EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

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

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

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.

ESRF	**Experiment title:** Melting of MgO to P~50 Gpa by *in situ* diffraction and CO_2 laser-heating DAC studies	**Experiment number**: HS-4484
Beamline: ID27	**Date of experiment**: from: 22/10/11 08:00 to: 25/10/11 08:00	**Date of report**:
Shifts: 9	**Local contact(s)**: Dr. Ashkan Salamat	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The aim of this investigation was to study the melting point of MgO up to pressures ~ 50 GPa and beyond by conducting laser-heated diamond anvil cell (LH-DAC) experiments using CO_2 laser heating combined with time resolved synchrotron x-ray diffraction. The CO_2 laser-heating system for *in situ* LH-DAC/XRD experiments was only recently installed at ID27, and this is the only beamline currently available to perform this type of experiment worldwide.

MgO is a major component of the Earth's lower mantle and knowledge of its melting properties at high pressure (P) and high temperature (T) is fundamental for evaluating phase relations and mantle rheology. It is also an important refractory ceramic and determination of its high-P,T properties is essential for understanding its behaviour as a structural material in environments combining high mechanical stress and high T conditions. The melting point of MgO at ambient P is T_m = 3125 K. *Ab initio* simulations predict that this should rise to T_m ~6000 K by 50 GPa (dash line in Fig 1). However, in the one previous experiment carried out to date using the CO_2 LH-DAC technique combined with monitoring the T *vs* laser power curve to indicate the onset of melting, T_m only achieved <4000 K by 35 GPa and is extrapolated to rise only little above that by 50 GPa (circles and solid line in Fig. 1). That represents a serious discrepancy between theoretical and experimental results, of >2000 K by 50 GPa. Such a discrepancy must be resolved in order to understand the high-P,T properties of this important mantle mineral and refractory ceramic material. We proposed to study this using synchrotron XRD as a diagnostic of melting from the first appearance of liquid S(Q) and disappearance of crystalline Bragg peaks in the pattern, under LH conditions in the DAC. The IR radiation used to heat the sample was supplied by a CO_2 laser (λ ~ 10.6 μm) in order to couple directly with phonon modes of the sample. As an optically transparent insulating material it does not directly absorb near-IR radiation supplied by the Nd^{3+}:YAG or YLF lasers in common use for LH-DAC experiments. This run was meant to be one of the first trials using the CO_2 LH-DAC system installed at ID27 for on-line experiments.

We initially requested 15 beamtime shifts to attempt these challenging and pioneering experiments. However, the allocation committee assigned us only 9 shifts. Since this was an experiment using a new experimental setup we were informed by the beamline scientist and team that our run would be scheduled at the beginning of a cycle dedicated to trialling the new CO_2 laser heating system. However when we arrived, we discovered that the previous user group had mistakenly requested CO_2 in their project proposal, when in fact they required Nd^{3+}:YAG/YLF heating lasers. That meant the local contact had to break down the CO_2 LH system to re-install the near-IR laser optics for their run. The result is that we lost a further 9 hours of crucial beamtime, as we had to re-install and re-align the CO_2 LH optics, that are quite different to the near-IR system. We noted in our user evaluation form for the run that the beamtime allocation committee as well as prospective users should pay sufficient attention to the laser system that is actually required for a given experiment.

Despite these setbacks we managed to obtain some data. We successfully heated several samples in the 3000-3750 K temperature range, but did not observe melting (vertical dotted lines in Fig. 1. represent P,T paths followed in some of our LH experiments). This apparently negative result may indicate that the previous Zerr and Boehler (ZB) determinations are likely too low, however, within experimental error we could be below the ZB melt line (Fig. 1). The promising aspect of these initial data is that we do not see the usual signs for the onset of melting in this region such as a decrease in intensity of the MgO peaks and/or rapid recrystalisation. In our new proposal we now request additional beamtime to continue these experiments and obtain new melting data on MgO up to and perhaps above 50 GPa.

During our initial runs we also gained valuable information on the sample and pressurisation environment that will feed into our new proposal. A critical component of LH-DAC experiments is the ability to thermally insulate the sample from the diamond anvils. This became a problem during our runs since all of the common pressure-transmitting media (KBr, LiF, NaCl, Ar etc) melt well below the ambient P melting point of MgO. We tried initial runs using LiF and KBr. The LiF melted and caused substantial motion of the MgO sample within the DAC chamber. However, the KBr was acceptable: although it exhibited partial melting, the sample migration was not substantial and we could obtain XRD patterns throughout the LH-DAC experiment. Nonetheless, because KBr contains high-Z elements any peak overlap could obscure the signal from MgO. In our future experiments we will select specific pressures where no peak overlap should occur for the wavelength used.

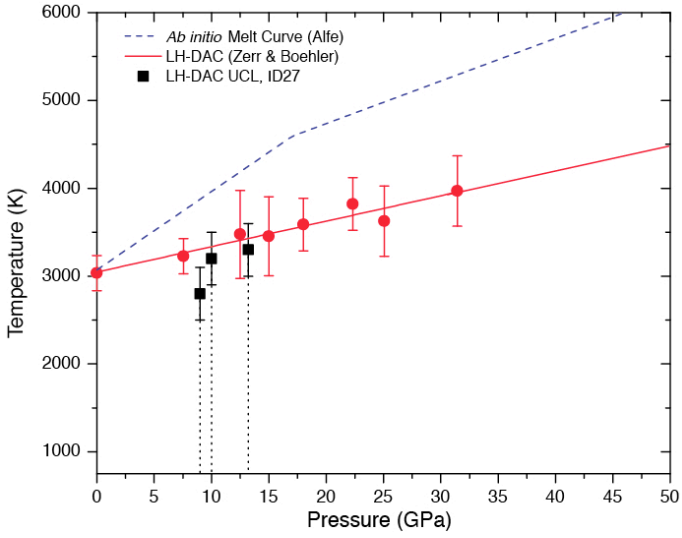

Fig. 1: Melting curves determined for MgO using *ab initio* theoretical techniques (dash line); ZB melting curve from experimental LH-DAC techniques (circle & line); in black (squares) we show some of our initial data collected where only solid MgO is observed in the X-ray diffraction patterns for the P,T path displayed.