FABRICATION OF CRYSTALS FOR CHANNELING OF PARTICLES IN ACCELERATOR

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Abstract
Channelling in bent crystals is technique with high potential to steer charged-particle beams for several applications in accelerators. Channeling and related techniques underwent significant progress in the last years. Distinctive features of performance increase was the availability of novel ideas other than new techniques to manufacture the crystal for channeling. We propose two methods to fabricate crystals through silicon micromachining techniques, i.e., isotropic or anisotropic silicon etchings. Characterization of the crystals accomplished for both methods to highlight that the crystals are free of lattice damage induced by the preparation. Crystals prepared by both kinds of methodologies were positively tested at the external line H8 of the SPS with 400 GeV protons for investigation on planar and axial channelings as well as on single and multiple volume reflection experiments by the H8-RD22 collaboration.

INTRODUCTION
Channeling consists of confinement between the atomic planes of charged particles traversing a crystal. If the crystal is bent, the particles can follow such bending. Deflection of high-energy positively charged particles by a bent crystal is a method, whose potential has been widely exploited, for beam steering in accelerator physics, e.g., for extraction [1], focusing [2], splitting [3], collimation [4], undulation [5]. We review the methods we developed at the Sensors and Semiconductors Laboratory in Ferrara about fabrication of crystals for channeling.

CRYSTAL FABRICATION
Crystal fabrication requires dicing of the sample starting from either a wafer or an ingot. This operation is normally accomplished by mechanical or laser-assisted cut of base material. Such operation alters the original quality of the crystal, leading to the formation of a superficial “dead layer” with a great number of defects and crystalline disorder, which does not act as an active layer for channeling [6]. Typically, preparation of suitably shaped crystals involves dicing of all crystal surfaces, both normal (entry face) and parallel to the beam direction. In order to restore the crystal quality, we studied and optimized two methods based on wet isotropic and anisotropic chemical etchings. Such methods are borrowed from micro-fabrication techniques of silicon revisited and adapted to the case of sample preparation for channeling.

Isotropic Etching
Isotropic etching of silicon is capable of removing the damaged layer at surface: etching is realized by a mixture of HNO₃, CH₃COOH and HF acids. The first one oxidize silicon to SiO₂, which is removed HF, while acetic acid acts as a solvent. Crystal surfaces have been analyzed by RBS-Channeling using 2MeV He⁺ to show that the damaged layer was actually removed [7]. A dedicated study carried out in an external line of IHEP with 70 GeV protons highlighted that chemically-treated crystals resulted in better performance as compared to equivalent untreated samples [8]. Samples produced by these methods were experimented in channeling [9] and volume reflection [9] modes.

However, the process of etching also resulted in significant roughness (Ra=140 nm) of the surface in spite of its crystalline quality. Indeed, etching was conceived for removal of the damaged layer at the entry face of the crystal and thereby a relatively rough morphology was not a problem. On the strength of recently proposed usage of a crystal in modern hadron colliders and, in particular, of the constraint on the roughness of the lateral faces, an innovative methodology to treat the crystal’s surface was developed.

Thus, chemical etching was reconsidered in order to decrease surface roughness. The keypoint of the methodology is an unusually large HNO₃ concentration to stimulate the oxidation and, simultaneously, the choice of properly short reaction timing as reported in Ref. [10]. The method allows the preparation of crystals with the right parameters to meet the requirements for channeling experiments in the new generation of hadron machines.

Anisotropic Etching
In contrast to previously used isotropic etchings of Si, there is class of chemical reactions whose erosion rate depends on the crystalline orientation. Thus, with proper choice of the components of the solution, anisotropic erosion would results in a high-precision cut of a crystal (see Fig. 1).

Si crystals have been prepared starting from a 4-inch (110) Si wafer with the wafer’s flat oriented perpendicular to <111> direction. A 100-nm layer of Si₃N₄ was deposited onto all faces of the wafer through low-pressure
chemical vapor deposition and patterned with standard photolithographic techniques [11] with the masking pattern aligned with the wafer’s flat.

Figure 1: Schematic view of fabrication of a silicon crystal via anisotropic etching. (a) sample after patterning with Si$_3$N$_4$ (dark regions) and prior to chemical attack; (b) the unmasked areas undergo etching along the $<110>$ direction while negligible erosion occurs along the $<111>$ direction. Proper timing allows one to make controlled indentations or complete cut of the sample.

The wafer was immersed in KOH solution (20% weight concentration) with the Si$_3$N$_4$ pattern as a masking layer [12], which resulted in erosion of uncovered regions of the wafer. For the experimental parameters of the solution we chose, the etch rate of (111) planes is negligible with respect to that of the (110) planes, thereby chemical erosion proceeds as depicted in Fig. 1b. The protecting layer of Si$_3$N$_4$ was finally removed from the lateral surfaces, leaving a wafer with regularly shaped even rectangular slots. Then, the wafer can be cut to achieve either a batch of independent strips (Fig. 2d) or a rigid frame interconnecting a series of regularly positioned strips (Fig. 2e). Strip crystals were employed to study channeling and volume reflection effects in a single crystal, while multi-crystals are useful to study multi-channeling or multi-volume reflection effects. Both this type of crystals were positively tested at the external line H8 of the SPS with 400 GeV protons for investigation on axial channeling and on single and multiple volume reflection experiments by the H8-RD22 collaboration.

Morphological investigation of the roughness of the crystal surface was done by atomic-force microscopy (AFM). Analysis was carried out over one of the (110) surfaces of the samples, i.e., to the lateral surface of the crystal, which are the planes interacting first with halo-beam particles. Clear evidence of ultra-flat surface with a roughness down to the monolayer level was achieved over a relatively wide scan as comparatively reported in Tab. I

<table>
<thead>
<tr>
<th>Methodology</th>
<th>$R_a$ (nm)</th>
</tr>
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<tbody>
<tr>
<td>Isotropic etching (first generation)</td>
<td>135 (Ref. [7])</td>
</tr>
<tr>
<td>Isotropic etching (second generation)</td>
<td>23 (Ref. [9])</td>
</tr>
<tr>
<td>Anisotropic etching</td>
<td>0.25</td>
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Table 1: Comparison between standard roughness, $R_a$, obtained by different etchings. $R_a$ was measured by AFM over 10x10 µm$^2$ large area.

Standard roughness featured $R_a=0.25$ nm, i.e., it was decreased by nearly two orders of magnitude with respect to the fabrication via isotropic etching.

Figure 2: Fabrication of crystals for channeling (not to a scale) (a) deposition of a uniform 100-nm thick Si$_3$N$_4$ layer, (b) patterning of Si$_3$N$_4$, (c) anisotropic KOH etching and mechanical dicing along either the dashed line to release a series of independent strip-like crystals or the solid line to manufacture a multi-strip crystal with a frame, (d, e) final removal of the Si$_3$N$_4$ film. Masking by KOH-resistant Si$_3$N$_4$ thin film patterned onto the surfaces of the Si crystal allows fabrication of rather complex geometries.

The crystal quality in proximity of cut surfaces was analyzed by High Resolution TEM (see Fig. 3), which showed an ordered arrangement of the atomic columns, which was preserved up to the crystal surface. Similar observations over different area of the sample confirmed previous determinations.
CRYSTALS FOR BEAM COLLIMATION

A pioneering experiment at Fermilab showed that the usage of a crystal interacting with a 900 GeV proton beam has halved the radiation background in the experimental areas as a result of halo cleaning [13]. Indeed, it has been proposed the usage of short crystals to aid the collimation of the intense beam halo in the large hadron collider (LHC) [14]. Here, the particles in the halo drift outwards at the rate of \( \sim 2 \) nm/turn. Since the tune is not integer, the particles will hit the crystal every \( \sim 10–20 \) turns and thereby the first impact parameter of the particles onto the crystal will be in the range of \( \sim 100 \) nm. [6]

An intense study is being carried out to propose methods for a smart solution to halo problems in the LHC by channeling-based techniques [15]. Both classical channeling or multiple volume reflections are traceable techniques. In any case, great attention is to be made to the lateral faces of the crystal exposed aside the beam. We believe that the method of anisotropic etching yields a crystal with appropriate surface parameters for this purpose.

REFERENCES


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Figure 3: High-resolution TEM characterization of the entry face of the crystal. Analysis highlights ordered and atomically sharp termination of the crystal surface. The amorphous material above the crystal is the embedding epoxy used in the preparation of the cross-section sample.

Figure 4: Schematic (not to scale) of the application of bent crystal for beam steering. The impact parameter \( b \) and the impinging angle of beam \( b' \) are shown. The surface roughness \( Ra \) of the lateral face of the crystal (parallel to beam direction) is required to be lower than the impact parameter \( b \) (100 nm for beam halo of LHC) for beam steering using the channelling effect.

Previous investigations highlighted the critical role of the parameters of the crystal surface for application under channeling mode [14]. Accordingly, grazing impact of the particles onto the crystal surface does demand a crystal with a roughness lower than 100 nm on the lateral faces of the crystal as schematically depicted in Fig. 4.