

# Nanomachining of Hard X-ray Crystal Optics

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Deterministic nanomachining based on ultraprecise axes and single point diamond tool was used to shape Ge and Cu crystal X-ray optics. Flat and modified channel-cut crystals (beam compressors and expanders) were nanomachined and their roughness, subsurface damage and shape precision were evaluated by means of AFM, micro Raman spectroscopy and X-ray diffraction.

## 1 Introduction

Standard technology of preparation of crystal X-ray optics (Si, Ge,...) is based on stochastic brittle mode of material removal. It is still valuable for the open planar and simple curved surfaces (spherical, cylindrical), and with some limitations also to the confined ones, e.g. channel-cut surfaces.

Ductile or plastic mode is known from metals and shaping of single Cu crystals in this mode is straightforward. This ductile mode is used nowadays also for preparation of the Ge infrared optics. Contrary to the standard stochastic technology this technique is deterministic in its nature because of ultraprecise NC axes and a single point diamond tool (SPDT) [1]. When decreasing the depth of cut or chip thickness below a critical value, material removal mechanism changes from the brittle through brittle-ductile to ductile regime even for brittle materials such as single crystal silicon and germanium. Simple and complex surfaces such as aspheres in general can be prepared with submicrometer precision in this way.

The application to the soft X-ray and even hard X-ray optics for  $> 5$  keV photon energies is in progress as this nanotechnology may significantly improve shape precision, surface roughness and subsurface damage (SSD) in crystal X-ray optics. Some development will be needed especially in the confined channel-cut monochromators unless special design of the channel shape be designed.

## 2 Experimental

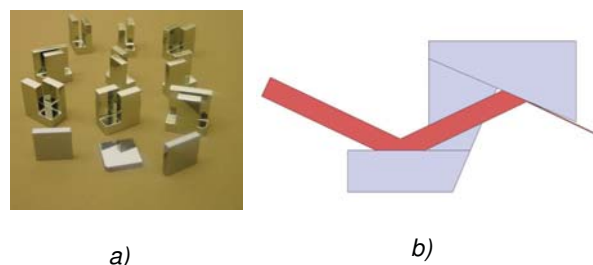
Low dislocation or dislocation-free Ge, Si and Cu single crystal ingots of (100) and (111) crystal orientation were used to cut (110) oriented plates of about 22x22x1.5 mm. Cutting with an ID diamond saw was done with X-ray orientation within  $\pm 0.1^\circ$ .

A set of samples with a miscut angle of  $17^\circ$  from (110) plane was prepared as well. Standard technology of lapping, chemomechanical polishing and final chemical polishing was used to prepare reference samples.

Deterministic crystal machining was performed in several modes of operation using an FG 350 nanomachining centre from Moore, Keen, USA, installed in Integra TDS premises (<http://www.integratds.eu>). The main difference between the tools for Cu and Ge crystals is that while for metals the rake angle of  $0-5^\circ$  is recommended (we used  $0^\circ$ ), for the processing of Ge a negative rake angle of  $25^\circ$  was used as recommended by the tool maker. Shank thicknesses of 6, 4, 3, and 1,7 mm were used. For the sake of analyses to check the technology, satellite flat samples were prepared in the same way in addition to the monochromators.

## 3 Crystal X-ray monochromators

Fig.1a) shows the most common X-ray monochromators and analysers prepared from single crystals of Ge and Si by standard technology.



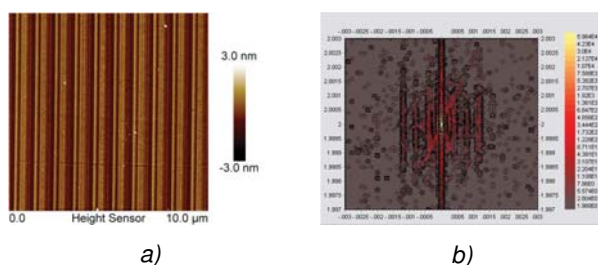
**Fig. 1** a) Several types of crystal X-ray monochromators including channel-cut monochromators, prepared by standard stochastic technology. b) beam compressing/expanding monochromator designed for Cu(200) symmetric-asymmetric diffractions.

Fig. 1b) shows a beam compressing/expanding channel-cut monochromator similar to Z-monochromator [2]. We proposed this design to machine the active in-channel surfaces by means of deterministic flycutting technique. Here, the tool can move up and down freely over the machined surface. Contrary to standard channel-cut monochromators, this kind of beam expanding/compressing monochromators is possible because the monochromators with unequal asymmetries can change beam cross sections according to total asymmetry factors. Several Cu and Ge based monochromators of this kind were prepared.

#### 4 Results and discussion

In addition to precise shape, the most important parameters for the surface quality of crystal X-ray optics are surface roughness and subsurface damage. A surface profiler was used to measure the height profiles, AFM to measure the 2D surface morphology and roughness, and micro Raman spectroscopy and the high-resolution X-ray diffractometry to measure the subsurface damage.

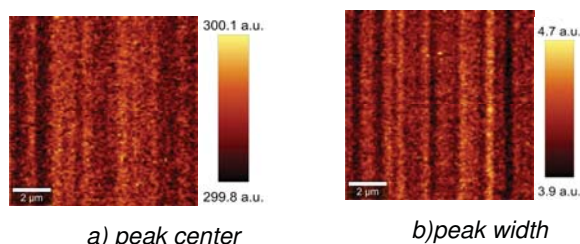
Figure 2a) shows an AFM image of a SPDT processed Ge(110) sample, Fig. 2b) its reciprocal space map with surface grating truncation rods corresponding to the surface texture visualized by AFM. Frequency of the stripes in real space in Fig. 2a) is inversely transformed into frequency of the grating truncation rods in reciprocal space in Fig. 2b). These rods represent the intensity scattered away from the direct X-ray beam and in this way they decrease the angular purity of the beam. These stripe inhomogeneities deteriorate to some extent monochromator properties, hence, some post-polishing lent from the standard stochastic technology must be used to remove them. A 15 min chemomechanical polishing with 60 nm SiO<sub>2</sub> slurry was shown to sufficiently suppress the stripes. However, additional artefacts from chemical reaction can appear [3].



**Fig. 2** a) AFM image of a SPDT processed Ge(110) sample, b) reciprocal space map with grating truncation rods.

Fig. 3 illustrates presence of subsurface damage at SPDT surfaces, in correspondence with stripes in Fig. 2a) and reciprocal space map in Fig. 2b). This indicates a positive correlation between the subsurface damage and surface roughness that is 1.14 nm rms in this sample. In principle, the sub-

surface damage is not inevitable to generate grating truncation rods in reciprocal space maps. The micro Raman spectroscopy is, on the other hand, sensitive to microstrains generated by subsurface damage.



**Fig. 3** Micro Raman spectroscopy of a SPDT processed Ge(110) sample. Image taken from the peak center position (a) and from the peak width values (b). Both characterize the subsurface damage.

As for the direct nanomachining inside the channel walls, it became clear that the tool shank of 1.7 mm thickness is too thin as its vibrations generate irregular stripes of variable depths.

#### 5 Conclusion

We applied the single point diamond technology to crystal X-ray optics, namely to channel-cut monochromators. Technologically, some postpolishing will be necessary to suppress the machining stripes and remnants of the subsurface damage. Other field of our activity are curved (spherical, logarithmic, cylindrical, parabolic) surfaces. Our technology is open also to non X-ray optics, e.g. to optical vortices. Collaborations in the field of ultra precise machining are welcomed.

#### 6 Acknowledgments

This work was done during implementation of the project *Research and Development Centre for Advanced X-ray Technologies*, ITMS code 26220220170, supported by the Research and Development Operational Programme funded by ERDF (0.5). Support of the Slovak Research and Development Agency, project No. APVV-0308-11, grant agency VEGA Bratislava, project No. 2/0004/15, and the COST Actions MP1203 and MP1207 is also acknowledged.

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