Study on Nanoscratching of C-plane Sapphire Wafer

Wangpiao Lin\textsuperscript{1, a}, Jun Shimizu\textsuperscript{2, b}, Libo Zhou\textsuperscript{2, c}, Teppei Onuki\textsuperscript{2, d}, Hirotaka Ojima\textsuperscript{2, e} and Takeyuki Yamamoto\textsuperscript{2, f}

\textsuperscript{1}Graduate School of Science and Engineering, Ibaraki University, 4-12-1 Nakanarusawa-cho, Hitachi-shi, Ibaraki 316-8511, Japan
\textsuperscript{2}Department of Intelligent Systems Engineering, Ibaraki University 4-12-1 Nakanarusawa-cho, Hitachi-shi, Ibaraki 316-8511, Japan
\textsuperscript{a}15nd206r@vc.ibaraki.ac.jp, \textsuperscript{b}jun.shimizu.nlab@vc.ibaraki.ac.jp, \textsuperscript{c}libo.zhou.1618@vc.ibaraki.ac.jp, \textsuperscript{d}teppei.onuki.nlab@vc.ibaraki.ac.jp, \textsuperscript{e}hirotaka.ojima.gen365@vc.ibaraki.ac.jp, \textsuperscript{f}takeyuki.yamamoto.955@vc.ibaraki.ac.jp.

Keywords: sapphire, C-plane surface, nanoscratching, anisotropy, force, depth, cutting chip.

Abstract. Single crystal sapphire wafer has been widely applied in many fields due to its special crystal structure and material properties. Thus, it is necessary to continue thorough research into its removal and deformation mechanisms from the viewpoint of crystal orientation and property. In this report, a scanning probe microscope was systematically used as an instrument for a series of nanoscratching experiments and measurements to investigate the influence of anisotropy of intact C-plane (0001) surface sapphire wafer on its ductile mode scratching behavior. At first, the occurrence of ductile regime scratching was confirmed through the scanning electron microscope observations of generated cutting chips which adhere to the sharply pointed triangular diamond probe tip used as the scratching tool. The effects of scratch direction on the depth of scratch groove and scratch force were evaluated, and the existence of anisotropy in the scratch force was clarified. Through an evaluation, it was clarified that the decrease in the scratch force concerned with the relation between the scratch and probe tip edge directions, and when the projected probe tip edge direction met [1\bar{1}00] or similar directions which possessed the lowest shear strength due to basal twinning, the scratch force became relatively low.

Introduction

Single crystal sapphire wafers composed of $\alpha$-Al$_2$O$_3$ molecules with the structure shown in Fig. 1 possess many superior physical, chemical and optical properties, such as high hardness, excellent thermal stability, which make it resistant to high temperature, thermal shock, water and sand erosion, and scratching. Hence, it is widely applied in jewelry industry, engineering and optics [1].

C-plane sapphire substrates are also widely used to grow III-V and II-VI semiconductor compounds such as GaN for blue LED and laser diodes. It is also used as the substrates for epitaxial growth of some kinds of oxide and metal films after finished by abrasive machining processes [2,3]. Particularly, growth of semiconductor compounds requires extremely smooth surface and high surface integrity of C-plane sapphire substrates. Material properties of sapphire exhibit anisotropy which would affect the deformation mechanisms and fracture behaviors at various sapphire orientations during abrasive machining process. Under such a background, some researchers have tried to clarify the behaviors in the ductile-brittle transition of C-plane sapphire substrates and some insights have already been obtained [4-7]. However, there is little researches that shed some lights on the relationship between the crystal anisotropy and ductile machining behavior of C-plane sapphire substrates, even though it is remarkably important for its finished surface integrity.
In this report, a series of nanoscratching experiments using a sharply pointed triangular monocrystalline diamond probe tip as the tool were conducted on an intact C-plane (0001) surface sapphire wafer at low normal loads for various crystal orientations on a scanning probe microscope (SPM) system to investigate its nanomachining mechanism. Particularly, the effect of scratch direction on the scratching depth and force were examined.

**Experimental Apparatus and Method**

Nanoscratching experiments were conducted with a SPM that had an environmental control function (SPA-300HV made by Seiko Instruments Inc.), as shown in Fig. 2. Its XY and Z axis positioning resolutions are 0.3 nm and several nanometers, respectively. Also, Fig. 2 shows the schematic of nanoscratching which the cantilever deflection is kept constant in a nanoscratching trial for keeping a constant normal load by controlling the piezo scanner. Its frictional force microscope (FFM) mode was used for the experiments, since the determination of the scratch or tangential force is remarkably important. Two inches double-sided polished intact sapphire C-plane (0001) surface wafers with A-plane (1120) cross section was utilized as the workpiece.

The schematic drawing of nanoscratching directions on C-plane sapphire is shown in Fig. 3. 10 μm x 10 μm scratched by applying 32 back and forth motions with 156 nm pitch intervals all along with the crystal orientations shown in Fig. 3 (red points). 7 different orientations were tested here.

The experimental conditions are listed in Table 1. After the experiments, the surface topography of sapphire sample was obtained directly by AFM and the diamond tip was observed by SEM (JSM-6330F made by JEOL), respectively.

![Fig. 1 Structure of monocrystalline sapphire](image1)

![Fig. 2 Appearance of SPM and schematic of nanoscratching using FFM mode](image2)
Table 1 Experimental conditions

<table>
<thead>
<tr>
<th>Instrument</th>
<th>SPM(SPA-300HV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Work piece</td>
<td>Sapphire (C-plane)</td>
</tr>
<tr>
<td>Diamond cantilever tip</td>
<td>Trigonal pyramid (Tip radius = 100 nm)</td>
</tr>
<tr>
<td>Cantilever stiffness (N/m)</td>
<td>200</td>
</tr>
<tr>
<td>Normal load (μN)</td>
<td>10, 20, 30</td>
</tr>
<tr>
<td>Scratch direction</td>
<td>[1010],[1120],[0110],[1210],[1100],[2110],[1010]</td>
</tr>
<tr>
<td>Scratch speed (μm/s)</td>
<td>16</td>
</tr>
<tr>
<td>Scanning area (μm²)</td>
<td>15×15 (128 back and forth motions)</td>
</tr>
<tr>
<td>Scratch area (μm²)</td>
<td>10×10 (32 back and forth motions)</td>
</tr>
<tr>
<td>Feed pitch in scratching (nm)</td>
<td>156</td>
</tr>
</tbody>
</table>

Results and Discussion

Fig. 4 shows an example of the atomic force microscope (AFM) or 3-D image of scratched sapphire wafer surface morphology under the conditions of 10μN in normal load, [1120] in scratching direction and 16 μm/s in scratch speed. The right figure shows the cross-section at the specific surface along the datum line shown in the left image after the nanoscratching. According to the cross-sectional image, 64 scratch paths evenly distributed in 10 μm × 10 μm scratching area with the interval of 156 nm. This might indicate a proof of ductile mode process. The average depth of scratch grooves was calculated from the cross-section with ignoring unusual scratch path. For example, the depth of the central groove is significantly deeper than that of others. This was due to the effect of the pre-scan which was done just before all the nanoscratching trials.
Table 2  The slip/twinning systems and shear strengths of C-plane sapphire crystal [8].

<table>
<thead>
<tr>
<th>Slip/twinning systems</th>
<th>Description</th>
<th>Critical shear stress $\tau_{c}$ (GPa)</th>
<th>Relative critical shear stress</th>
</tr>
</thead>
<tbody>
<tr>
<td>[10\̅10] (0001)</td>
<td>Basal twinning</td>
<td>$2.7 &lt; \tau_{c} &lt; 5.0$</td>
<td>1</td>
</tr>
<tr>
<td>[01\̅10] (0001)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>[\̅1\̅00] (0001)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>[\̅10\̅10] (0001)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>[1\̅1\̅20] (0001)</td>
<td>Basal slip</td>
<td>$\tau_{c} &gt; 4.0$</td>
<td>103</td>
</tr>
<tr>
<td>[1\̅2\̅10] (0001)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>[2\̅1\̅10] (0001)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Fig. 5 SEM images of cutting chip after nanoscratching at normal loads of 10 μN, 20 μN and 30 μN, and crystal orientations of [01\̅10] and [1\̅2\̅10] in C-plane surface sapphire, respectively.

In order to illustrate the deformation mechanism of sapphire when nanoscratching, the available slip/twinning systems and shear strengths of C-plane sapphire crystal were listed in Table 2 [8]. From this table, it can be estimated that such differences in the shear strengths due to the differences in the crystal orientation influences upon the nanoscratching behavior.

To reveal the materials removal behaviors of C-plane sapphire (0001) surface during nanoscratching with different normal loads and orientations, SEM images of the cutting chips which adhere onto the diamond probe tip after scratching are shown in Fig. 5. The following mechanisms of chip formation should be induced: continuous chip formation, lamellar chip formation, segmented chip formation and discontinuous chip formation depend on the workpiece material properties and the cutting conditions [9].
The main chip removal mechanism in the scratching of [0\bar{1}0] and [\bar{1}2\bar{1}0] orientations were based on the continuous or lamellar cutting chips formations, as shown in Fig. 5. Similar results were also obtained for other orientations. Such results show that there is no direct influence of crystal orientations on the fundamental chip formation mechanism when the normal load was lower than 30 \( \mu \)N. Therefore, the ductile regime materials removal which was affected by slip/twinning system would occur at the low normal load such as less than 30 \( \mu \)N in the nanoscratching of monocrystalline sapphire. Meanwhile, there was a difference in the cutting chip widths between [0\bar{1}0] and [\bar{1}2\bar{1}0] orientations at the normal load of 30 \( \mu \)N and it indicated a kind of anisotropy.

![Scratch force and depth of scratch groove graphs](image)

**Fig.6** The relationship between crystal orientations and scratch force (a), crystal orientations and the depth of scratch groove (b), respectively.

![Schematic of nanoscratching model](image)

**Fig.7** Schematic of nanoscratching model when scratch direction is [\bar{1}2\bar{1}0].

Figs. 6(a) and (b) show the relationship between crystal orientations and scratch force and depth of scratch groove, respectively.

According to Table 2, the basal slip/basal twinning system in various crystal orientations, basal twinning shows lower relative critical shear stress (RCSS). However, Fig. 6(a) demonstrates a strange phenomenon that the higher scratch forces were obtained in [0\bar{1}0], [\bar{1}2\bar{1}0], [\bar{1}1\bar{0}0], and [\bar{1}0\bar{1}0] orientations at the normal loads of 20 \( \mu \)N and 30 \( \mu \)N, although such orientations might show lower scratch forces based on the basal twinning. It would be affected by the pyramidal shape of the probe tip, as shown in Fig.7. When the scratch direction was [\bar{1}2\bar{1}0], the projected direction of one of the probe tip edges became parallel to [\bar{1}0\bar{1}0] orientation and corresponded to the basal twinning orientation. In such a case, a kind of stress concentration would occur around the probe tip edge and higher shear stress field would be generated. On the contrary, the anisotropy was not clearly
displayed at lower normal load like 10 μN, because a kind of tool edge effect mentioned above did not occur due to the microscopic roundness of the probe tip apex.

Fig. 6(b) indicates that the depth becomes greater with increasing the normal load regardless of the scratch directions. However, it is difficult to catch the trend about the anisotropy. One of the reason would be the adhered cutting chips sometimes had some troubles for the accurate determination of the groove depths. Therefore, solving such an issue can be a future task.

Summary

In order to clarify the nanomachining properties, particularly the anisotropy of the intact C-plane (0001) surface sapphire wafer, nanoscratching tests were conducted under a few low normal loads and several scratch directions. Conclusions obtained are summarized as follows:

(1) Continuous or lamellar cutting chips can be produced at C-plane surface under the normal loads less than 30 μN regardless of the scratch directions. It proves the occurrence of ductile regime machining during nanoscratching.

(2) The effect of anisotropy has also been examined through the nanoscratching experiments, and the results showed that a higher shear stress field given by the tool tip edge influenced upon the anisotropy in the scratch force.

Acknowledgement

This work was partially supported by JSPS KAKENHI Grant Number 15H02213.

References