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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:** **In Situ Topographic Investigation of Ion Implanted $A^{III}B^{V}$ Semiconductor Compounds during Thermal Annealing**	**Experiment number**: HE1277
Beamline: ID19	**Date of experiment**: from: 08. 03. 2002 to: 10. 03. 2002	**Date of report**:
Shifts: 8	**Local contact(s)**: dr. Jürgen Härtwig	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

*Dr. Wojciech Wierzchowski, Institute of Electronic Materials Technology, Warsaw, Poland

*Dr. Krzysztof Wieteska, Institute of Atomic Energy, Świerk, Poland

*Dr. Stephan Grigull, ESRF, Grenoble, France

Report:

In Situ Topographic Investigation of Ion Implanted $A^{III}B^{V}$ Semiconductor Compounds during Thermal Annealing

1. Introduction

Thermal annealing is present in a great number of technological processes used to produce modern electronic devices. The achievment of good technological results is therefore strongly dependent on the understanding and controllability of the physical phenomena taking place in during the thermal processes.

The study of strain relaxation of ion implanted crystals during the annealing allows establishing the role of different point defects and their complexes. Information on these phenomena is usually obtained through the identification of characteristic activation thresholds corresponding to different point defects and their complexes [1-4].

In the present project we performed *in situ* measurements of thermal annealing in implanted $A^{III}B^{V}$ semiconductors with the method eliminating some disadvantages of nuclear analysis, as damaging of the sample by the probe ion beam and relatively long time of measurements.

2. Experimental

The idea of the experiment came from our previous experience with studying layered structures with Bragg-case section topography, which pointed out the possibility of obtaining the information equivalent to those from rocking curves using a highly asymmetric reflection [5-7]. As for the usual Laue method, the section patterns do not directly reveal the effect of lattice parameter changes but only show lattice orientation changes.

The realisation of the experiment was possible thanks to high brilliance of the source and availability of high resolution CCD camera at ESRF. White beam Bragg-case section patterns were recorded on a high resolution CCD camera placed at relatively large (50 cm) film-to-crystal distance. The sample was located in the heating device in an Ar atmosphere. The width of the beam was close to 7 μm and the glancing angle was 5^o. The CCD camera recorded chosen 511 reflection at 0.77 Å. Images with reasonable quality required 20 seconds of data collection. The observations were performed during a linear increase (constant rate 6^o/min) of temperature from room temperature to 400º C. This allowed recognizing the activation thresholds of point defects. The observation with the CCD camera was continued also during the cooling of the sample.

The *in situ* observations were performed in a number of samples including MOCVD grown epitaxial layers of $Al_xGa_{1-x}As/GaAs$ with x=0.43 and GaAs substrates implanted with various doses of 1.5 MeV Se^+, 170 keV He^+ and 90 keV H^+ ions. In case of light H^+ and He^+ ions the doses reached 1×10^{17} /cm^2 and for Se^+ 6×10^{15} /cm^2. The samples contained an initial dislocation density smaller than 10^3cm^{-2}. In all cases the ion penetration range was close to 1μm.

2. Results

The character of the section pattern was different for $A^{III}B^V$ semiconductors implanted with different ions. Low energetic light H^+ and He^+ ions formed a strain maximum located very close to maximal ion penetration range. This local strain maximum caused formation of characteristic "strain modulation fringes" in the section pattern caused by beating of the radiation reflected by the layers with the same deformation on two slopes on the maximum. For lower doses of high energetic heavy Se^+ and As^+ ions, when the effect of point defects of the matrix dominated, we often observed almost constant strain in a layer close to the surface. In that case the implanted layer formed a single stripe in the section pattern.

The results of strain relaxation are illustrated by the plots of strain as function of temperature, shown in Figs. 1-2. The strain was determined from distance of corresponding maxima in the plots of intensity distribution obtained from recorded image files. In case of hydrogen implanted samples the strain for higher temperatures was determined with lower accuracy measuring the area with noticeable increased intensity.

In all obtained plots the relaxation started at 120^o – 150^oC but further changes were different for various kinds of implantation.

As may be seen in Fig. 1, the fastest relaxation takes place in case of implantation with H^+ ions. At the beginning the relaxation is faster for strain in the maximally deformed layer, becoming slower upon 200^o C when the strain is less than 1/3 of the initial value. After reaching 320^o C the strain falls in the whole layer to less than 10% of initial value. The character of strain relaxation for implantation with hydrogen ions was similar in GaAs and $Al_xGa_{1-x}As/GaAs$.

For samples implanted with He^+ ions the relaxation is slower (Fig. 2) and at 400^oC we still have 30% of initial strain value. In case of $Al_xGa_{1-x}As$ with x=0.43 implanted with 170 keV He^+ ions to the dose 6×10^{16} /cm^2 we observed a rapid complete delamination of the shot through layer when the temperature reached 250° C.

For investigated $Al_xGa_{1-x}As/GaAs$ implanted with 1.5 MeV Se^+ ions to the dose 6×10^{15} cm^{-2} the strain profile is a sum of two components coming from the point defects induced in the matrix and from the Se atoms. The relaxation of first strain component is similar as in case of He^+ implanted samples. The strain starts to relax close to 140° C and decreases to less than 45% of the initial value with the rate firstly slightly increasing, but later again decreasing close to 350° C. The second component is much less affected by the applied heating and decreased less than 15%.

References:

[1] Partyka P., Averback R.S., Coleman J.J., Erhart P., Jäger W.: Mat. Res.Soc. Symp. Proc. **354** (1995) 218
[2] Tan H.H., Jagadish C., Wiliams J.S., Zou J., Cockayne D.H.J., Sikorski A.: J. Appl. Phys. **77** (1995) 87
[3] Stonert A., Turos A., Breeger A., Wendler E., Wesh W.: Nucl. Instr. Meth. B **157/177** (2001) 219
[4] Turos A., Stonert A., Nowicki L., Breeger A., Wendler E., Wesh W., Fromknecht R.: Nucl. Instr. Meth. B **157/177** (2001) 219
[5] Wierzchowski W, Wieteska K, Graeff W, Turos A.: J Alloys and Comp **286 (**1999) 342
[6] Wierzchowski W, Wieteska K, Turos A, Graeff W, Gawlik G.: Vacuum **63 (**2001) 767
[7] Wieteska K, Wierzchowski W, Graeff W, Turos A. J Phys D: Appl Phys **34** (2001) A122.

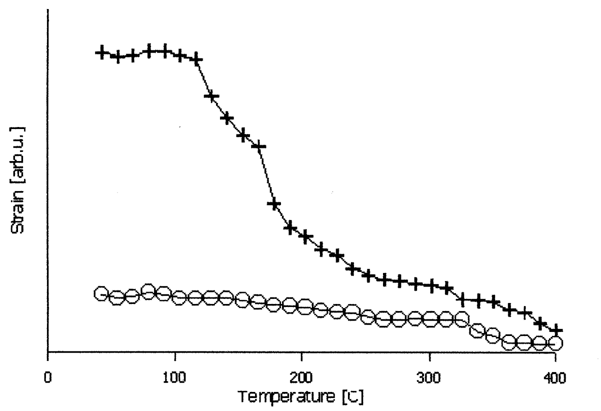

Fig. 1. Plot of strain changes in course of heating from 25° C to 400° C of GaAs implanted with 90 keV H^+ ions to the dose 3×10^{16} cm^{-2} presenting the strain in mostly deformed layer (crosses) and the strain close to the surface (circles).

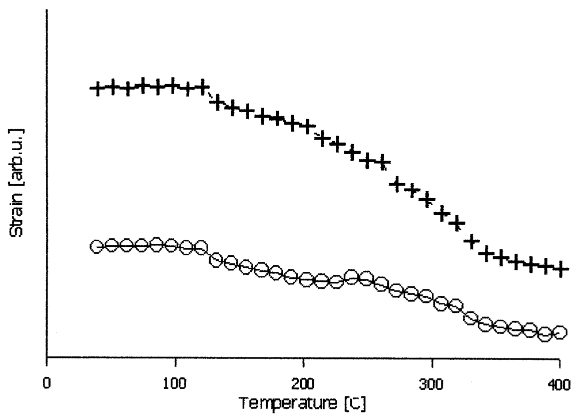

Fig. 2. Plot of strain changes in course of heating from 25° C to 400° C of $Al_xGa_{1-x}As/GaAs$ with $x=04,3$ implanted with 170 keV He^+ ions to the dose 3×10^{16} cm^{-2} presenting the strain in mostly deformed layer (crosses) and the strain close to the surface (circles).