	Experiment title: High pressure phase diagram of titanium under hydrostatic Conditions	**Experiment number**: HS-3797
Beamline: ID09A	**Date of experiment**: from: **11/07/09** to: 13/07/09	**Date of report**: 03/09/09
Shifts: 6	**Local contact(s)**: M. Hanfland	*Received at ESRF:*

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Report:

The first part of our experiment has been devoted to the study of titanium quasi-hydrostatically compressed up to very high pressure. In fact, several phase transitions have been observed under non-hydrostatic compression [1,2,3], but the lack of reproducilility of experimental findings suggests that non-hydrostatic compression may have influenced the behaviour of Ti. No precise equation of state has been measured in this pressure range.

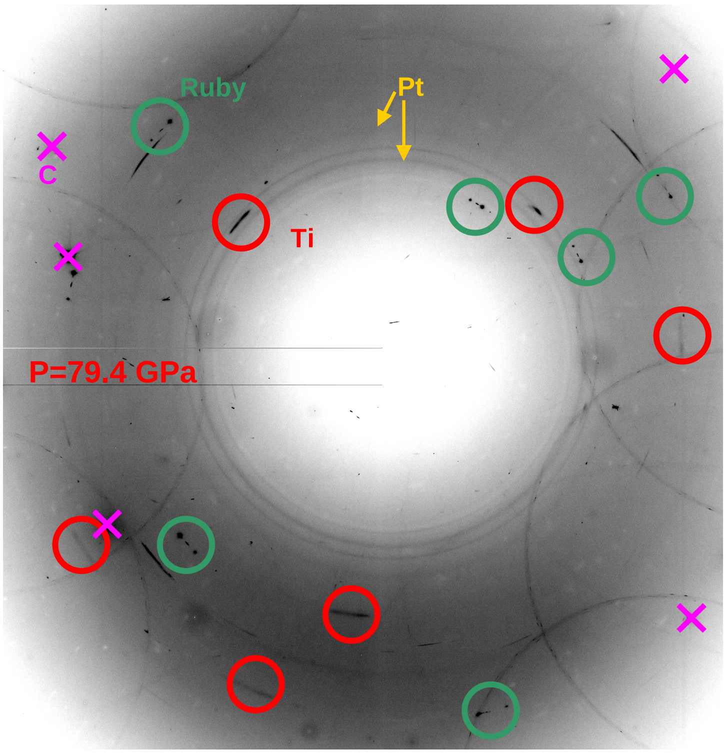
We have prepeared a diamond anvil cell, loaded with Ti, ruby and Pt (both pressure markers) embedded in helium pressure transmitting medium. We have used angle-dispersive X-ray diffraction (λ=0.4155 Å) with a MAR imaging plate. Pressure was primarily estimated from ruby luminescence signal.

We have reached 165 GPa with this cell**,** taking two x-ray exposures (one on the titanium sample, one on the Pt grain) every ~7 GPa. An x-ray diffraction spectrum recorded at 79.4 GPa is presented Figure 1. The signals from the different samples, and diamond anvils, can easily be recognized. Unfortunately, above 110 GPa the signal from the titanium sample became very weak. The analysis is in progress. Preliminary results do not evidence any phase transformation in titanium under that pressure. On figure 2, the evolution of the c/a ratio for ω-Ti is presented together with a low pressure run (performed a few years ago on the ID30 beamline) results. The two runs lead to measurements which are in excellent agreement.

In the second part of our experiment, we have determined the pressure conditions of the α-Ti -> ω-Ti phase transformation under high temperature. In fact, despite the relatively low pressure conditions of this transition, its conditions have not clearly been characterized, its measured pressure typically varying between 2 and 12 GPa at ambient pressure.

A diamond anvil cell with 600 microns culets diamonds, loaded with titanium and SrB_4O_7 luminescence pressure gauge in neon was used. It was resistively heated up to 723 K; neon pressure medium remained liquid in the scanned pressure range (1-17 GPa). The titanium sample was then compressed under pure hydrostatic conditions.

We have observed that a large hysteresis exists for the α–ω transformation, even at that temperature. The direct and reverse transformation pressure differ by ~5 GPa. We were planning to follow one more isotherm but we did not have enough beamtime to finish this experiment.



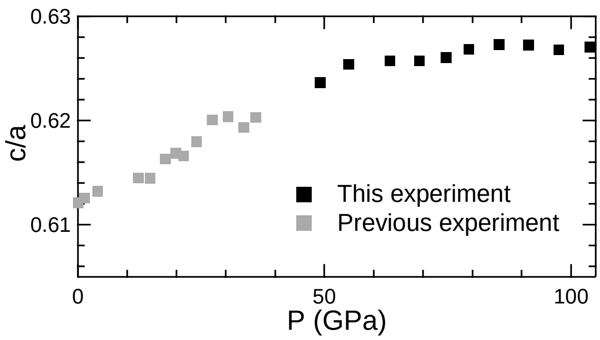


Figure 1: X-ray diffraction spectrum recorded at 79.4 GPa **Figure 2:** evolution of the c/a ratio for ω-Ti with P

[1] Y. K. Vohra and P. T. Spencer. Novel γ-Phase of Titanium Metal at Megabar Pressures. Phys. Rev. Lett. **86**:3068 (2001).
[2] Y. Akahama, H. Kawamura, and T. Le Bihan. New δ (Distorted-bcc) Titanium to 220 GPa. Phys. Rev. Lett. **87**:275503 (2001).
[3] R. Ahuja *et. al*. Titanium metal at high pressure: Synchrotron experiments and ab initio calculations. Phys. Rev. B **69**:184102 (2004).