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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 using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

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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.

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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 of defect structure and annealing behavior of $LiNbO_3$ before and after He-implantation by X-ray diffuse scattering under grazing incidence	**Experiment number**: SI-455
Beamline: ID10B	**Date of experiment**: from: 26-Feb-99 to: 02-Mar-99	**Date of report**: 1-Mar-01
Shifts: 12	**Local contact(s)**: Dr. Bernd STRUTH	*Received at ESRF:*

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Report:

He-implantation is considered as a promising way to fabricate near-surface waveguide layers in $LiNbO_3$ for optoelectronics and surface acoustic wave device applications. Modification of the refractive index is due to the implantation-induced lattice damage. However, point defects which are created as a result of ion implantation are very sensitive to subsequent heat treatments. It turned out, that annealing at T > 200 C leads to the clustering of points defects which gives rise to an intense diffuse scattering in the vicinity of bulk Bragg reflections [1]. X-ray topography studies of He-implanted $LiNbO_3$ crystals showed the presence of the sub-micron bubbles or cavities separated by 300 – 400 micron distances [2,3]. In order to receive more detailed information on defect formation and clustering near the surface of the He-implanted $LiNbO_3$ crystals we accomplished this study which utilizes the measurements of x-ray diffuse scattering under grazing incidence conditions.

The sample set consisted of the 10x15 mm^2 pieces: virgin, as implanted, implanted and annealed for 30 min in forming gas at temperatures between 200 and 400 C, all of them cut from the Y-oriented $LiNbO_3$ wafers. He-ions with an energy of 320 keV were implanted through the polished surface of $LiNbO_3$ up to a dosage of 10^{16} ions/cm^2. Special precautions were undertaken in order to keep the temperature of the samples near the room temperature during the long-term implantation procedure. A forming gas flux and pressure were optimized to prevent an erosion of the sample surface during heat treatments.

The x-ray diffuse scattering measurements were performed by collecting reciprocal space maps (RSM) in the vicinity of surface reflections (006) and (220). Corresponding diffraction vectors are parallel to the Z and X-axes of the $LiNbO_3$ unit cell (in hexagonal setting). Together with bulk (030)-reflection measurements (diffraction vector parallel to the Y-axis) these surface reflections provide comprehensive information on defect distributions. A usage of a position sensitive detector (PSD), which was placed parallel to the sample surface, reduced the data acquisition time and enabled as to collect the scattering intensity from the reciprocal space region within about 10% of the Brillouin zone. Most of the diffraction scans were collected at incidence angle $\alpha = 0.45^o$, which is larger than the critical angle ($\alpha_c = 0.3^o$). By using the PSD, the scattered intensity was integrated in the direction perpendicular to the surface, around an exit angle which also was larger than the critical angle. Thus, the grazing incidence diffraction signals were collected from a layer approximately hundred of nm in depth. In order to discriminate the static diffuse scattering from the thermal diffuse scattering, the RSMs were also taken from virgin samples for background subtraction.

The RSMs taken from implanted samples in the vicinity of (220) reflection and (006) reflection revealed significant anisotropy of the defect strain fields. In the (220) maps the diffuse scattering contours were found to be centered exactly at the (220)-node of reciprocal lattice. The annealing procedures did not result in visible changes of diffuse scattering intensities. On the contrary, the diffuse scattering contours measured around (006)-reflection, were found to be centered at the position significantly shifted from the (006)-node along the $q_{||}$ direction. In the as-implanted samples the diffuse scattering intensity was so strong that it looked as an additional peak separated from the Bragg maximum by an angular distance which corresponded to $(\Delta d/d)_z = -1.1\cdot10^{-2}$ (lattice contraction effect). Post-implantation heat treatment led to considerable reduction of both the additional peak intensity and its separation from the Bragg peak. It was also found that annealing at temperatures higher than 400 C resulted in complete vanishing of the second peak.

We assumed that the strong diffuse scattering is originated in local domains near the crystal surface enriched by vacancies, which are responsible for lattice contraction there. Vacancy-rich near surface layer is formed as a part of implantation damage after fast diffusion of the part of interstitial defects to the crystal surface. Similar features were observed in x-ray diffraction studies of implantation damage induced by the 320 keV oxygen ions in Si wafers [4]. By drawing the cross-sections in the RSMs over appropriate directions in reciprocal space it is possible to extract angular diffraction profiles of the diffuse scattering along the $q_{||}$ and $q_{\perp}$ axes. These diffraction profiles are well described by Lorentzian functions with equal widths, which correspond to the domain radius R = 6.3 nm in as implanted samples. Basing on the integrated intensity of diffuse scattering, we estimated the volume fraction of vacancy-rich regions in the He-implanted 100 nm thick near-surface layer to be about 25%. In case that interstitial atoms are preferably those of Li and He, the Li-vacancies will be ordered along the Z-axis, which explains the observed differences between the (006) and (220) RSMs.

This experiment continued the methodical aspect of existing studies of implantation-induced defects by means of grazing incidence surface scattering [5-7]. It also gave complementary results to RBS channeling [8] and conventional large-angle x-ray diffraction studies [1,7] on defects in $LiNbO_3$ induced by He implantation.

[1] Y. Avrahami and E. Zolotoyabko, NIM B **120** 84 (1996)

[2] E. Zolotoyabko, D. Shilo, W. Sauer, E. Pernot and J. Baruchel, Appl. Phys. Lett. **73** 2278 (1998)

[3] E. Zolotoyabko, D. Shilo, W. Sauer, E. Pernot and J. Baruchel, Rev. Sci. Instr. **70** 3341 (1999)

[4] T. Ahilea and E. Zolotoyabko, J. Crystal Growth **198/199** 414 (1999)

[5] W. Sauer, T.H. Metzger, J. Peisl, Y. Avrahami and E. Zolotoyabko, Physica B **248** (1998) 358 (1998)

[6] S. Grotehans, G. Wallner, E. Burkel, H. Metzger, J. Peisl and H. Wagner, Phys. Rev. B **39**, 8450 (1989)

[7] E. Zolotoyabko, Y. Avrahami, W. Sauer, T.H. Metzger and J. Peisl, Materials Letters **27** 17 (1996)

[8] A. Kling et al, NIM B **113** 293 (1996)