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:** Metal-to-insulator electronic phase transition in the Re_3Ge_7 endohedral cluster compound	**Experiment number**: HC-4105
Beamline:	**Date of experiment**: from: 07th April 2021 to: 10th April 2021	**Date of report**: 03rd June 2021
Shifts: 9	**Local contact(s)**: Ms. Grendal Ola Gjonnes	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr. Valeriy Verchenko, Department of Chemistry, Lomonosov Moscow State University, 119991 Moscow, Russia (remote user)

Report:

As a result of the HC-4105 experiment, the crystal structures of two compounds, Re_3Ge_7 and $ReGa_{4.5}Ge_{0.5}$, have been probed at temperatures between 10 K and 300 K. Re_3Ge_7 and $ReGa_{4.5}Ge_{0.5}$ exhibit phase transitions at the critical temperatures of 58.5 K and 258 K, respectively. For Re_3Ge_7, the transition is accompanied by the change in transport behavior from metallic at high temperatures to semiconducting at low temperatures. The room-temperature high-resolution powder X-ray diffraction (HRPXRD) pattern of Re_3Ge_7 is shown in Figure 1.

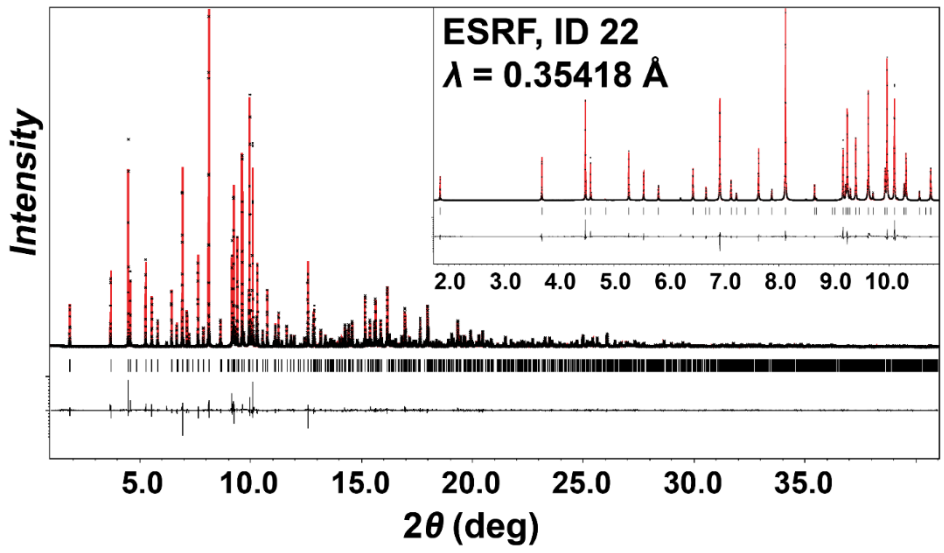

Fig. 1. HRPXRD pattern of Re_3Ge_7 registered at room temperature. The experimental data are shown by black points, and the calculated curve by red line. Positions of peaks and the difference curve are given by black ticks and black line, respectively, in the bottom part of figure.

The Rietveld refinement was performed yielding the orthorhombic crystal structure, *Cmcm* space group, a = 3.230964(6) Å, b = 9.055455(18) Å, and c = 21.98890(4) Å. This unit cell persists upon cooling down to the lowest measured temperature of T = 10 K, and no structural transformation occurs in the vicinity of the critical temperature, which is T_c = 58.5 K. The evolution of unit cell parameters within the studied temperature range is presented in Figure 2. The transition is clearly visible as a sharp kink at the critical temperature. Thus, the metal-to-semiconductor electronic phase transition in Re_3Ge_7 is second-order, and should be connected with fine details of the electronic structure.

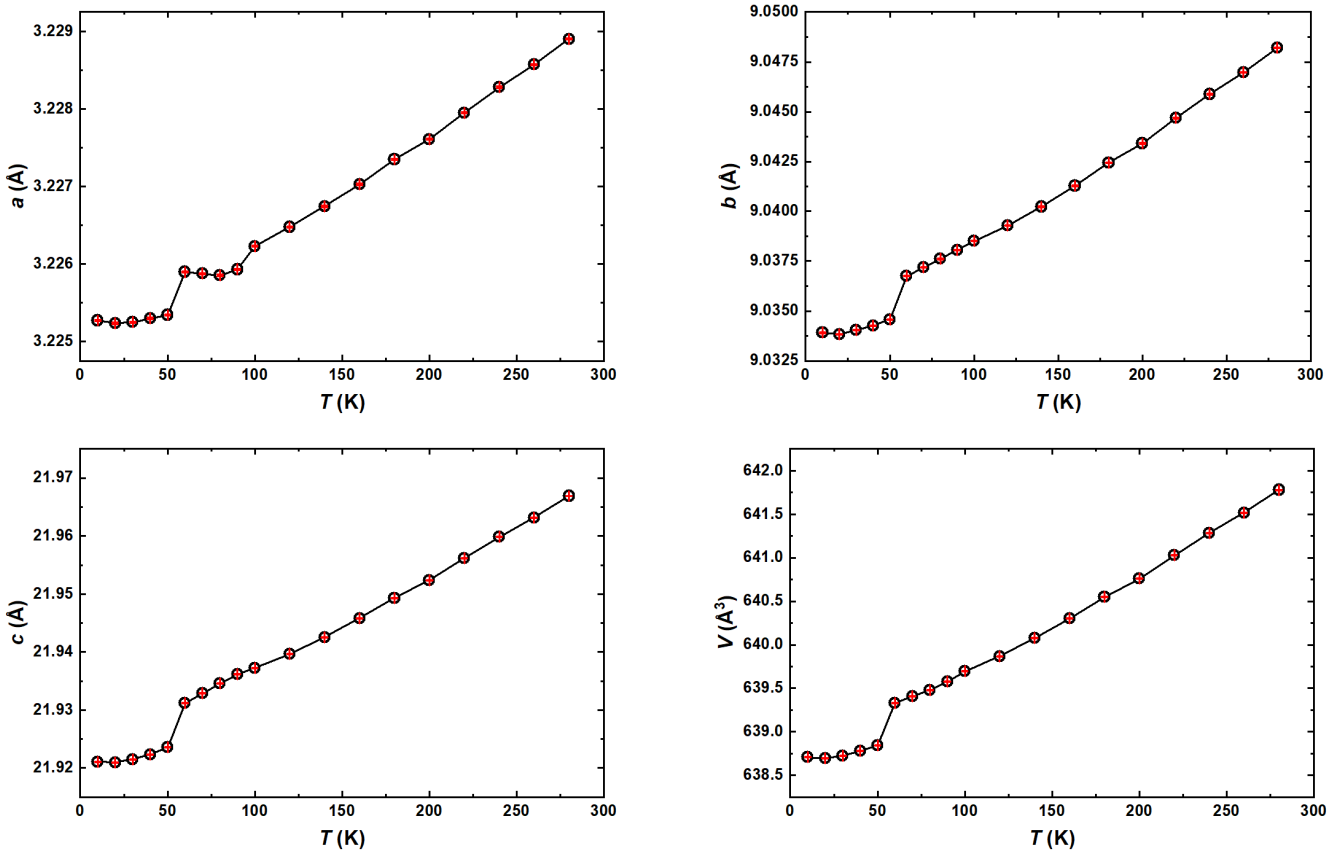

Fig. 2. Unit cell parameters of Re_3Ge_7 as a function of temperature.

In contrast to Re_3Ge_7, the second studied compound, $ReGa_{4.5}Ge_{0.5}$, exhibits a first-order phase transition. The Rietveld refinement against the room-temperature HRPXRD data yields a tetragonal unit cell, $P4_2/mmc$ space group, a = 8.06530(2) Å, and c = 6.72137(2) Å in good agreement with the single-crystal X-ray diffraction data. All HRPXRD patterns registered below room temperature contain additional superstructure reflections, what indicates the lowering of symmetry as a result of the first-order phase transition. In Figure 3, the HRPXRD patterns collected at room temperature and at T = 50 K are shown, and the latter demonstrates superstructure reflections marked by asterisks. The low-temperature HRPXRD data can be indexed in the monoclinic or triclinic unit cell, but these results require further investigation.

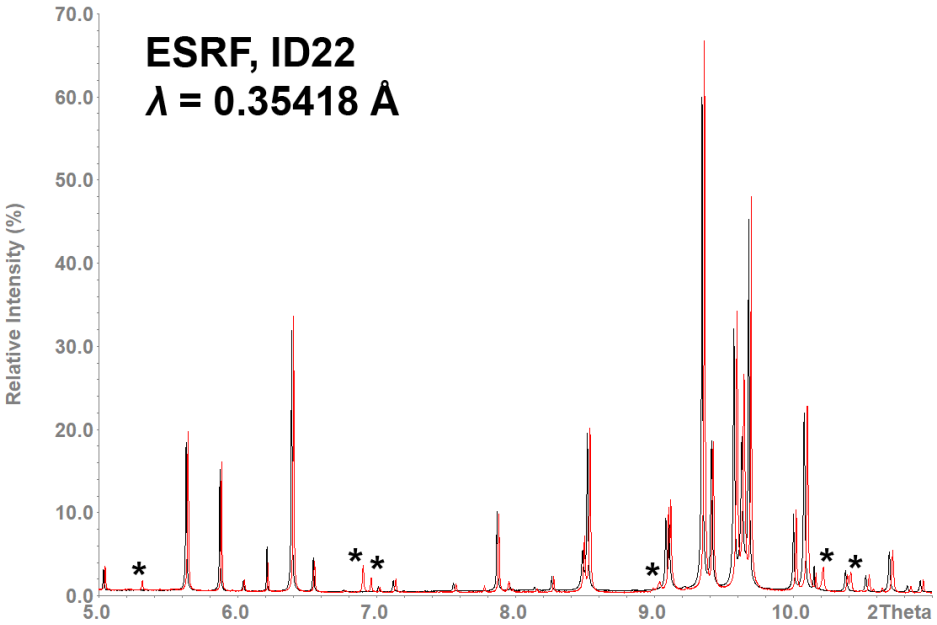

Fig. 3. Room-temperature (black) and low-temperature (red) HRPXRD patterns of $ReGa_{4.5}Ge_{0.5}$. The superstructure reflections are marked by asterisks.