MATERIALS SCIENCE APPLICATIONS OF HEAVY ION BEAMS FROM THE GUSTAF WERNER CYCLOTRON

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Abstract

Heavy ion beams, most often xenon at 8.3 MeV/u, from the Gustaf Werner cyclotron at The Svedberg Laboratory are used to create ion tracks in various materials such as quartz, polymers, mica and glass. For some applications these ion tracks are etched in order to create micro structures like tuning forks or materials with well defined pore geometries. In other applications the deposited energy is used for disordering the structure in order to modify superconducting films or to create ultra-hard materials. We describe the experimental setup with its control system and discuss the various experiments.

1 THEORY OF ION TRACKS

When high energy heavy ions impinge on matter, they travel a certain distance in the matter and slow down by transferring their energy (mostly) to the electrons. The range of a 8.3 MeV/u xenon ion in a polymer is 0.1 mm. As a result, a very small volume (a tube with a length of 0.1 mm and a radius of 7 nm) has to dissipate this energy.

When the matter has a crystalline structure (like quartz), the thermal shock leaves an amorphous track behind, which is shown figure 1.





In the case of a plastic, all long polymer strings will be broken and lighter molecules will be left along the track. These smaller molecules are easy to flush out by alcohol or can be etched out in alkalic solutions.

When well controlled 3 dimensional structures are required one has to use masks. One can use masks during etching or during the irradiation.

The easiest of the two ways is to first irradiate the complete structure. After this a lithographic mask can be mounted on the surface. It is then impossible for the etchant to reach the pores under this mask, allowing only the required structure to be etched out.

The main disadvantage of this technique is that the complete structure contains latent tracks, diminishing material properties. Using a mask during the irradiation does solve this problem. These masks need an aspect ratio comparable with the stuctures that are going to be produced, making them rather expensive.

The ion track technique actually introduces an anisotropy that can be determined by the user, while normally this is determined by the crystal structure.



Figure 2

2 THE FACILITY

The ion track irradiation facility is placed in the Gamma Cave at the The Svedberg Laboratory. It has the following features:

- Homogeneous irradiation of 40 x 40 mm² samples • by beam-scanning
- Doses from 10^6 up to 10^{12} tracks/cm²
- Remote control of 5 samples at a time •

- Fast sample change modules (click connection)
- Samples in fixed 90 degree or free rotational (1axis) mode
- Angular accuracy of 1.5 mrad in fixed 90 degree mode
- Angular accuracy of 0.8 degree in free rotational mode with on-line laser alignment
- Online feedback of delivered dose

Normally, xenon ions with an energy of 8.3 MeV/u are used, but in neon was also used and an uranium beams are being developed. The typical range of the xenon ions are 30 µm in dense scintillator materials like CsI, 55 µm in quartz and silicon and over 100 µm in polymers.

The irradiation facility itself consists of a vacuum chamber in which a rack of 5 samples can be mounted by a click connection. The sample changer and rotor is an in-house developed mechanism that spirals down a sample rack so it both changes and rotates the sample to the desired position.

Figure 3 shows how the different control elements are connected through VME modules to the central cyclotron control system.

The sample movement is controlled by a stepping motor. Most samples are irradiated at 90° incident angle of the ions. The samples can however also be placed in a rotational mode. For precise determination of the angle, a laser and camera can be used for on-line calibration. The delivered dose is also measured and registrated by a current digitizer. The complete chamber acts as a Faraday cup. The beam scanning is performed by standard steering magnets fed by a fast bipolar power supply.



Figure 3

In order to control the irradiation dose without retuning the complete cyclotron the beam is chopped by an electrostatic deflector at a 50 Hz frequency with variable duty cycle. In order to automatically determine the parameters the beam current is measured regularly with a Faraday cup upstream.

As described before, many elements which are used in this facility are standard beam line items. Since these are normally controlled by a SUN work station (routed to VME controllers), the experiment itself is software controlled from this station. This task was eased a lot by using the TCL scripting language combined with the WISH graphical user interface. In a special version of these script languages, the control elements are all defined as variables, which makes the control to the VME crates transparent. Also the graphic interface is straight forward, which makes extensive knowledge of C++ and the X-windows interface unnecessary

Another nice feature recently introduced is to make patterns on the samples. Not only gradients can be made but also complete bitmaps.

Figure 4 shows an example of the user interface of the scan control. The window indicates a module to completely pre-program an irradiation for a sample rack. For more sophisticated runs, other TCL-modules can take over which tailors the irradiation parameters.

ildem3.tcl							
Sample holder type	Sample	Sample holder name: Sample_d_31.dat					5
🕹 5 samples	Sam	ple positio	n dose	degree	measured	Dose:	2.0E+07
🕹 6 samples	1	0 front	2.0E+07	0	-	Measured dose:	1.16704e+07
	2	0 back	2.0E+07	0	-		
↓ 12 samples	3	54 front	2.0E+07	0	2.95407e+07	Scan ove	erview
J	4	54 back	2.0E+07	0	-	00000	0000
	5	108 front	2.0E+07	0	-	00000	0000
PANIC	6	108 back	1.0E+07	0	-	00000	0000
	7	162 front	1.0E+07	0	-		0000
	8	162 back	1.0E+07	0	-	00000	0000
Commands	9	216 front	1.0E+07	0	-	00000	0000
Start scan Load sample	10	216 back	1.0E+07	0	-	00000	0000
Complete scan Save sample	11		1.0E+07	0	-	00000	0000
Reset Exit	12		1.0E+07	0	-	00000	0000
			Copy down	Copy down			
L.							

Figure 4

3 APPLICATIONS

3.1 MITE

MITE relies on the merging of tracks etched into pores in certain areas as defined either by a mask during etching, or as presented here, by a stencil mask during ion beam irradiation. In contrast to the earlier MITE, this latter concept does also define the intended microstructure laterally, and could hence be regarded lithograpical.

In the first study close to vertical walls and nearly perfect corners could be realized in single crystalline quartz irrespective of crystallographic direction. Figure 5 contains an illustrative example of localized ion track etching in quartz.



Figure 6

Figure 5

The etching has been interrupted half way and the mask material has been removed. In this case the area was defined by thin film masking and conventional lithography after irradiation.

By using non-orthogonal irradiation, inclined structures, figure 6, have been accomplished.

From the theory it is evident that latent, undeveloped, tracks still exist in the remaining parts of the structure. These tracks, which could have a deteriorating influence in some cases, can sometimes be removed by annealing but in quartz however at the price of a phase transition that lowers the piezoelectric performance of the material.

Therefore a new scheme is introduced - the projection MITE. The technique employs stencil mask during irradiation to protect the parts intended to be retained after etching not only from developed tracks, as before, but also from latent tracks.

Perhaps not very surprising, but in contrast to earlier results with uniformly irradiated samples, the appearance (edge and corner definition) was found to improve with higher fluence for projection MITE. Figure 7 shows three structures with inclining fluence and decreasing etch times. The portions shown in the first two figures are masked by the same LIGA mask which was also aligned along the same crystallographic direction with an accuracy of a few degrees. The sample in figure c) was shadowed by a slightly different mask and can therefore be compared to the others by edge and corner



Figure 7

definitions only, not by widths or pitch.

The surface roughness (between ridges) differed much between sample a) (10^8) and b) (10^9) , while another magnitude's increase in fluence, sample c) (10^{10}) , seemed to have little effect.

3.2 Photonic bandgaps

Photonic bandgap structure in the 10 mm wavelength band may be interesting in applications like, e.g. laser protection. The aim is to produce a photonic bandgap structure in the 10 μ m wavelength band. This will require the fabrication of a highly porous structure with the symmetry of the fcc lattice and with sufficient refractive contrast in air. The repetition distance should be in the range 5-15 mm. An attractive possibility for this new joint project is to use the ion track microtechnology to prepare the laterally controlled elongated voids in a suitable polymer matrix, that subsequently are filled with high index material. The polymer is either left, if its refractive index is low enough, or etched away. To be able to make this we need at least shadow masked MITE, and maybe also combined with an lithographically defined surface mask.

3.3 Beam splitter for soft X-rays

In X-ray optics there is a wish to have beam splitters which can be used for radiation of wavelengths less than 10 nm and with a lateral coherence of several microns or less. In the present project we aim at manufacturing a structure which allows X-ray photons to be coherently split in a transmitted part and a reflected part. The structure consists of a grazing incidence mirror, with a thickness of a few microns and supported by an integrated thick structure in order to maintain a flat surface figure. The mirror should have a great number of holes of diameter less than a micron, directed at an angle of typically 5 degrees with respect to the surface and going all the way through the mirror. For 5 degrees angle the length of the holes would be about 50 microns for a 4 microns thick mirror. In order to produce such a device we propose to use energetic ions to create tracks in a suitable material which can subsequently be etched to obtain long straight holes through the structure.

3.4 Porous media

Data was taken on polymer foils irradiated by 8.3 MeV/u ¹²⁹Xe ions at the TSL cyclotron. The pores were saturated with KCl solution of a wide range of concentrations, and the samples were mounted between metallic electrodes.

In impedance spectroscopy, one applies an ac signal between the electrodes and measures the in-phase and out-phase response in a wide frequency interval. Three different processes for ion mobility/electrode effects were seen. They vary in a characteristic manner when the ion concentration is varied. The high frequency process corresponds to bulk conduction in the pores. At low concentrations conduction at pore surfaces is observed, while the bulk contribution dominates at higher concentrations. The underlying physics is not understood in detail, but extensive analytic work will be commenced as the measurements are completed.

4 CONCLUSIONS

The facility has within short time attracted many users, of only only a few projects were discussed here. We believe that the facility is a powerful tool for modern Materials science research.