample. The blue dashed lines are tentative phase boundaries.

The melting curve measured in this study using the onset of a diffuse X-ray diffracted signal for the sample is up to 500 K higher than previous measurement based on an optical detection of melting [4].

The isothermal equation of state of ω-Ti has the following parameters: K0=106.9, K'0=3.68. K0 is lower than previous measurements but agrees with density-fonctional theory energy calculations [2].

References:

[1] Y. Akahama, H. Kawamura, and T. L. Bihan, New δ (Distorted-Bcc) Titanium to 220 {GPa}, Phys. Rev. Lett. 87, 275503 (2001).
[2] Z.-G. Mei, S.-L. Shang, Y. Wang, and Z.-K. Liu, Density-Functional Study of the Thermodynamic Properties and the Pressure–temperature Phase Diagram of Ti, Phys. Rev. B 80, 104116 (2009).
[3] R. Ahuja *et al.*, Titanium Metal at High Pressure: Synchrotron Experiments and Ab Initio Calculations, Phys. Rev. B 69, 184102 (2004).
[4] D. Errandonea *et al.*, Systematics of Transition-Metal Melting, Phys. Rev. B 63, 132104 (2001).