TOWARDS X-RAY CRYSTAL CHANNEL-CUT MONOCHROMATORS PREPARED BY NANO-MACHINING TECHNIQUE

Z. Zápražný[1], D. Korytár[1], M. Jergel[2], Y. Halahovets[2], C. Ferrari[3], C. Frigeri[3],
E. Dobročka[1], J. Keckes[5], J. Hagara[1,4]

[1]Institute of Electrical Engineering, Slovak Academy of Sciences, Dúbravská cesta 9,
841 04 Bratislava, Slovakia
[2]Institute of Physics, Slovak Academy of Sciences, Dúbravská cesta 9, 845 11 Bratislava, Slovakia
[3]CNR-IMEM Institute, Parco Area delle Scienze 37/A, 431 24 Parma, Italy
[4]Integra TDS s. r. o., Pod Párovcami 4757/25, 921 01 Pieš any, Slovakia
[5]Erich Schmid Institute of Materials Science, Austrian Academy of Sciences and Department of Materials
Physics, Montanuniversität Leoben, 8700 Leoben, Austria
zdenko.zaprazny@savba.sk

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Abstract

We study and develop methods for producing diffraction surfaces in X-ray crystal monochromators using advanced deterministic nano-machining technique. Applying nano-machining, it is possible to produce various surfaces (free-form) with high accuracy of the shape. This technique is well developed for producing surfaces of infrared or visible light optics, however, its implementation for hard X-ray optics, especially for channel-cut monochromators, is a very challenging task. Very small values of surface roughness and a good planarity are important because of very small incidence or emergence angles of hard X-ray beam in highly asymmetric channel-cut crystal monochromators designed for high resolution X-ray imaging or metrological applications. We have produced high-planarity diffraction surfaces with a planar deviation down to 3 nm over 50 µm. Surface roughness (RMS) far below the level of 3 nm was reached.

Introduction

Diffraction surfaces in X-ray crystal channel-cut monochromators working in high asymmetry mode require excellent parameters in terms of surface roughness, surface planarity and shape accuracy. The important task is also minimization of subsurface damage in order to maintain homogenous X-ray beam penetration and information depth in the whole diffraction area. It is difficult in standard finishing procedures using various modified acid solutions (HF, HNO_3, CH_3COOH) to produce highly planar surfaces for high resolution X-ray imaging or metrological applications without an "orange peel" morphology. In this work, we study and apply advanced finishing methods to X-ray crystal optics. In particular, we use advanced deterministic nano-machining technique [1] followed by optimal chemo-mechanical polishing. The nano-machining uses a single point diamond tool to remove material from the surface. There are two methods of nano-machining: single point diamond turning (SPDT) and fly cutting. Applying nano-machining, it is possible to produce not only flat but also curved or free-form surfaces with a very high accuracy. The nano-machining allows an accuracy 0.15 µm over 75 mm diameter and a surface roughness RMS 3 nm, according to manufacturer of the Nanotech 350 FG nano-machining centre (Moore Nanotechnology Systems, LLC). The technique of nano-machining is very well developed for producing surfaces of infrared or visible light optics [2]. However, its implementation for hard X-ray optics is a very challenging task. In a so called ductile mode, this technology is applicable even to brittle materials like germanium or silicon that are difficult to be machined, being only little invasive to crystal lattice beneath the surface. In our previous works [3, 4] several advanced analytical techniques have been used for evaluation of diffraction surfaces prepared by nano-machining. High resolution reciprocal space mapping using X-ray diffraction (HRXRD), atomic force microscopy (AFM) and surface profilometry have been used for evaluation of surface topology and surface roughness. High resolution transmission electron microscopy (HRTEM) and Raman spectroscopy have been used for observing the sub-surface regions. Surface roughness below 1 nm (RMS) with minimal damage of the subsurface crystal lattice was locally achieved after post-polishing process on open flat surfaces of monocrystalline Ge according to AFM measurements [3]. We were able to produce high-planarity surfaces with a maximum planar deviation of 3 nm over 50 µm. The local surface roughness below 1 nm was achieved directly after nano-machining process [4] using feed rates in the range from 2 to 0.125 mm/min. HRTEM measurement indicated very low damage of the subsurface region. In this work we continue with the analysis of the Ge sample prepared by fly cutting method shown in [4], on which we have applied five different feed rates ranging from 2 to 0.125 mm/min on separate areas. Using high resolution X-ray reciprocal space mapping and transmission electron microscopy we have confirmed that the nano-machining such as the single point diamond technology is a promising technique for a high-quality deterministic surface treatment to replace the less homogeneous stochastic chemical finishing methods traditionally used in the X-ray crystal optics. Our experience gained on planar open surfaces will be utilized for reproducible production of high quality diffraction surfaces, in particular in highly asymmetric channel-cut crystal monochromators [5]. The first results obtained on diffraction surfaces in the channel-cut monochromators prepared by nano-machining are shown in this article.

Experimental setup

The crucial parameter of high performance X-ray diffraction surface is the surface roughness and subsurface damage. The diamond tool produces periodical surface ripples,

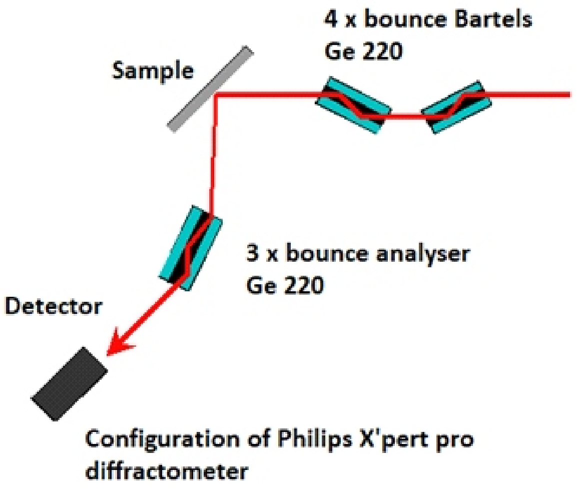

Figure 1. HRXRD setup: High-resolution Philips diffractometer with a 3 bounce analyser crystal.

which can cause unwanted extra diffractions or interference effects in the case of hard X-ray radiation. Our previous calculations showed that the periodicity of the ripples can vary from 670 to 43 nm depending on the feed rate from 2 to 0.125 mm/min [4]. High resolution X-ray diffraction measurements confirmed these results [3]. A scheme of these measurements used also in this work is shown in Figure 1.

Experimental results

A monocrystalline germanium sample with open flat surface of (220) orientation was prepared by fly cutting method. Five separate areas with different feed rates ranging from 2 to 0.125 mm/min were machined. A diamond tool tip with a rake angle of -12° brazed on the side of a 6 mm thick shank was used. The removal depth of finishing cut was 2 µm. We analysed the surfaces by reciprocal space mapping using a three-bounce Ge 220 analyser. In this configuration no effect of the crystal analyser is seen (analyser streaks). Diffuse scattering parallel to the sample surface is present in all samples and decreases with the feed rate (Figure 2). Streaks at 30° with respect to surface normal are observed in the samples at higher feed rates, which can be possibly associated with higher strain or dislocations in the lattice using higher values of feed rates. No periodic features due to the surface ripples visible in AFM [4] are visible.

To observe a very close subsurface region, we have performed several high-resolution TEM measurements. HRTEM image shows that the top surface is not perfectly flat as some valleys and crests of a few atomic layers depth/height with respect to an average surface occur (Figure 3). The perfect contact and sticking of the glue to the Ge sample excludes that such a roughness is due to the removal of the very top atomic Ge layers by the Ar ion bombardment during the sample preparation. It is worth noting that the HRTEM images have revealed absence of any dislocations in the subsurface region of the nano-machined Ge samples using the feed rate of 0.125 mm/min (Figure 3), which locally confirms the non-invasiveness of the nano-machining process.

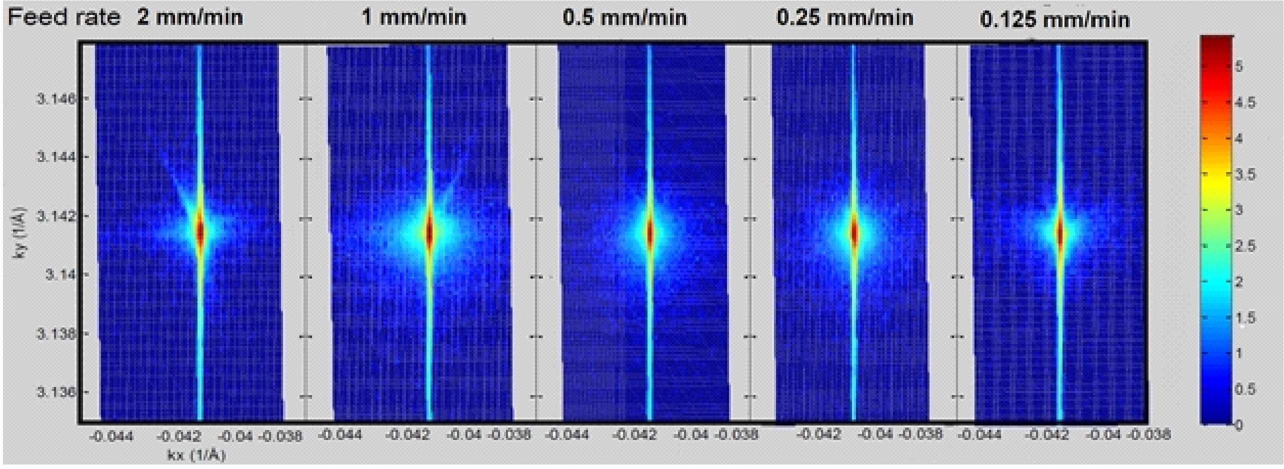

Figure 2. HRXRD measurements in reciprocal space show streaks at 30° with respect to surface normal in the samples at higher feed rate.

To obtain the best possible quality of the diffraction surfaces in the channel-cut monochromators, various machining parameters such as spindle rotation (rpm), feed rate (mm/min), number and depth (µm) of individual cuts were tested. Development of special tools allowing to finish inner active surfaces of channel-cut monochromators has been also a very important part of our research. For economic and safety reasons, the first test was performed on an "artificial" channel-cut monochromator (Figure 4). The "artificial" channel-cut monochromator is composed of three separate crystal blocks glued together to create conditions as in a real channel cut monochromator. It was first prepared with rather wide 6.5 mm wide channel to test the tools with a brazed diamond tip on the side of 3 mm and 6 mm thick shank. The tool with 6 mm thick shank showed better vibration resistance. However, it is less suitable for monolithic channel-cut monochromators typically manufactured with narrower channels (3 mm). These require a completely different tool to be developed.

As the samples are mounted on the spindle site in SPDT mode, the fly cutting mode has to be used for the channel-cut monochromators. In the case of fly cutting the shank tool is mounted on the spindle site and the sample is

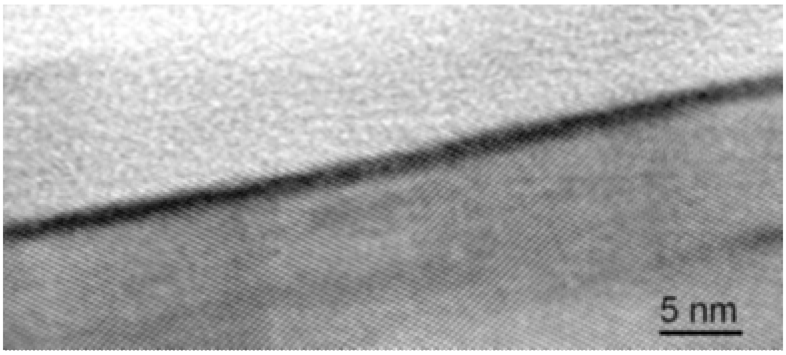

Figure 3. The Ge lattice terminates fully crystalline without any dislocations in the subsurface region

Figure 4. "Artificial" Ge channel-cut monochromator (assembled, glued together). Red arrow shows machined inner diffraction surface of the channel-cut monochromator glued to the metal block.

moving towards the tool. The machining parameters were tested in selected intervals as shown in Table 1, using a constant rotation rate of the spindle for both, roughing and finishing processes. The tool geometry as the radius of the diamond tip, the rake angle, etc. were used according to the tool manufacturer's recommendations.

The active surface in the channel-cut monochromator presented in Figure 4 was machined using a diamond tool with the rake angle of -25° brazed on the side of a 3 mm thick shank using a feed rate of 1 mm/min. The removal depth of finishing cut was 2 µm. The reciprocal space map (RSM) shows periodic vertical lines (truncation rods) with a spacing of 1.51 10^{-3} Å^{-1} (Figure 5a) suggesting presence of periodic surface ripples with a planar spacing of 416 nm. After a 90° rotation the map shows no truncation rods (Figure 5b). This is typical behaviour of surface gratings measured in coplanar geometry [6]. In fact, the ripples need not be set exactly perpendicular (Figure 5a) or parallel (Figure 5b) to the plane of incidence of the probing X-rays. However, such a slight misalignment would be below our resolution.

Discussion and conclusions

We have applied the fly cutting method of nano-machining to prepare X-ray diffraction surfaces with very high shape accuracy, low surface roughness and minimal subsurface damage. The open planar surfaces and channel-cut monochromators (CCM) were addressed. The diffraction surface in the channel-cut monochromator was machined with a special diamond tool with the rake angle of -25° brazed on the side of a 3 mm thick shank using a feed rate of 1 mm/min. The removal depth of finishing cut was 2 µm. High resolution reciprocal space map of the diffraction surface in a channel-cut monochromator showed periodic vertical lines (truncation rods) corresponding to a planar

Table 1. Selected machining parameters of the nano-machining process.

	Feed rate (mm/min)	Depth of cut (µm)	Rotation of the spindle (rpm)	Radius of the diamond tool (mm)	Working room temperature (°C)
Roughing	4-20	3-10	3000	1.485	23.2-23.5
Finishing	1-3	1-2	3000	1.485	23.2-23.5

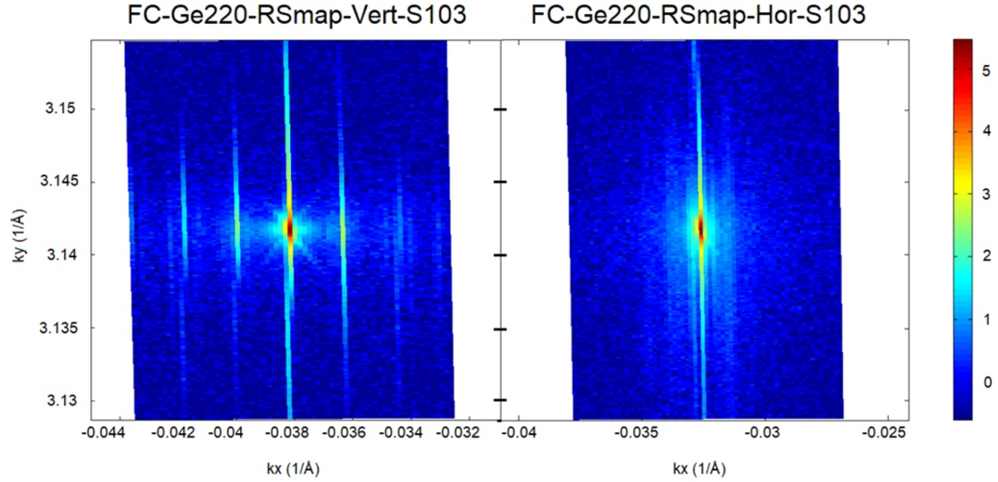

Figure 5. a) Reciprocal space map shows vertical lines (truncation rods) with a spacing 1.51 10^{-3} Å^{-1} corresponding to a planar spacing of 416 nm. b) RSM after 90° sample rotation.

spacing of 416 nm. We suppose that this effect is due to the deterministic character of nano-machining. We also extended the previous AFM, micro Raman spectroscopy and HRTEM analyses of germanium open flat surfaces with (220) orientation [4] by HRXRD and another HRTEM measurement. Five different feed rates ranging from 2 to 0.125 mm/min were used in separate surface areas. A diamond tool tip with a rake angle of -12° brazed on the side of a 6 mm thick shank was employed. The removal depth of finishing cut was 2 µm. Here, the surface ripples visible in AFM were not manifested in the high resolution reciprocal space maps in the form of truncation rods. Presumably, the ripples were not properly oriented with respect to the plane of incidence of the probing X-rays. It is also possible that the more vibration resistant tool with 6 mm thick shank produced "shallower" ripples comparing with the 3 mm thick shank used for the channel-cut monochromator. Further analyses of these effects will continue. Nevertheless it shows up that to further reduce surface roughness and suppress the ripples on nano-machining, development of a new tool for the most commonly used monolithic channel-cut monochromators with the channel width of about 3 mm is necessary. The repeated HRTEM confirmed excellent quality of the Ge crystal lattice beneath the surface on nano-machining. In particular, absence of dislocations in the subsurface region confirmed non-invasiveness of the nano-machining process.

References

1. G.M. Robinson, M.J. Jackson: A review of micro and nanomachining from a materials perspective, Journal *of Materials Processing Technology,* Volume **167**, Issues 2–3, 30 August 2005, Pages 316–337.
2. Robert E. Parks: Fabrication of infrared optics*, Optical Engineering* **33**(3), 685—691 (March 1994).
3. Z. Zápražný, D. Korytár, M. Jergel, P. Šiffalovič , E. Dobročka, P. Vagovič, C. Ferrari, P. Mikulík, M. Demydenko, M. Mikloška.: Calculations and surface quality measurements of high-asymmetry angle X-ray crystal monochromators for advanced X-ray imaging and metrological applications, *Optical Engn.* **54** (2015) 035101.
4. Z. Zápražný, D. Korytár, M. Jergel, P. Šiffalovič, Y. Halahovets, J. Keckes, I. Ma ko, C. Ferrari, P. Vagovič, M. Mikloška.: Nano-machining for advanced X-ray crystal optics*, AIP Conf. Proc.* **1764** (2016) 020005.
5. C. Ferrari, F. Germini, D. Korytár, P. Mikulík, L. Peverini: X-ray diffracted intensity for double-reflection channel-cut Ge monochromators at extremely asymmetric diffraction conditions*, J. Appl. Crystallogr.* **44**, 353–358 (2011).
6. P.Mikulík, M. Jergel, T. Baumbach, E. Majková, E. Pinčík, Š. Luby, L. Ortega, R. Tucoulou, P. Hudek, I. Kostič: Coplanar and Non-Coplanar X-Ray Reflectivity Characterization of Lateral W/Si Multilayer Gratings, *J. Phys. D* **34,** A188-A192 (2001).

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