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:** The structure of MnF_2 layers within CaF_2/MnF_2 superlattices, grown on Si(111).	**Experiment number**: 28-01-069
Beamline: BM28	**Date of experiment**: from: 7th April 2000 to: 11th April 2000	**Date of report**: 5th Jan 2002
Shifts:	**Local contact(s)**: Anne Stunault	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Scientific Background Magnetically ordered epitaxial films grown on semiconductor substrates are of great interest since they could lead to integrated magnetic and microelectronic systems. Of the possible materials being considered for this application, manganese-based compounds are the prime candidates. The crucial question is whether or not they can be grown on a substrate such as silicon, epitaxially and with minimal disorder. In the bulk, manganese difluoride (MnF_2) is an anti-ferromagnetic insulator with a tetragonal lattice (rutile) structure. Recent reflection high energy electron diffraction (RHEED) studies have suggested that MnF_2 can be grown by MBE on a Si(111) substrate if a thin buffer layer of CaF_2 is first deposited [1]. The results indicate that MnF_2 films thinner than three molecular layers have a cubic (fluorite) structure like that of the buffer CaF_2 layer. It is a possible example of a metastable phase being stabilised by epitaxial growth. We note, however, that the atomic arrangement of growing layers at the surface can be significantly different from that of the 'mature' interface, buried deep inside the final structure [2]. In attempting to understand the properties of heterostructures, it is essential to measure the atomic arrangement of the complete assembly, rather than rely on the evidence of surface sensitive techniques alone.

Aims of the experiment The aim of the experiment was to determine, for the first time, the crystal structure of ultra thin MnF_2 layers grown by MBE. Specifically, we aimed to determine: the atomic geometry of the MnF_2 layers; whether or not the MnF_2 layer undergoes a phase change as the thickness increases beyond 2ML; the registry of the upper CaF_2 layer to the underlying buffer layer; the interfacial roughness; and the effect of the growth temperature.

Experimental procedure A series of MnF_2 / CaF_2 superlattice structures, deposited on Si(111) substrates, were grown by our collaborators at the Ioffe Institute in St Petersburg, Russia. Thick CaF_2 buffer layers (typically 15ML) were deposited at 750 ºC, forming a well-ordered interface. It has been shown that the CaF_2 buffer layers are pseudomorphic (coherent to the silicon substrate). Onto this buffer layer, a superlattice structure is built up, each repeated unit within the superlattice containing thin films of MnF_2 (1-5ML) and thicker CaF_2 films (5-15ML). The final structures were checked for perfection using AFM just after production. X-ray reflectivity scans (along the (00*l*) direction up to *l*=8 to encompass the Si(111) and Si(222) bulk peaks) were completed for 8 different samples on the XMaS beamline at the ESRF, using an X-ray wavelength of 1.24Å, with the samples exposed to the atmosphere. Several regular sets of peaks were observed in the reflectivity scans, each set produced by the different repeated distances contained within the superlattice sample (such as the interlayer spacings, the repeated unit thickness and the entire superlattice thickness). Interference between these sets of peaks means that the reflectivity scan is very sensitive to small changes in the structure and spacing of the layers, particularly in the vicinity of the Si(111) peak and the absent Si(222) peak. A full analysis of the data from a number of samples, using a modified version of the software package *ROD*, is being completed. *ROD* has been used for many years to refine structural models of atomic surfaces. Our software is highly flexible allowing the determination of the 3D co-ordinates of each atom in the superlattice unit cell, rather than simply the electron density profile normal to the surface, as employed in other methods.

Experimental results and analysis – a) MnF_2 layer thickness

The red curve in figures 1 represents the normalised experimental reflectivity measurement of a superlattice with the following parameters:

- CaF_2 buffer 14ML + (MnF_2 2ML / CaF_2 14ML) x 10.
- Type-B Si / CaF_2 interface.

The blue curve represents a simulation with the following parameters:

- CaF_2 layers in bulk state.
- Fluorite MnF_2 layers in the superlattice, with MnF_2 layer spacing equal to 0.97 $\times$ the bulk CaF_2 layer spacing.

Measurements of the average spacing between sets of peaks in the experimental data indicate that the thicknesses of the repeated unit and the entire superlattice are 50.3Å and 548.8Å respectively. The thickness of the entire superlattice is equivalent to 174 layers of bulk CaF_2. This experimental result indicates that there are 14ML of CaF_2 in the buffer layer and within each repeated unit, giving a total of 174 layers (14ML CaF_2 buffer + (2ML MnF_2 + 14ML CaF_2) repeated unit $\times$ 10 = 174 layers). To show that the MnF_2 layers for this sample exhibit the fluorite structure, a new simulation was undertaken to determine how much the layers needed to be contracted to achieve a fit to the data. An unrealistic contraction of the rutile MnF_2 layers by a factor of 0.89, in a direction perpendicular to the Si(111) direction, was required to achieve an acceptable fit between the theoretical and experimental curves.

Experimental results and analysis – b) Structural type of MnF_2 layers Confirmation of the fluorite structure for the MnF_2 layer is provided by the measurement of the scattering near the Si(222) peak; superlattices with rutile layers scatter approximately 10 times more intensely. The results demonstrate that the modified *ROD* software can be used to successfully determine the structure of complex superlattices. The analysis of other structures is continuing with the aim of identifying and characterising the structural phase transition of the MnF_2 layers between the fluorite and rutile structures. This work forms part of an on-going collaboration, between the University of Leicester, the Ioffe Inst. at St Petersburg and financially supported by the Royal Society, concerned with the growth and atomic structure of fluoride layers and superlattices.

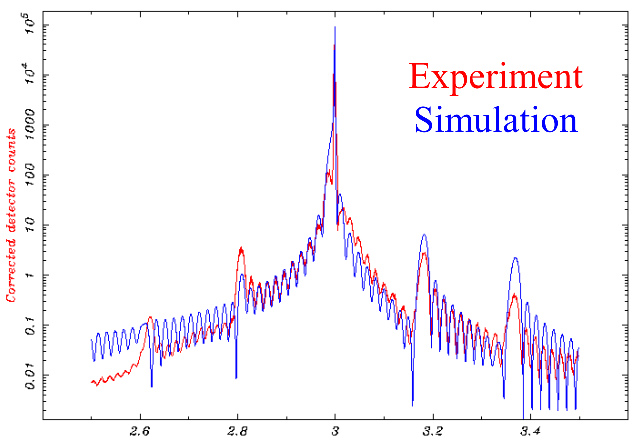

Figure 1: Scattering around the Si(111) peak with Fluorite MnF_2 layers

References

1. N. L Yakovlev, A. G. Banshchikov, M. M. Moisseeva, N. S. Sokolov, J. L. Beeby and P. A. Maksym, Surf Interface Anal. **27** (1999).
2. K. A. Edwards, P. B. Howes, J. E. Macdonald, T. Hibma, T. Bootsma, M. A. James, PHYSICA B, 1996, Vol.221, No.1-4, pp.201-204.