## 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.

ESRF	Experiment title: Investigation osf an EuSe/PbSe superlattice using contrast variation by anomalous x-ray scattering	Experiment number: SI 648
Beamline: ID01	<b>Date of experiment</b> : from: 06.06.2001 to: 12.06.2001	<b>Date of report</b> : 30.08.2001
Shifts: 18	Local contact(s): Till Hartmut Metzger	Received at ESRF:

Names and affiliations of applicants (\* indicates experimentalists):

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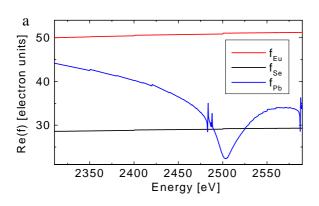
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### Report:

In recent years, the fabrication of self-organized 3D ordered semiconductor quantum-dots has progressed to become a promising tool for a future nanoscale technology. Most systems where this method is applied are grown in hetero epitaxial Stranski-Krastanow mode. In all of these cases, the dots are embedded in a matrix of a similar lattice structure, but slightly different lattice parameter. The mutual strain in both compounds initiates the dot growth. The strain modulation of the matrix material induces a self-organized ordering of the dots especially when dots are deposited in multilayers. In the system PbTe/PbSe nearly perfectly ordered quantum dot crystals have been grown [1-2]. Novel electronic, optical and in some materials even magnetic properties are expected, thus strain characterisation is necessary to develop and apply these growth modes to further sytems.

The main problem in the x-ray investigation of such systems is the superposition of the Bragg reflections from dot- and matrix material. To overcome this problem we performed anomalous scattering, in the case of our system at very low x-ray energies. As the lead salts crystallize in the rock salt structure, the Pb M-edge at about 2.502 keV allows a strong variation of the scattering contrast at the (111) superstructure reflection. The structure amplitude at this reflection is  $F_{111} = f_{Pb} - f_{Se}$ , therefore we can selectively suppress the (111) intensity for PbSe at a certain energy.

We have calculated the (111) intensity for PbSe and EuSe as shown in Fig. 1b. In Fig. 1a the energy dependent real part of the complex scattering factor f for Eu, Pb, and Se is presented according to Refs. [3-4] taking into account the momentum transfer corresponding to the PbSe-(111) Reflection . Additionally, in Fig. 1b the result of an experiment at ID01 is presented where the intensity minimum at the (111) reflection for a PbSe Film was determined. A shift in energy of the minimum of about 6 eV with respect to the calculated values could be due to the limited energy resolution on an absolute scale. (note that the energy was determined from a high-angle bragg-peak of a high quality BaF<sub>2</sub> single crystal) .



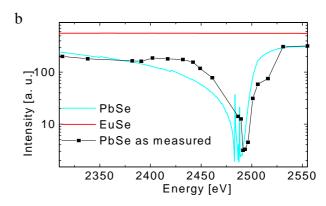


Fig. 1: a: Calculated real parts of the complex scattering factors f of Eu, Pb, and Se dependent on the x-ray energy [3]. At 2.485 keV, just below the Pb M-edge (2.502 keV), one can tremendously decrease, the PbSe (111)-intensity at the point of intersection of  $f_{Pb}$  and  $f_{Se}$ , which improves the visibility of the EuSe signal.

b: Calculated  $|F_{hk}|^2$  at the PbSe- and EuSe (111)-reflections together with the measured relative intensity at the (111)-reflection of a PbSe (111) film with respect to the energy (calibrated via the Bragg reflection of the BaF<sub>2</sub> substrate).

At these energies one expects a strong enhancement of the ratio of the scattered intensities from the dots with respect to the matrix; whereas in the usual hard x-ray regime around 10 keV, the scattering from PbSe is always stronger then from EuSe.

In addition, the extreme contrast between both materials as well as the high resolution in reciprocal space that is achieved at the low x-ray energy allows a reciprocal space mapping in which the strain in the quantum dot superlattice can be distinguished from the strain modulation in the matrix material. Close to 2.5 keV diffractometry has to be carried out under vacuum conditions which can solely be accomplished at the windowless beamline ID01. In Fig. 2, specular scans on the (111) reflection are shown for two different energies, 2.49 keV (a) and 12.4 keV (b). The sample consisted of a 2D 30x PbSe/EuSe superlattice with a PbSe thickness of 70 Å and a EuSe thickness of about 30 Å. The quality difference due to the differences in resolution and contrast in the two datasets is clearly visible. The blue lines in both graphs are logarithmic Gauss fits to the maxima of the superlattice peaks. For the SL-peaks one expects an oscillating  $\left(\sin\left(d \cdot (Q_z - Q_0)/2\right) / \left(d \cdot (Q_z - Q_0)/2\right)\right)^2$ -shaped envelope function with d being the thickness of one single layer, so

the gaussian is an approximation to estimate the centre only for the central maximum of this envelope. For Fig. 2a the centre of this envelope lies on the right side of the highest SL peak, corresponding to the EuSe lattice parameter, whereas in Fig. 2b it is shifted to the right, where the PbSe lattice parameter is expected. The suppression of the PbSe scattering leads also to a much less pronounced intensity from the 2 micron PbSe buffer (peak to the right from the highest SL-peak) whereas the low penetration depth leads to a significant weakening of the BaF<sub>2</sub> substrate reflection (peak to the left). At higher Energies one cannot profit from these advantages and the central part of the scan is dominated by the reflections from substrate and buffer.

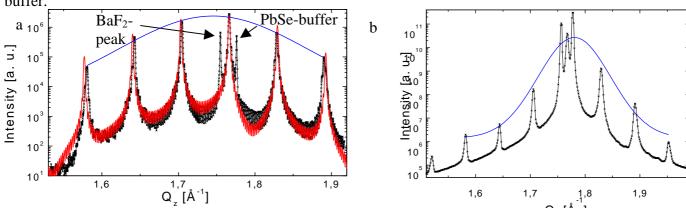


Fig. 2 a: Specular scan on the (111) reflection (growth direction) of a 30x PbSe/EuSe-quantum dot apperlattice at 2.49 keV, together with a kinematic simulation (red line, which does not include the reflections from substrate and buffer). b: The same reflection at 12.4 keV. The visibility of the interference pattern is less pronounced, originating from a much weaker contrast between the two materials as well as from the shorter coherence length which scales with the wavelength. The blue lines are logarithmic Gauss fits to the maxima of the superlattice peaks in order to estimate the centre of the envelope.

In this first experiment at the Pb M-edge, the intensity minimum for the (111) PbSe reflection could be determined to be of the order of magnitude as calculated. For the first time, bragg diffraction from a PbSe/EuSe multilayer was carried out at these low energies yielding excellent data quality. For multilayers or quantum dot samples of the lead-europium-chalcogenide system we have now a powerful tool to discriminate between materials by enhancing or suppressing the contrast via anomalous scattering.

#### References

- [1] G. Springholz, V. Holy, M. Pinczolits, G. Bauer, Science 282, 734 (1998).
- [2] G. Springholz, J. Stangl, M. Pinczolits, V. Holy, P. Mikulik, P. Mayer, K. Wiesauer, G. Bauer, D. Smilgies, H.H. Kang, L. Salamaca-Riba, Physica E 7, 870 (2000).
- [3] B. L. Henke, E. M. Gullikson, J. C. Davis, Atomic Data and Nuclear Data Tables 54, 181 (1993).
- [4] J. Baró, M Roteta, J. M. Fernández-Varea, F. Salvat, Radiat. Phys. Chem. 44, 531 (1994).