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 using the Electronic Report Submission Application:

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now 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.

	Experiment title: Melting line and evolution of the structure factor of liquid Gold to 140 GPa.	Experiment number: HC 2187
Beamline: ID27	Date of experiment: From: 05/11/2015 to 09/11/2015	Date of report: 17/05/2016
Shifts: 18	Local contact(s): G. Garbarino	Received at ESRF:

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The goal of this experiment was to determine the melting curve and measure the liquid structure factor of Gold up to 140 GPa. Due to its exceptional chemical inertness and structural stability, gold is a widely used high-pressure standard and is an important material in many applications. The melting line of Gold has been investigated by several authors in a large-volume apparatus up to 6-7 GPa, using the change of the sample electrical resistance to detect melting. The same criteria has also been used in recent experiments using the resistively heated DAC up to 9 [Zha] and 21 GPa [Weir]. To our knowledge there is only one attempt to determine melting in the LHDAC, to 35 GPa [Pippinger]. While all these melting lines roughly agree within their uncertainties below 15 GPa, there is a 200 K difference between the resistive and laser heating DAC measurements around 20 GPa. Furthermore these two works suggest very different slopes of the melting lines above 15 GPa. Finally, the high pressure evolution of liquid gold is totally unknown.

Experimental method

Five membrane diamond anvil cells were equipped with diamond anvils with culet sizes ranging from 400μm to 150μm. The cells were prepared with a gold sample confined in a capsule formed by two boron-doped diamond bowls. Each bowl was deposited in a pit, realized by Focussed Ion Beam, at the center of the culet of the diamond anvils. Argon was used as a pressure transmitting medium since its melting curve crosses gold's at about 40 GPa. In the first cell equipped with 300 microns culet, we increased the pressure to 40 GPa in order to melt the gold sample while keeping the argon medium solid. The capsule was then heated several times up to a maximum temperature of 3000 K, taking care to equilibrate temperature over the two sides of the sample. Thermal expansion, solid recrystallization and melting events were observed but we couldn’t obtain a fully melted sample signal. We think that the bowls were damaged during the pressure

increase, so that the sample was not sufficiently confined. In the second cell equipped with 400 microns culet, we heated the sample at the loading pressure (12 GPa) and we were able to obtain a liquid diffraction signal of the fully melted sample up to 35 GPa. In the third and fourth cells equipped with 150 microns culets we attempt to apply the same experimental procedure (increase the sample pressure up to 10 GPa and start the laser heating at this relatively low pressure). Unfortunately in both cells, the gasket hole has open during heating. We believe that the boron disk diameter was initially too large (40 microns) with respect to the sample cavity (55 microns) and the diamond culet (100 microns). Additionally, we observed that the bowls systematically tear due to (as measured) strong non hydrostatic conditions within the capsule. The last cell equipped with 300 microns culet was installed at the end of the experiment and we were able to measure two additional melting point (at 5 GPa and 13 GPa) before the shutdown. Preliminary results on the melting line of Gold to 35 GPa are presented on Figure 1A and suggest a revision of the latter. Additionally a liquid diffraction signal of gold at high P-T and the pressure evolution of the main diffusion peak are presented on Figure 1B. The high quality of these data allow to extract the S(Q) and g(r) of the liquid.

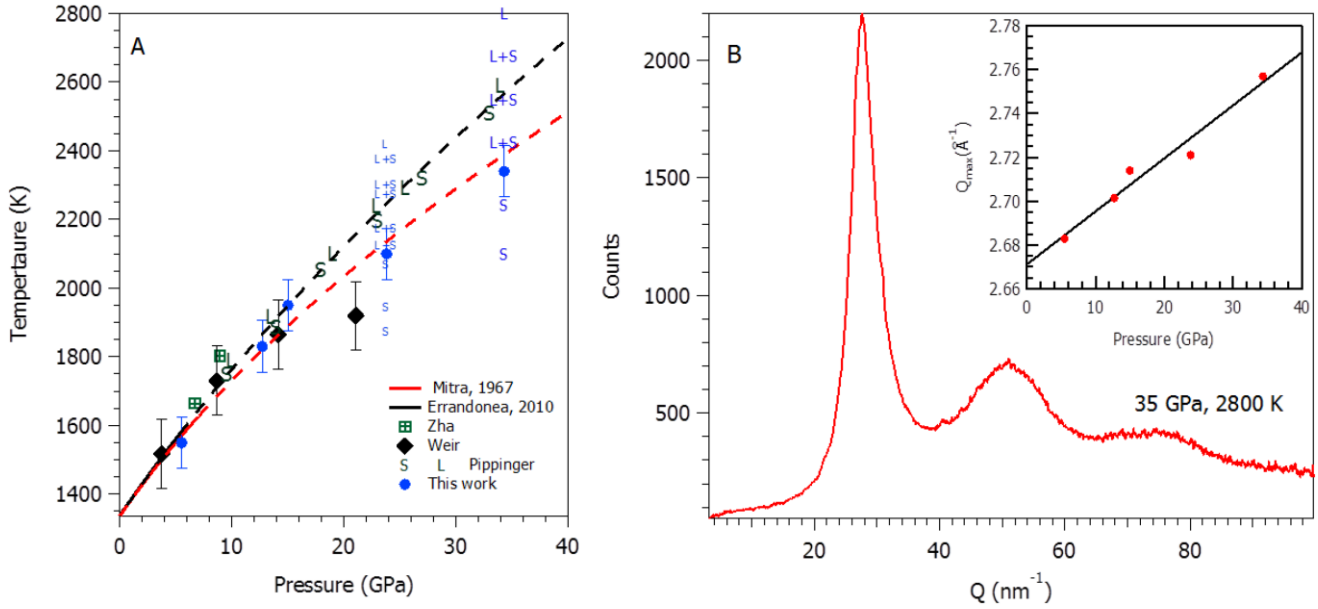

Figure 1: Preliminary results obtained during the last run of LTP HD-463 and HC-2187. A: Melting line of gold. Our measurements are indicated with blue dots; B: Liquid diffraction pattern of the fully melted gold sample.

References

[1] C.-S. Zha and W. A. Bassett, Rev. Sci. Instrum. 74 , 1255 (2003)
[2] S. T. Weir et al., Rev. Sci. Instrum. 80, 013905 (2009).
[3] Pippinger, T. et al., arXiv:1104.1304. (2011)