EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

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

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report"),*
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

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.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number 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:** High Pressure, X-ray Diffraction Study of a novel compound: SiOS	**Experiment number**: CH-5608
Beamline: ID27	**Date of experiment**: from: 18/09/2018 to: 21/09/2018	**Date of report**: 12/02/2020
Shifts: 9	**Local contact(s)**: Mohamed Mezouar	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

We synthesized the SiOS compound in situ from the elements, high purity Si, S and molecular oxygen, directly in the DAC, with O_2 also working as the pressure transmitting medium, PTM, similarly to our recent synthesis of SiS_2 in the presence of Ar as the PTM (Wang et al, JCP 2018). Our DACs were equipped with 250-350 mm culet diamonds and Re gasket with an initial hole of typically 100-150 mm diameter and 40-50 mm thickness. We first squeezed S powder between the two diamonds, by closing the DAC with no gasket, and when the cell was open, a few microns thick S layer remained well adherent to one of the diamonds. Subsequently, we squeezed Si powder on top of the S layer by following the same procedure, and we obtained a few microns thick Si layer well adherent to the S layer. The double S/Si layer was covering the entire surface of the diamond. We then carefully removed part of it in order to make the layer approximately round shaped, right in the middle of the diamond culet, with a diameter smaller than the gasket diameter. Finally, we put the gasket between the diamonds and we also inserted one KCl chip and one ruby chip in the sample chamber, well away from the S/Si double layer, in order to measure pressure using both the equation of state of KCl and the ruby fluorescence technique. Liquid O_2 was then cryoloaded at 77 K and approximately 1 bar. After loading, the Si/S/O_2 sample was compressed at room temperature and powder XRD patterns were measured with pressure steps of 2 GPa, up to about 8 GPa, showing the well known phases of S, Si and O_2, respectively. We then gently laser heated the Si/S/O_2 sample from the side of S, by using a CW solid state laser at 1064 nm, at the starting pressure of 7.7-8.0 GPa, up to approximately 1000 K or slightly above, in order to melt together and react elemental S, Si and O_2. Indeed, at this P-T conditions, all three elements were expected to melt. It was mainly Si that was directly heated up by the NIR laser, subsequently transferring the heat to the attached S layer on one side and to bulk O_2 on the opposite side. On the other hand, S and O_2 worked as thermal insulating layers for Si. After laser heating, the sample was temperature quenched and XRD patterns were measured. The comparison of measured and DFT predicted XRD patterns revealed that indeed the *Cmc2₁* structure of SiOS was synthesized. Instead, laser heating of the sample from the side of O_2 promoted the reaction of Si with O_2 with the formation of silica phases rather than the simultaneous reaction of the three elements together. XRD data analysis is presently very preliminary and work is in progress on it.