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# Experiment Report Form

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If your report is to support **a new proposal**, the orginal report form should be sent with the new proposal form, and a copy of your report should be attached to each copy of your proposal. The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

***Reports on experiments relating to long term projects***

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## Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

## Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.
- bear in mind that the report will be reduced to 71% of its original size. A type-face such as "Times", 14 points, with a 1.5 line spacing between lines for the text, produces a report which can be read easily.

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| ESRF | **Experiment title:** Is osmium stiffer than diamond? An ultra high accuracy study | **Experiment number**: HS-2152 |
|---|---|---|
| **Beamline**: ID09A | **Date of experiment**: from: 12/04/2003 to: 15/04/2003 | **Date of report**: |
| **Shifts:** | **Local contact(s)**: M. Hanfland | *Received at ESRF:* |

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## Report:

In a early study of the Equation of State (EoS) of Osmium, Cynn *et al.*[1] inferred that this 5d hcp transition metal was stiffer than diamond (a bulk modulus of 462 GPa was found for Os, to be compared with 446 GPa for diamond[2]). However, the experimental procedure Cynn *et al.* used (energy dispersive diffraction on a powder, very few data points, and Ar used as a pressure transmitting medium up to 65 GPa) seemed us to be too insensitive to support their conclusions. In order to clarify the question of whether or not a metallic bond could be stiffer than a covalent bond, we were allocated 9 shifts of beamtime on ID09A in April 2003. Fully following our proposal, we performed the experiment using a state-of-the-art experimental method including the use of the very sensitive angle-dispersive x-ray diffraction (ADX) technique and the use of helium as a pressure transmitting medium. The pressures were measured using NaCl from 0 to 26 GPa and thus the ruby fluorescence pressure scale up to 75GPa. Emphasis was given to the collection of a large number of data points in the low pressure range by small steps for a better constrain of the bulk modulus value. The use of the ADX technique permitted us to record 41 diffraction patterns at various pressures where twelve osmium reflections are resolved with a signal/noise ratio reaching 50 for the most intense line. An important feature of this x-ray technique is that it makes it possible to rely on the reflections relative intensities and then to process the data using a crystal structure refinement procedure based on the Rietveld method[3,4]. This further permitted us to make sure no structural phase transition occurs within our pressure range and to lower *a minima* the errors on the cell parameters values. Figure 1-a shows typical diffraction patterns, and figure 1-b shows the resulting EoS. A least-square fit of this EoS using Vinet formalism[5] yields $K_0$=410±6GPa, $K_0$'=4.2±0.2, and $V_0$=27.943A$^3$ while a 3$^{rd}$ order Birch-Murnaghan fit yields $K_0$=411±6GPa, $K_0$'=4.0±0.2, and $V_0$=27.945A$^3$. An additional fit of our dataset using a Reverse Monte Carlo algorithm to the third order B-M fit yields $K_0$=410.9±1.2 GPa, $K_0$'=3.97(4), and $V_0$=27.9490(14) A$^3$. These results show that i) Osmium is indeed a very stiff material but still less stiff than diamond ii) the classical high pressure standards yield a $K_0$' value that is close to 4, which is expected for very stiff materials: this supports the validity of the ruby pressure scale (see [6,7] for more details about this issue).

However, these first conclusions about Os structural properties might have to be reconsidered soon: Figure 2 shows the c/a ratio value of the Os hcp unit cell plotted as a function of the reduced volume. It is seen a faint line break at 0.95 (⇔20 GPa) that seems to be reproduced over all of the 3 samples we have used for achieving this project. But that feature remains within our experimental error bars. If this line break did be real, it could be the sign that a topological phase transition (a.k.a. Lifschitz transition[8]) occurs at 20 GPa in Os, which i) would result in a change of Os elastic constants at 20GPa and thus invalid the EoS parameters given above ii) would give a new insight into the hcp transition metals properties in that a major argument in high pressure physics deals with the existence or the non-existence of such a transition in Zn at 6 GPa[9].

We won't ask for more beamtime for this project in the next run: our dataset has already reached the highest possible precision of measurement to date. A deep improvement of high pressure metrology techniques will be necessary to definitely address this phase transition issue. An article detailing the results summarized herein is to be submitted soon.

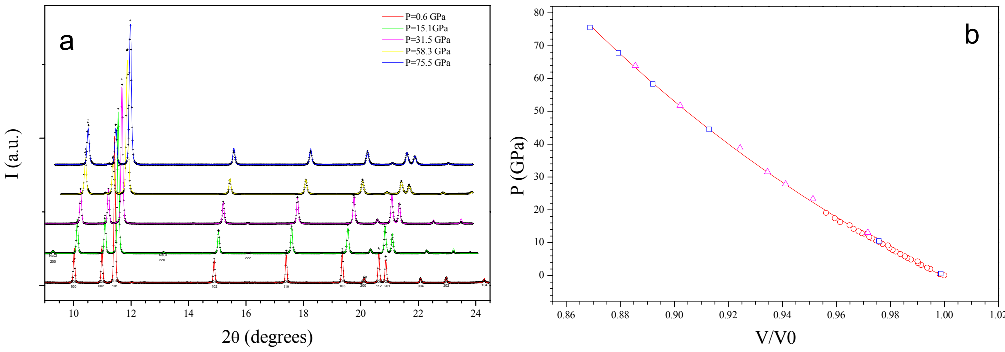


Figure 1: a) typical diffraction patterns recorded along this experiment - b) the resulting equation of state.

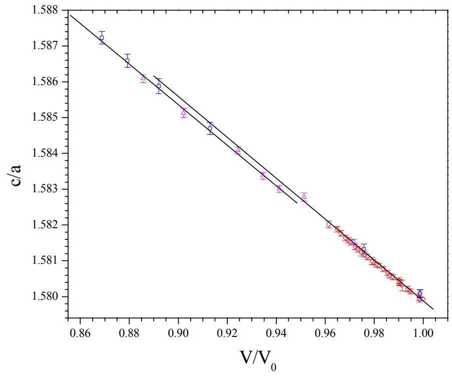


Figure 2: c/a ration of Os hcp primitive cell as a function of the reduced volume. A faint line break is seen at 0.95, which corresponds to 20 GPa. The 3 different symbols correspond to our 3 different samples. The lines are guides for the eye.

References:

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