EUROPEAN SYNCHROTRON RADIATION FACILITY

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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

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:** Determination of the dependence of the elastic tensor of $M_2Mo_6Se_6$ on uni-axial strain	**Experiment number**: HC-117
Beamline: ID28	**Date of experiment**: from: 26/11/13 to: 3/12/13	**Date of report**:
Shifts: 18	**Local contact(s)**: Alexey Bosak	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Mr. Liam Gannon* University of Oxford

Dr. Moritz Hoesch * Diamond Light Source

Report:

Technical Background.

The molybdenum selenides $M_2Mo_6Se_6$ where *M* is either an alkaline or transition metal are a series of intercalated quasi one-dimensional single crystals that demonstrate interesting electronic and physical properties. The crystal structure consists of long chains of stacked Mo-Se triangles at 180^o rotation to each other, which form a hexagonal lattice that can be intercalated by a number of different guest ions. Previous studies have reported a metal to insulator transition in the Rb,Na,Cs and K samples whilst the Tl and In samples demonstrate metallic behaviour down to a superconducting transition. The mechanism behind the metal-insulator transition seen in the alkaline metal samples is not well understood, electronic and magnetic measurements favour a charge density wave model [1] however there remains no structural evidence for such a state. To investigate the role of the lattice (if any) on the metal-insulator transition longitudinal phonon modes of several different $M_2Mo_6Se_6$ samples were measured to identify any signs of electron-phonon coupling.

Another aspect of these samples investigated during the beamtime was the effect of uniaxial strain on the longitudinal speed of sound. Uniaxial strain was applied to $Cs_2Mo_6Se_6$ and $K_2Mo_6Se_6$ samples using a novel piezoelectric straining device Fig.(1).The straining device that was used consists of two shear piezoelectric shear actuators positioned parallel to each other on a baseplate Fig.(1). Each actuator is constructed from several individual piezoelectric elements. Applying a bias voltage across these elements causes them to displace in such a way that the top of the actuators displace with respect to one another. This increases the distance between the ceramic top plates attached to the top of the piezoelectric actuators. The sample is secured across this gap (between the macor top plates) using an epoxy glue (Epotek E-4110) in such a manner that increasing the size of the gap induces a strain in the sample. The baseplate provides an additional method of applying strain to the sample, by tightening four screws at the corners of the plate the piezoelectric stacks pivot about a centre point increasing the size of the gap between them. The uniaxial strain applied to the sample was quantified by calculating the expansion of the c-axis lattice parameter through the measurment of the 2Θ position of the [002] Bragg peak.

Experimental Details

The beamline was operated in 9/9/9 mode (17.794keV incident energy, 3meV energy resolution and a flux of 2.7 10^{10} ph/s/200mA) for the duration of the experiment. For strain measurements the adth stage was used in conjunction with a 1005 Huber goniometer allowing the straining device to be mounted such that the sample c axis was perpendicular to the incident beam, the theta stage was used for measurements in which strain was not applied. The straining device was controlled from within the control cabin via a +5V DC voltage source amplified up to a maximum 250V by a PI voltage amplifier (E-413.00). All samples were aligned on the [002] Bragg spot and phonons with energies up to 25meV were measured along the 00l direction with special attention paid to a guest ion mode observed at 5meV.

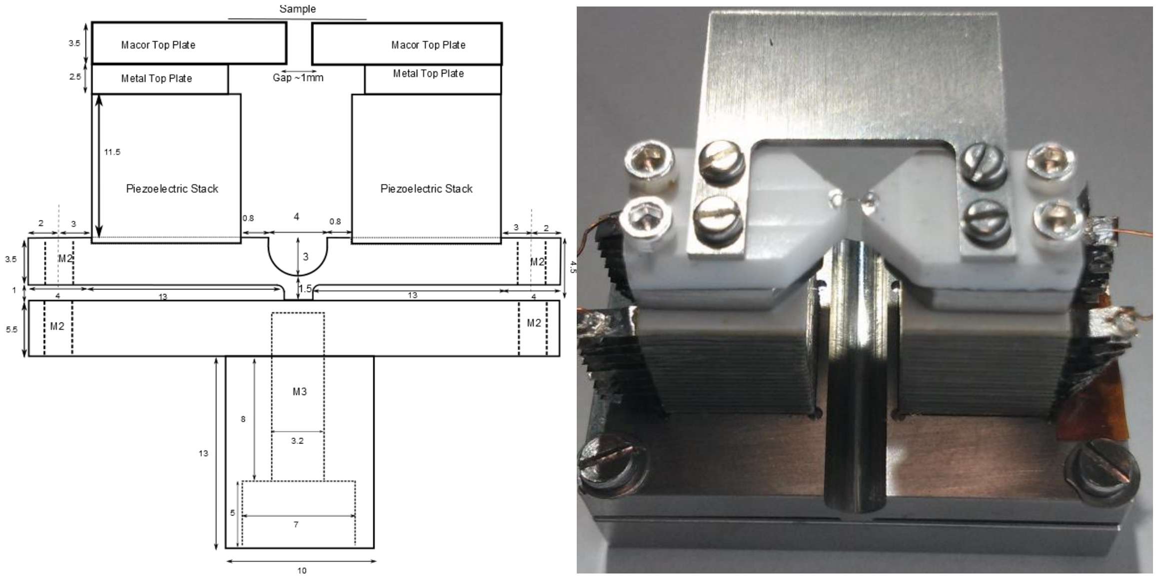

Fig.1 a) Straining device schematic and b) the straining device with a sample mounted across the macor top plates. A fixing bracket used for sample transfer attached.

Experimental Results.

The longitudinal phonon modes of several unstrained $M_2Mo_6Se_6$ samples (Rb,K,Tl,In and Cs) were successfully measured Fig.(2a). In all samples the guest ion mode showed a softening around the [003] Γ-point. Normalising the phonon energy about this point Fig.(2b) to the square root of the guest ion mass reveals that the energy of the mode differs depending on the choice of guest ion. The phonon softening is indicative of electron-phonon coupling and may help elucidate the nature of the metal to insulator transition observed in these materials. Further studies specifically temperature dependent Raman spectroscopy will reveal the exact role this softening plays in the electronic behaviour of $M_2Mo_6Se_6$.

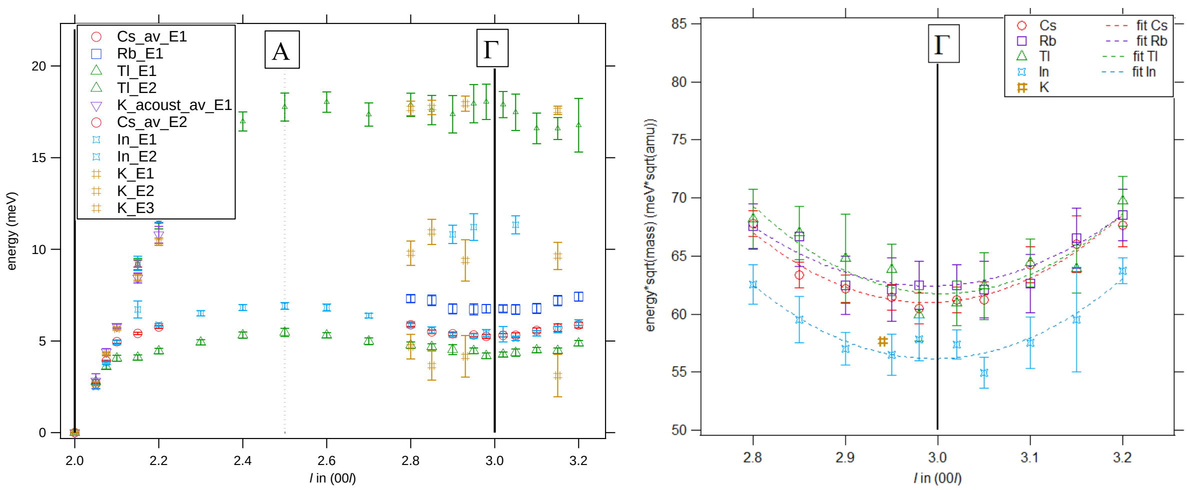

Fig.2 a) Unstrained longitudinal phonon modes of several $M_2Mo_6Se_6$ for phonon energies up to 20meV. b) 5meV phonon mode about the [003] gamma point normalised to the square root of the guest ion mass.

The phonon modes of two samples, one intercalated with Cs and the other K, were successfully measured under strain. The maximum strain achieved was 0.077% in $Cs_2Mo_6Se_6$ and 0.083 in $K_2Mo_6Se_6$ after which the samples broke under the application of mechanical strain. For both of these samples the low q (acoustic) phonon was measured as a function of strain, the gradient of which was used to calculate the longitudinal speed of sound through the sample. There was an observed decrease in the speed of sound with increasing applied voltage (strain) Fig.3 (b).

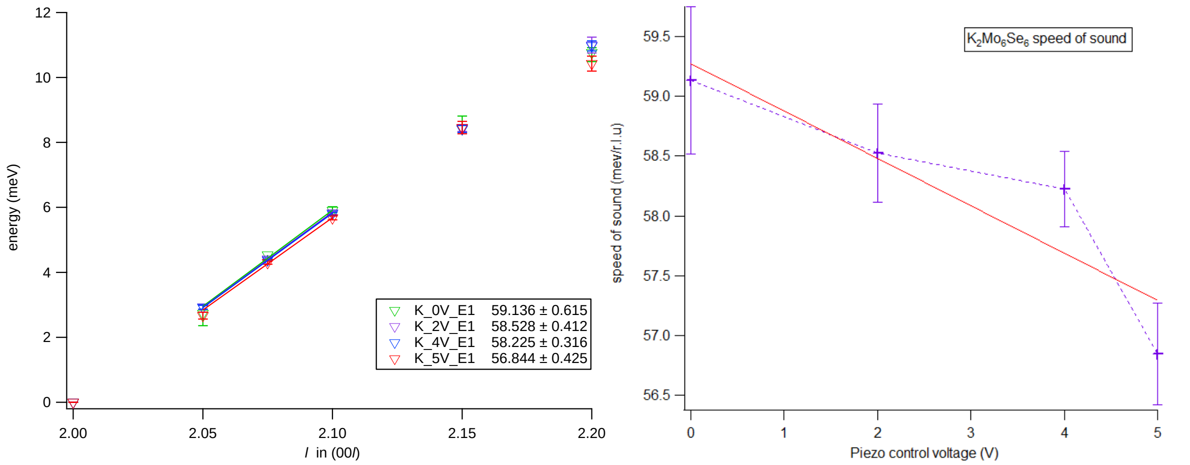

Fig.3 a) Acoustic phonon mode of $K_2Mo_6Se_6$ under increasing strains and b) the corresponding longitudinal speeds of sound as a function of applied voltage.

References

[1] A.P.Petrovic et al *PRB* **82** 235129 (2010)