Standard Project

Experimental Report template

Proposal title:Formation and quantitative structure determination of PtSe2 obtained by selenization of Pt(111)		**Proposal number:** **20161086**
Beamline: IF-INS	**Date(s) of experiment:** from: 2017/01/24 to: 2017/01/31	**Date of report:** 2017/02/15
Shifts: 18	**Local contact(s): Gilles Renaud**	*Date of submission:* *2016/10/14*

Objective & expected results (less than 10 lines):

We planed to investigate the formation and structure of Platinum diselenide ($PtSe_2$) layers obtained by selenization of the surface of a Pt(111) single crystal. $PtSe_2$ belongs to the family of transition metal dichalcogenides (TMD). These layered materials have attracted much attention due to their intriguing properties when reaching atomic thicknesses[1,2,3]. A single molecular layer of $PtSe_2$ is formed by an atomic trilayer which consists of two adjacent hexagonal layers of the chalcogen (Se) covalently bonded by an hexagonal layer of the transition metal (Pt) forming a Se-Pt-Se layer configuration. In addition, growth by direct selenization on a Pt(111) surface, results in a Pt(4X4)/PtSe2(3X3) reconstruction [4] Our purpose here, consists in slowly heating the sample to form the $PtSe_2$ layer by selenization of a Pt(111) surface previously covered with Se. During the heating procedure our aim was to follow the appearance and cristallinity of the $PtSe_2$ layer. Then once the PtSe2 layer is formed we expected to observe and analyze in detail the reconstruction by performing a complete Surface X-Ray Diffraction measurement [5].

Results and the conclusions of the study (main part):

For this study, we deposited a few nm of amorphous selenium at room temperature on a clean Pt(111) single crystal in our MBE setup (CEA/INAC). Then using a HV suitcase (1e-7 Torr) we transferred it to the INS2-BM32 setup. After preliminary alignments of the single crystal and RHEED and Auger verifications of the surface quality, we measured the reference structural state of the sample by performing rocking curves, radial scans (fig 3) and GIXD reciprocal mapping (fig 2 left). These measurements show the Pt(111) single crystal alone confirming the amorphous character of the selenium layer whose presence was checked from Auger measurements. Then we heated the sample to activate the selenization process [4]. In order to avoid selenium evaporation before the selenization occurs, we heated very slowly (see fig. 1 right) while measuring rocking scans at the <110> Bragg position expected for $PtSe_2$ (fig. 1 left).

At the beginning of the heating procedure, a very broad modulation of the background intensity can be observed (also weakly visible on the mapping, fig. 2 left). While the temperature increases (~200C) a broad peak appears at the <110> $PtSe_2$ expected position (around 0 deg) with two satellites peaks at about 19deg. This means on the one hand that $PtSe_2$ small domains aligned with the Pt substrate are formed but with a large in plane mozaicity and on the other hand rotated domains appear due to some in-plane lattice coincidence. For further annealing, the satellites peaks disappear gradually and the width of the central peak decreases up to about 0.5deg (FWHM) while its maximum intensity increases correlatively resulting in a good crystalline and alignment quality of the PtSe2 layer. During the heating procedure in plane reciprocal radial and mapping scans were performed in order to investigate the intermediate crystallization states. Since these scans (not shown here) take time, the temperature was lowered enough in order to slow down the kinetic of the crystallization during their processing (fig. 1 right). The final state after heating at (~400C) was investigated in detail by performing in plane reciprocal radial (fig.3) scans and mapping (fig. 2). These measurements showed that in addition to fine $PtSe_2$ peaks , numerous reconstruction peaks appeared corresponding perfectly to the diffraction of an hexagonal lattice with a supercell $PtSe_2$(3X3)/Pt(4X4). The intensities of these reconstruction peaks exhibit magnitude comparable to the $PtSe_2$ Bragg peaks, possibly meaning that strong displacements of atoms occur in the $PtSe_2$ layer and in few Pt layers close to the surface. For a more quantitative investigation of this reconstruction according to the method described by Drnec et al **[5], we performed in-plane rocking scans then CTR and rod scans (fig . 4) for various positions of moiré superlattice. We also performed the classical reflectivity measurement to verify the layer thicknesses and associated parameters. The quantitative analysis of the latter measurements needs a larger computational effort for correct intensity integration from 2D detector images, structure simulation and optimization fitting.**

Justification and comments about the use of beam time (5 lines max.):

The INS2/BM32 setup is ideally suited for this experiment (excellent UHV; AES; RHEED; 2D Maxipix). Furthermore our sample holders being identical to that of INS2/BM32 this allowed us vacuum transport with an home made suitcase. The 18 shifts enable us to follow carefully the $PtSe_2$ phase formation and domains reorientation during a slow heating procedure then followed by the measurement (numerous CTR including specular (00l) one and rod scans) for the structure determination.

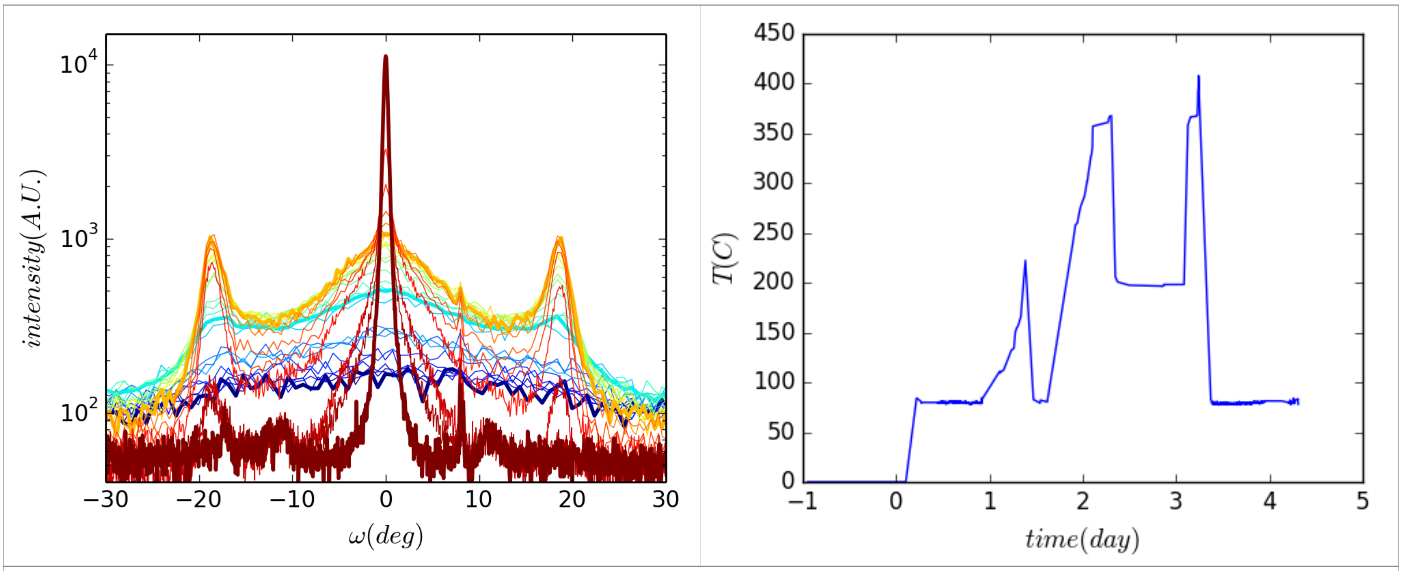

Fig. 1. left : evolution of the rocking scan on $PtSe_2$<110> position during the slow heating procedure. The color indicates progress on the heating process (blue : before, red after). Right :shows the temperature versus the time. Long unidimensional scans and reciprocal maps were performed with temperature well below the current annealing temperature in order to slow down the recrystallization process.

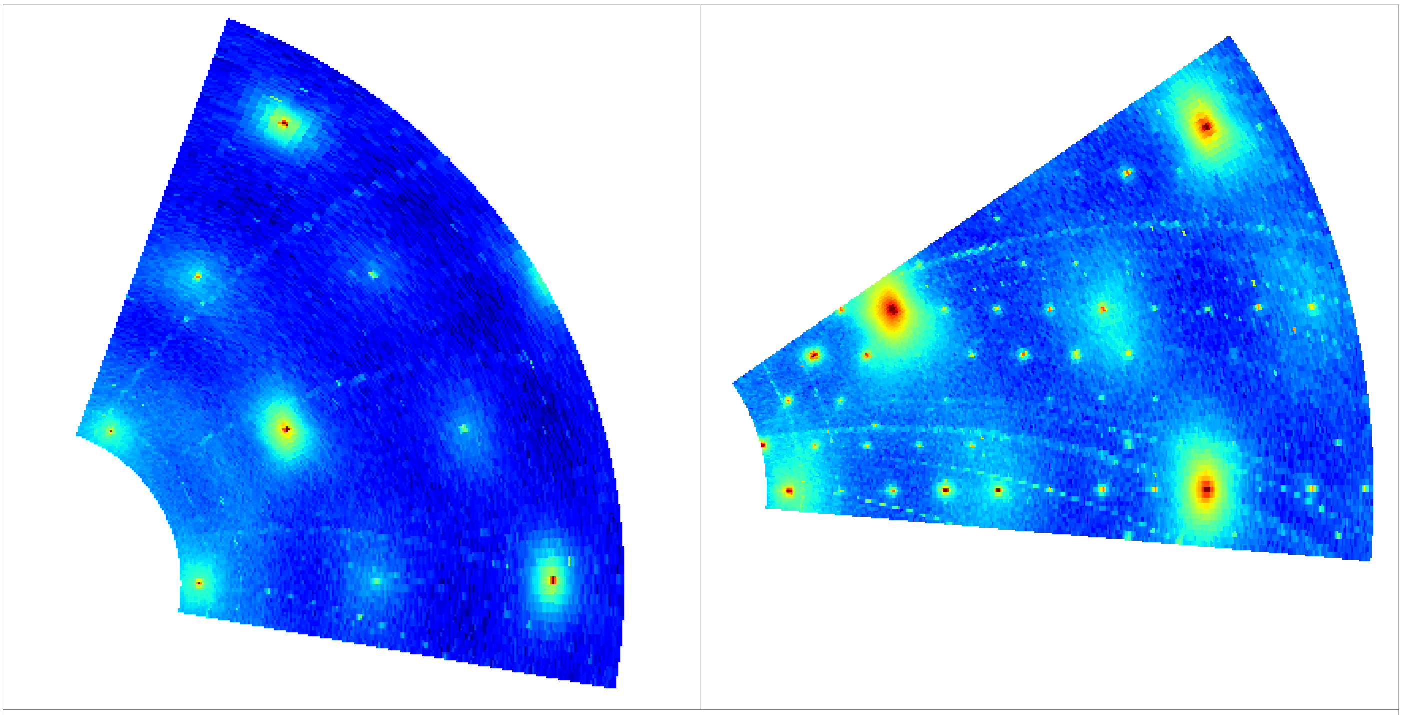

Fig 2 : GIXD mapping before (left) and after (right) the heating procedure. Before annealing only the Pt(111) peaks are visible. After annealing new peaks appeared due to $PtSe_2$ and also due to the 4x4 reconstruction (with respect to the Pt basis). Some artifacts (dots along circles) are present but could be removed when analyzing images acquired with 2D detector. Here the mapping was constructed using integrated intensity taken in a fixed region of interest (ROI).

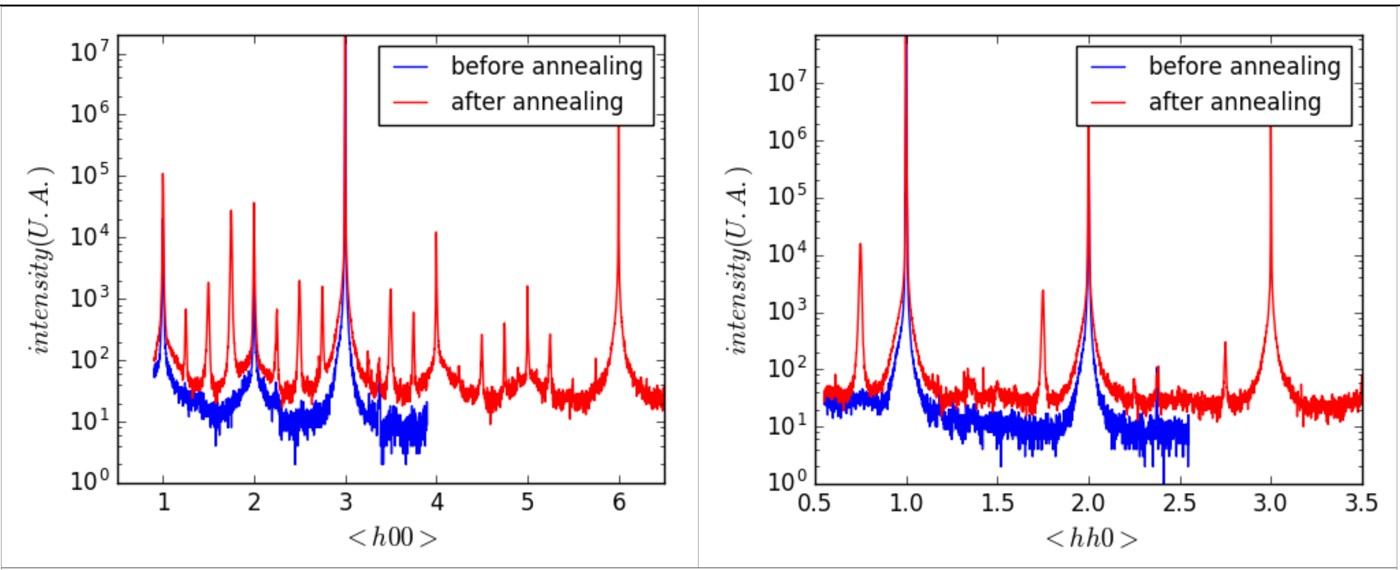

Fig 3 : Radial in plane hk0 scans along the main reciprocal directions <h00> and <hh0> before and after annealing. After annealing, in addition to $PtSe_2$ Bragg peaks, reconstruction peaks appear with intensity comparable to those of the Bragg ones.

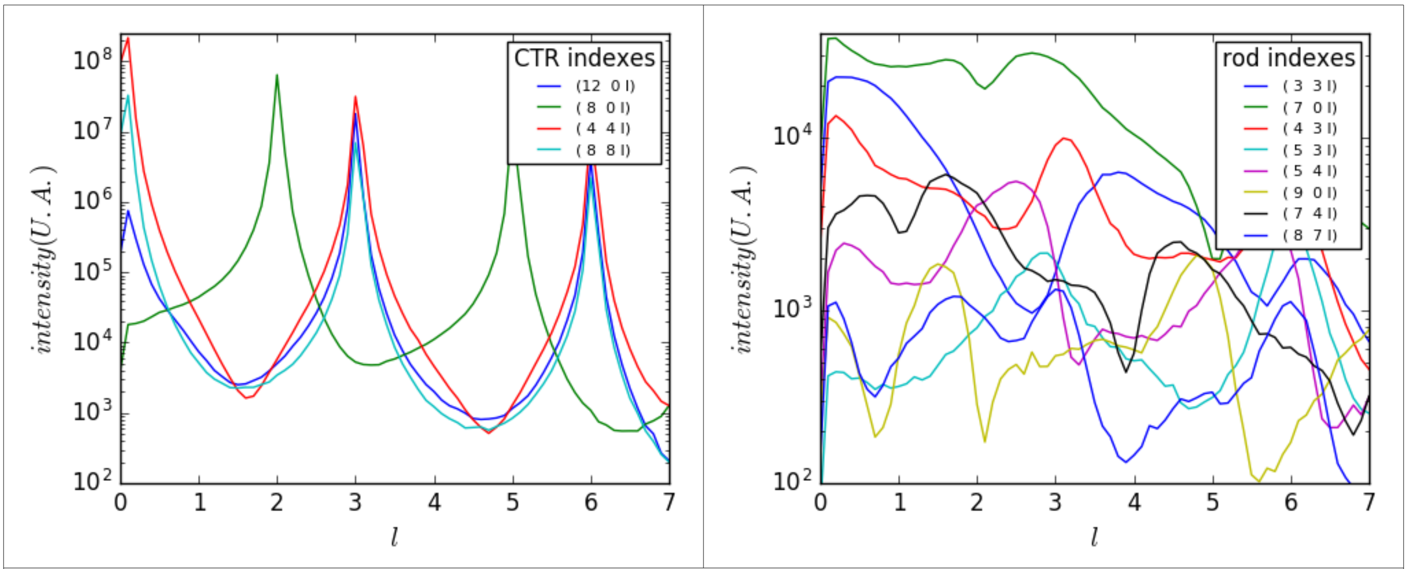

Fig 4. left : Pt CTR scans. Right : $PtSe_2$ rods scans. Rod scans show strong modulations possibly indicating a possible perturbation of the first Pt layer below the $PtSe_2$ layer.

Publication(s):

-[1] D. Jariwala et al. ACS Nano 8, 1102

-[2] Z. Lin et al 2D Mater. 3, 022002 (2016)

-[3] H. Liu et al. ACS Nano 8,6619 (2014)

-[4] Y. Wang et al. Nano Lett, 15, 4013 (2015

-[5] J. Drnec et al. J. Appl. Cryst. 47, 365 (2014)