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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

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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 logo] ESRF	Experiment title: "Study of surface acoustic wave devices by X-Ray stroboscopic diffraction imaging" Experiment session: BM05 10-12-2008/15-12-2008.	Experiment number: MA/622
Beam line: BM05	Date of experiment: from: 10/12/2008 to 15/12/2008	Date of report: 29/08/2009
Shifts: 15	Local contact(s): Jürgen Hartwig. B.Ziegler.	*Received at ESRF:*

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Report:

Study of surface acoustic waves propagating on quartz and lithium niobate.

These experiments were designed to understandand some unexpected contrasts previously observed at ID19 in white beam section topographs images of surface acoustic waves (SAW) propagating along the YZ direction in lithium niobate resonators. Two different kinds of images were observed respectively with SAW of moderate and of large amplitude using similar diffraction conditions (0,3.0 reflexion, Bragg geometry) that give mostly information about the normal component of the SAW.

a/ Section images of SAW with a moderate amplitude (propagation along YZ in LiNbO₃).

When the collimation slit, used to define a thin X-ray (white) beam, is, as usual, oriented parallel or perpendicular to the acoustic wave front no particular featu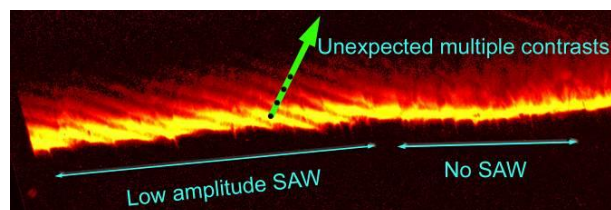res appear in the section images. However, when the resonator is rotated around its normal, starting with a slit parallel to the surface wave front, unexpected white beam section images of the SAW are obtained that contain multiple contrasts extending far away from the expected width of the section image (figure 1: rotation Ψ# 25°). Five to six successive fringes of the SAW with decreasing intensities are visible. The first one is much larger than the corresponding image of the non-vibrating part of the resonator. Such contrast is not observed in monochromatic images (figure 2). Several mechanisms can explain the multiple contrasts appearing in figure 1. One possibility is related to the role of the harmonics of the experimental wavelength (λ=1.25Å for θ=25°) that
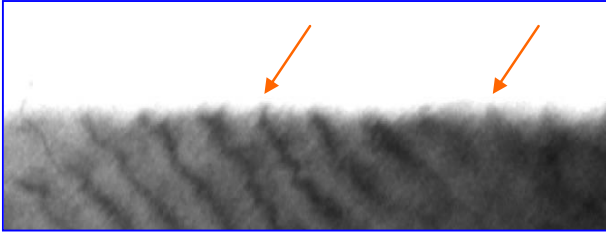

Figure 1:White beam section image{ (0,3.0) reflexion, Bragg geometry, diffraction vector normal to the surface, θ=25° λ=1.25Å, Ψ# 25° around the normal, false color to enhance the visibility of the multiple contrasts.

Figure 2:Monochromatic image limited on one side by a slit (20keV same reflexion). No multiple contrast

can image parts of the SAW situated at depth increasing with the harmonic rank. Another possible explanation can be linked to the multi-trajectories diffraction that can exist in deformed crystals [1][2]. Such multi-path diffraction was predicted and observed in crystals curved by a constant strain gradient [1]. It may be extended to the case of a sinusoidal displacement, as in the case of SAW, which gives a somehow similar distribution of strain in several parts along a period.

The images as well as the rocking curves registered, using a monochromatic radiation (20 keV) do not show multiple contrasts nor oscillations in the rocking curves. Similar experiments made with SAW propagating on ST cut quartz have given similar results (figure 3).
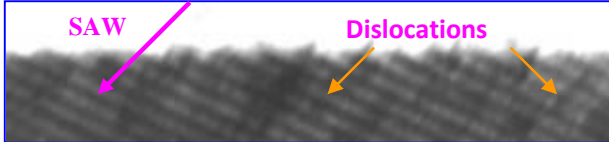

Figure 3:Monochromatic image of SAW on ST quartz. The contrasts nearly orthogonal to the SAW wave fronts are due to the dislocations present in this substrate. (20keV, (01.-1) reflexion) .

This indicates that the contrasts appearing in the white beam images, as those of figure 1, are most likely due to the harmonics contained in the white beam. In order to formulate a definitive conclusion, simulations are in progress to confirm the experimental results and to gain further insight of the diffraction mechanism involved in the formation of the white beam translation images of surface acoustic waves. The experimental results have been presented and published [4-5]. It is planed to publish the results of the comparison between the experimental images and the simulated ones as soon as the simulation program will be ready.

It should be noticed that the knowledge of the mechanism responsible for the multiple contrasts will allow to get a better understanding of the variations of the amplitude as a function of the depth as it was previously fund by means of simulations [6] .

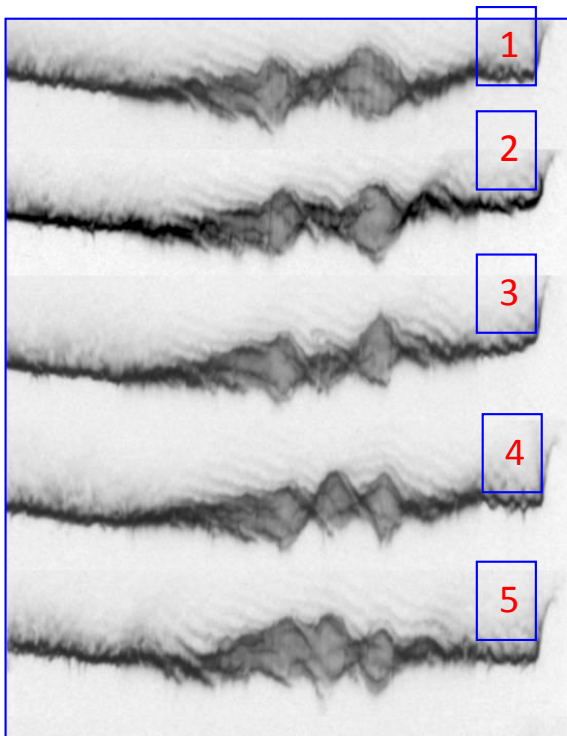**b/ Observation of the propagation of SAW having a large amplitude.**

Figure 4 : Large SAW amplitude. Series of section topographs giving a cartography for the amplitude.

The section topographs, obtained with the same diffraction conditions as for the small amplitude and with a much larger excitation voltage, exhibit another kind of contrast whose width is proportionnal to the local amplitude of vibration. The previous multiple contrasts exist also in the section images (figure 3).

When a monochromatic radiation is used these constrasts as well as the multiple ones disapear. A small increase of the width of the rocking curve is observed for the large amplitude SAW indicating that the acceptance of the reflexion increases. The variation of the width of the section image with the amplitude of the SAW is due to a mechanism similar to the one observed for the bulk acoustic wave of large amplitude [3] (see below) which appears when the strains become too large to satisfy the hypothesis of the dynamical theory. Then, a quasi kinematical process due to a too important rotation of the lattice plane, occurs and enlarges the section image as a function of the amplitude of the SAW. This phenomenum can produce a non negligible divergence of the diffracted beam (figure 4) and explains the influence of the film-sample distance on the shape of the image.

This contrast may be explained according to the drawing of figure 5: the incoming wave is scattered at different depths inside the resonator.
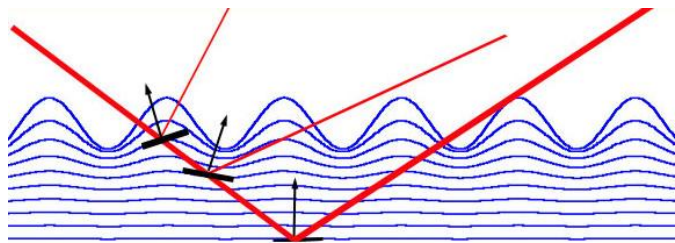
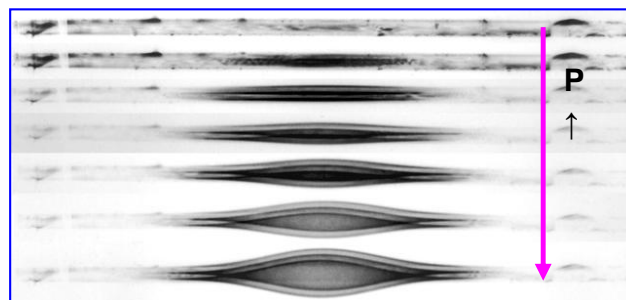Figure 5 : mechanism for image formation of the SAW contrast

Figure 6: Section images of a bulk wave (thickness shear) resonator with increasing amplitude of vibration. Experiment made at ESRF Beam line ID19 07/2001

The mechanism is the same as the one already oserved [3] in the case of bulk waves (figure 6). The local width of the section topograph becomes proportional to the amplitude of vibration, when the amplitude is sufficiently important; it becomes also proportional to the film-sample distance.

A first communication was given at the 2009 IEEE International Frequency Control Symposium [5]. It has been accepted for publication in the proceedings [6].

The practical interest of the observations made is quite important since it allows to obtain very simply, a map of the vibration amplitude in SAW devices (figure 4).

Work is in progress to extend the domain of validity of the already known programs for the simulation of X-Ray topographs to simulate these images.

Détailed study of the propagation of high amplitude bulk shear waves.

Stroboscopic translation topographs have been recorded with a monochromatic wave for highly excited bulk wave quartz resonators. The corresponding rocking curves have also been registered. The aim was to further understand the observations reported in figure 6 in section topographs. These experiments (E=20Kev, Laue Geometry, (-2,1.0) reflection) show a noticeable enlargment of the rocking curve when the amplitude of the vibration mode is increased to the values where these new contrasts appear.

These contrasts occur when the vibration amplitudes becomes too large to give rise to the usual dynamical constrasts observed for lower wibration amplitude which can be predicted by simulations made using the equations of the dynamical theory of X-ray diffraction. The simulations should be able to better take into account the kinematical part of the contrast. These observations for the bulk wave resonators are of similar nature as those made for large amplitude surface wave (figure 4). They show that the new contrasts observed in both cases have the same origin. However, due to the resonant nature and the high Q of the vibration mode of the bulk wave quartz resonators, a more intense effect can be observed for the bulk waves than for the surface wave devices.

Preliminary observation of irradiation effect on quartz resonators (white beam and monochromatic beam) and new possibilities offered by the synchrotron radiations.

The experiments made to study the bulk wave propagation under strong excitation were a good opportunity to study the variations of the resonance frequencies of the quartz samples as a function of the exposure to the white beam or to a monochromatic beam (20 keV). Noticeable frequency shifts occur during these experiments. After an exposure of the order of 10-20 minutes (white beam) the frequency becomes stable, the value being very dependant on the purity and on the perfection of the quartz material used to build the resonator. The starting frequency was always 5680691.Hz, the frequency shift in the range of 8-10Hz for the best material, and of 24-26 Hz for another resonator of high purity. These variations are linked to irradiations defects resulting from the capture, by the impurities or the lattice imperfections, of the electronic carriers generated during x-ray exposure. These irradiations defects have different energies and thermal stabilities. Their destruction leads to carrier recombination and often to luminescence. A very intense luminescence was observed at room temperature for some quartz samples exposed to the white beam.

The study of this luminescence can be a powerfull method to study the defects creation-anhilation equilibrium at different temperature. Other methods to study the irradiation defects can also be used simultaneously (ESR, etc..).

On the whole, the interest in the use of the synchrotron radiations (possibility a large flux in a large energy range) combined with various kinds of spectroscopies, is very probably important to efficiently study many kinds of irradiation defects in various materials.

A better knowledge of irradiation effects in quartz is presently needed to improve the performances of the high stability oscillators used in space applications.

References

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[5] Bernard Capelle, Yves Epelboin, Jacques Détaint, Alain Soyer, Jürgen Härtwig. Time Resolved Synchrotron Radiation X-Ray Topography Study of Surface Acoustic Waves Propagation Communication to the joint meeting of the 2009 International IEEE Frequency Control Symposium and of the European frequency and time Forum Besançon Avril 2009.
[6] Bernard Capelle, Yves Epelboin, Jacques Détaint, Alain Soyer, Jürgen Härtwig Accepted for publicationin the 2009 IEEE Frequency Control Symposium proceedings (IEEE Press 2009).