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: Proximity-induced exchange interaction vs. strain in heteroepitaxial stacks of 2D ferromagnetic layers and topological insulators studied by grazing incidence diffraction	Experiment number: HC-4766
Beamline:	**Date of experiment**: from: 21.06.2022 to: 27.06.2022	**Date of report**: 22.02.2023
Shifts:	**Local contact(s)**: Dr. Juan Rubio Zuazo	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Dr. Martin Schmidbauer (IKZ, Berlin)

Rania Oueslati* (PDI, Berlin)

Report:

To study the in-plane structures of the grown layers we have performed synchrotron-based grazing incidence diffraction (GID) at the Spanish beamline SpLine, BM25B at the European Synchrotron Radiation Facility (ESRF) in Grenoble. GID serves as a highly surface sensitive technique to probe in-plane lattice parameters and their azimuthal distribution in thin layers. If the angle of incidence is close or even smaller than the critical angle for total external reflection the impinging X-ray wave field decays exponentially with increasing depth. In this scattering geometry the diffraction plane stands upright to the surface normal and therefor GID implements the most non-coplanar case. An X-ray wavelength of 0.729 A, corresponding to a photon energy of 18 keV, has been selected by a Si(111) monochromator, in order to access a comparatively large area in reciprocal space. Fig. 1(a) shows the in-plane intensity distribution plotted in hexagonal coordinates according to the symmetry of the SiC(00.1) substrate. For convenience we use the four-component vector notation for hexagonal symmetry: (h k . l) = (h k –[h+k] l). The only substrate contribution within the investigated area in reciprocal space appears as a single (2-1.0)-type of reflection. All further features are either caused by the grown layers, which form hexagonal FGT, tetragonal FeTe and, as a protecting cap, hexagonal Te. This topmost layer yields a sequence of low-index poly-rings with different lattice spacings. However, the most intense and prominent diffraction feature originates from the FGT layer itself. There are two different sets of reflections, namely FGT(11.0) and (22.0), and FGT(h0.0) with [h=1..3]. Please note that both refer to an orientation with its c-axis along the surface normal. The three most intense diffraction features at (11.0), (22.0) and (30.0) result in a FGT in-plane lattice parameter of 4.049(13) A. The intensity modulation *along* the FGT(11.0) ring depicts maxima if the azimuth fits with the SiC[2-1.0] substrate direction indicating a preferential in-plane orientation, i.e. the FGT[11.0] net planes are aligned with SiC[11.0]. There are two more ring-like contributions detectable in the map and in the attached line profile taken at *K*=0, which cannot be related to FGT. These are most probably due to a smaller, non-intentional fraction of tetragonal FeTe agreeing very well with FeTe(200) and (220) in-plane lattice spacing. Interestingly the c-axis of tetragonal FeTe points along the surface as well.

Complementary to GID we have performed omega/2theta measurement, which probes exclusively out-of-plane features of the layer, see fig.1(b). A set of FeTe(00*l*) reflections confirms the presence of tetragonal FeTe with its out-of-plane orientation along SiC[00.1] as can already be anticipated from the GID in-plane data. A further set of eight subsequent FGT(00.*l*) reflections with l = 3, 6, .., 24 proves (in conjunction with the presented in-plana data) the formation of Fe5GeTe2. The position and intensity ratios fit well with a numerically derived powder pattern (black bars). Based on the positions of the most intense contributions at l=3, 6, 18 and 21 we have deduced an out-of-plane lattice parameter of 29.16(9) A. We might emphasize that a potential formation of [00.1]-oriented Fe3GeTe2 can be excluded due to a well distinguishable, different diffraction pattern.

This paragraph will be part of a forthcomming publication (close to submission) with the title *Large-area synthesis of ferromagnetic* $Fe_{5-x}GeTe_2$*/graphene van der Waals heterostructures with Curie temperature above room temperature.*

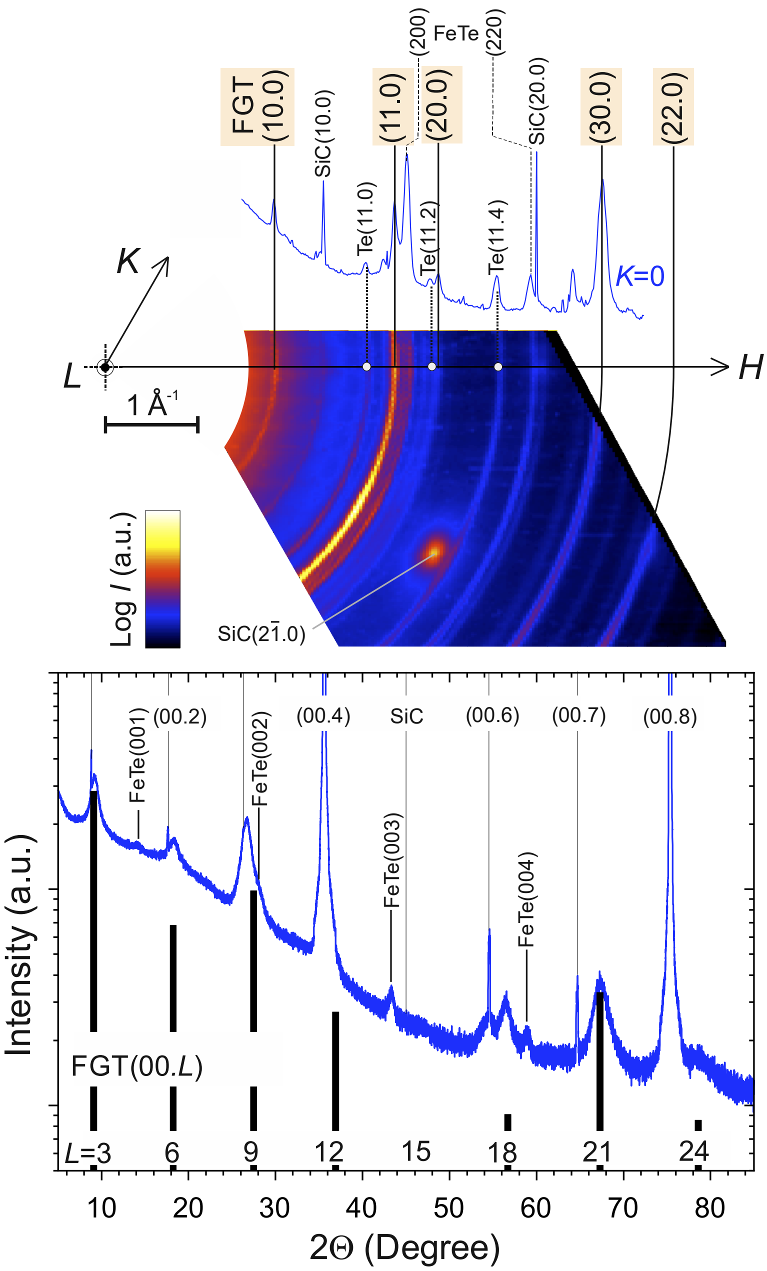

Fig.: 1 Diffusely scattered intensity in-plane as probed by grazing incidence diffraction containing the SiC(2-1.0) substrate reflection. Contributions along particular rings (corresponding to discrete in-plane lattice parameters) can be well attributed to FGT, FeTe and the Te capping layer. The out-of-plane omega/2theta scan contains a series of FGT peaks, which intensities match well with the calculated ones.