

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: High-Temperature and high-pressure study of the stability conditions of the new layered transition metal sulfides with trigonal prismatic coordination	Experiment number: HS-3547
Beamline: ID27	Date of experiment: from: 8h00 12/03/2008 to: 8h00 15/03/2008	Date of report: 26/02/2009
Shifts: 9	Local contact(s): Dr. Wilsaon A CRICHTON	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Ikuya Yamada*, Matteo d'Astuto, Andrea Gauzzi*, and Yann Christian Le Godec*, Université Pierre et Marie Curie Institut de Minéralogie et de Physique des Milieux Condensés (I.M.P.M.C.) 140 rue de Lourmel F-75015 PARIS		

Report:

In this experiment, we intended to study pressure-temperature (P - T) phase diagram of delafossite-derived sulfide AuCrS_2 and related sulfides. AuCrS_2 , which is synthesized in evacuated quartz tube at high temperature, has a delafossite-derived structure represented as a stacking of the gold atoms and edge-shared CrS_2 octahedra shown in Fig. 1 [1]. On the other hand, another delafossite-derived sulfide AuVS_2 , which is synthesized under high pressure and high temperature (HPHT), has trigonal prismatic VS_2 layer (Fig. 1) [2]. To our first prediction, AuCrS_2 might be transformed into AuVS_2 -type structure under HPHT. The ID 27 beamline is the most stable one for studying the structural phase transition under HPHT.

Unfortunately, we failed in heating of sample due to furnace trouble. However, we observed pressure-induced phase transition for AuCrS_2 at high pressure of 6.5 GPa at room temperature. Fig. 2 (a) shows the X-ray diffraction (XRD) patterns collected under high-pressure. The (00 l) reflections are almost unchanged below 6.5 GPa while the others were shifted to higher angle by compression, which indicates a strong anisotropic compressibility of AuCrS_2 . Anisotropic compressibility was reported for delafossite-type oxides [3,4]. However, AuCrS_2 is less compressive along c axis than oxides. The strong anisotropic property of AuCrS_2 probably originates from the covalent property of the linear $\text{S}^{2-}-\text{Au}^+-\text{S}^{2-}$ bond stronger than that of $\text{O}^{2-}-\text{Cu}^+-\text{O}^{2-}$ in delafossite oxides. The lattice constants changed discontinuously at 6.5 GPa; c axis expanded while a axis shrunk. Note that such an expansion of the axis (c axis in this case) has never been reported for delafossite oxides although pressure-induced phase transition was known [3]. Since the XRD patterns in decompression recovered into those below the transition pressure shown in top two patterns in Fig. 2(a), the

phase transition is reversible. That is contrast to the irreversible phase transition in delafossite oxide [3]. The detailed crystal structure analysis of the high-pressure phase was impossible because the quality of the data was insufficient. However, optimisation of the setting and improvement of sample preparation should make detailed crystal structure analysis possible. Furthermore, comparison study of AuCrS_2 , AuVS_2 , and the solid solution $\text{AuCr}_{1-x}\text{V}_x\text{S}_2$ under HPHT may lead to remarkable results in the phase diagram of delafossite-derived sulfides.

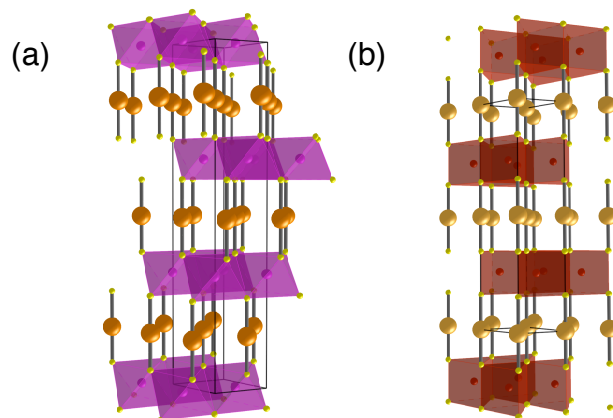


Fig. 1 Crystal structure of (a) AuCrS_2 and (b) AuVS_2

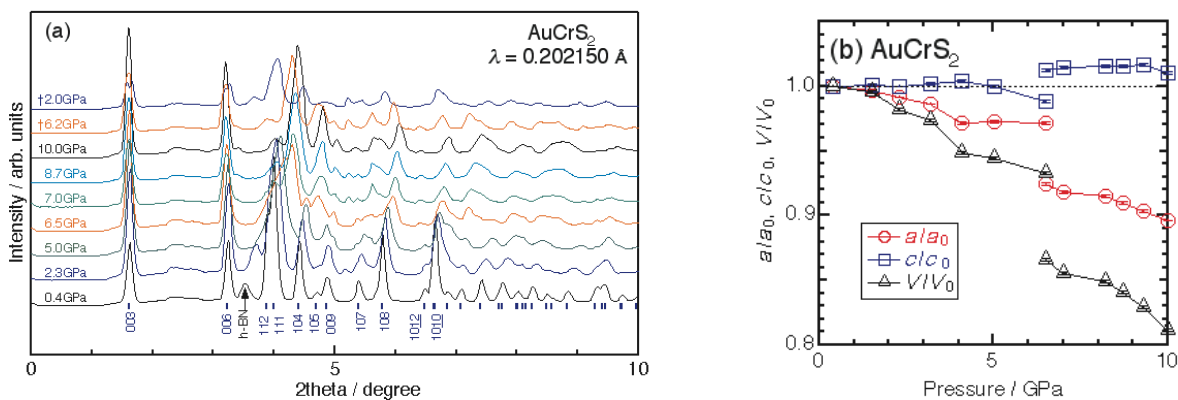


Fig. 2 (a) XRD patterns under high pressure collected at the ID 27 beamline. $00l$ reflections were unchanged below 6.5 GPa and slightly shifted to lower angle above 6.5 GPa while other reflections monotonically shifted to higher angle with increasing the pressure. The top two patterns show those on decompressing. (b) Lattice constants and unit cell volume as a function of the applied pressure. Clear discontinuity can be seen at 6.5 GPa.

References

- [1] H. Fukuoka et al., *J. Solid State Chem.* **148** (1999) 487.
- [2] A. Gauzzi et al., *Phys. Rev. B* **66** (2002) 0855106.
- [3] J. Pellicer-Porres et al., *Phys. Rev. B* **69** (2004) 024109.
- [4] M. Hasegawa et al., *Solid State Commn.* **128** (2003) 303.