# Comparative investigation of subsurface damages induced on sapphire with different machining methods using micro Raman spectroscopy

Teppei Onuki [a*], Ke Wu, Kazuki Kamoshida, Piao Lin, Nao Sugano, Hirotaka Ojima, Jun Shimizu and Libo Zhou
1

[1]Faculty of Mechanical systems Engineering, Ibaraki University,

4-12-1 Nakanarusawa, Hitachi, Ibaraki, 316-8511 Japan

[a]teppei.onuki.nlab@vc.ibaraki.ac.jp

**Keywords:** sapphire; surface integrity; grinding; laser ablation; micro Raman spectroscopy

**Abstract.** The objective of this research is to propose and develop an evaluation procedure by use of Raman spectral features to distinguish the mechanical, thermal and chemical effects on the surface integrity of sapphire wafer generated by different machining process. In this paper, several types of process; grinding (brittle-mode and ductile-mode), chemo mechanical abrasive machining, ion milling and laser ablation, have been put into investigation. The obtained results have been discussed to understand the measurement properties of material damages by various machining method, and to understand the dominant factors of damage induction.

## Introduction

Surface integrity is an important index to evaluate process availability for surface finishing of single crystal materials sapphire, as it significantly affects productivity and quality in subsequent process and end-products as epitaxial wafer, durable parts in optics or sensors. 3D micro Raman microscopy is capable of sense nondestructively of atomic-scaled latent defects which is normally undetectable by conventional visual inspection methods, via variations of the eigen modes of lattice vibrations by lattice strain around the flaws. Since these beneficial characteristics, we expect the application of 3D micro Raman microscopy as the inspection instruments of the surface integrity of the sapphire products. The reliability of the measured result in the inspection is significant for the purpose of the practical use. Then, it is a meaningful work to secure the reliability that comparative analysis of the results for comprehension of detection properties of various state of machining damages with different quality and quantity of the damages which are induced by a variety of the process methods and the process conditions.

Hitherto, the authors have been studied on the detection properties of nanoscale the material failure in subsurface of sapphire induced by different machining methods (grinding and laser processing) with various conditions by 3D micro Raman spectroscopy [1][2]. In fact, different changes in the spectral features and their spatial distributions are found in comparison among the material failure by them, moreover, involving the previous works, e.g. on an impression of micro indentation hardness testing, polycrystalline material as ceramics and particles, due to qualitatively and quantitatively different substance of the damages by different damaging mechanisms.

The objective of this research is to propose and develop an evaluation procedure of machining damages by use of Raman spectral features (ex. peak position, peak width and baseline) to distinguish the mechanical (twining, slip or fragmentation as plastic deformation), thermal (amorphization and polymorphism by phase transition) and chemical effects (diffused impurity defects as contamination) on the integrity of sapphire wafer surface generated by different machining process. In this paper, several types of process; grinding (brittle-mode and ductile-mode), chemo mechanical abrasive machining, ion milling and laser ablation (thermal-processing nonthermal-processing), have been put into investigation. The obtained results have been discussed to understand the dominant factors of inducing lattice degradations on c-plane surface of sapphire.

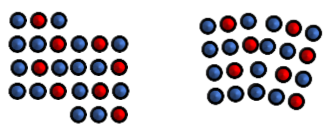
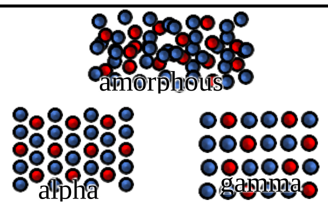
## Method and procedure

### Sample preparation method

Sapphire is a hard-to-machine material in mechanical machining due to its remarkable hardness and the toughness, and in laser machining due to high melting point and weak photon-material interaction by its wider energy bandgap of the electron system. Control of damage is difficult due to the specific energies of material removal as high as the one of damaging in the machining. Moreover, sapphire doesn't have obvious cleavage plane, and variable properties in material yielding by the temperature make the process mechanisms complicate. Therefore, detailed mechanisms of the material failure have still under consideration in both grinding and laser processing. Kinds of "soft surface processing" as chemo mechanical abrasive process (i.e. polishing (CMP) and chemo mechanical grinding (CMG)), or ion milling are used for finishing surfaces to remove the roughness and damaged layer. These soft processes also left the damaged layer in the subsurface when improper process conditions are unexpectedly used. In grinding process, the types of the process modes are categorized as brittle mode (BMG), ductile mode (DMG), and chemo mechanical mode (CMG) by different processing mechanisms, which depends on the grinding conditions like grains and depth of cut. In laser machining, the types of the process modes are categorized as nonthermal laser ablation (NTL), thermally melting/vaporization (TLP), and material separation as dicing/slicing by material modification or cleavage induced by controlled laser irradiations (LS). In ion milling, the types of the process modes are categorized as ion bombardment (IB), ion implantation (II), and reactive ion etching (RIE), which depends on the dose, acceleration of the ion beam and the ion species.

Generally, the material damage in machining are caused by excessed stress, heat and chemism induced in the machining. These damaging factors induce crack, plastic deformation, phase transition, and chemical change on the surface as shown in Table.1. In the discrimination of the damages in atomic sale substances as the causal relationship, crevices and internal fragmentations, plastic deformations like twining and slip of dislocation are majorly caused by the stress, phase transitions like amorphization, polymorphism are caused by the heat, and and

Table 1 discrimination of damaging factor and material failure by machining

| Damaging factor | Force | | chemism |
|---|---|---|---|
| | Heat | | |
| Material Failure | plastic deformation | phase transition | chemical change |
| |  |  |  |
| | twinging, slip, fracture | crystal polymorphism | defects, hydration, |

air void, impurity, valence modification by peroxidation or hydration are caused by the chemical effect, respectively. They decrease the regularity of atomic arrangement in crystal lattice around them, and the lattice degradations behave as sources of elastic strain field. These emerge in the Raman spectrum as broadening and shifting of the Raman peaks and variations of the baseline.

At the outset of this study, surfaces of c-plane sapphire wafers with grinding as BMG and laser machining as NTL were investigated. Grinding was conducted with a diamond wheel (SD500K50V) using a rotary grinding machine (UPG-150, in Ibaraki University). Detail descriptions of the process conditions of the BMG had published [3][4]. Laser machining was conducted using Nd:YAG laser pulsed with active mode locking (100MHz) and enhanced Q-switching (1kHz) as 100 pico sec-pulse duration, and focused with an objective lens (NA 0.45). Stage positioning was used for scanning the focus spot on the surface or inside of the material as the planar machining [5].

**Measurement method**

The causality of the study is schematically represented as in Fig.1. In the Raman spectra, the positions of Raman peaks are determined by the crystal type, and the widths of the peaks are used as the index of the irregularity of the crystal lattice (i.e. plastic strain). The shifts of the wavenumber of the peaks are occurred by the elastic strain due to the anharmonic terms of the chemical bondings. Hence the peak-shift to higher wavenumbers corresponds compressive strain, and the opposite shift corresponds tensile strain, respectively. The phenomena of the peak-shift are usually accompanied with the peak-broadening because the lattice degradation is the source of the elastic residual strain. The peak-shift without changing the width also occurs in the region of intact crystal covered with the elastic strain field. If fluorescent defects are generated in the material, they appear in the Raman spectra as increase of the baseline.

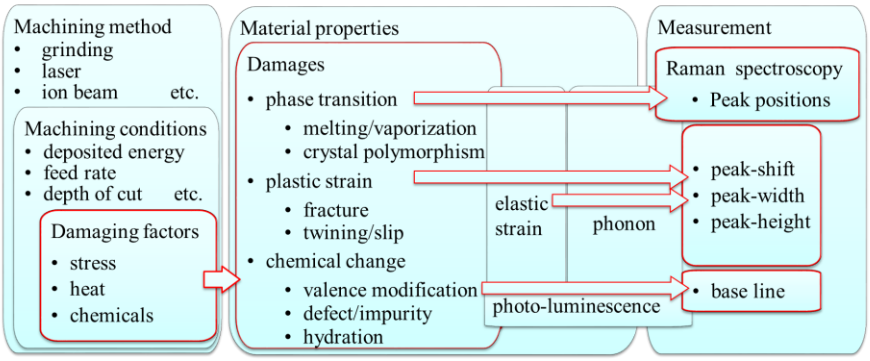



Fig.1 relationship among machining, damaging and measurement of Raman spectroscopy.

Several instruments of commercial 3D micro Raman spectrometers were used in this study (NanoPhoton, Thermofisher science etc., for scientific instrumentation) despite of the comparative investigations. However, they have similar measurement properties and the differences deriving the instruments were ignorable in the evaluations of the measurement data. The measurement conditions are shown in Table 2. The spatial resolution was about 1 μm, and the spectral resolution was about 2 $cm^{-1}$. Peak fitting with Lorentzian function was used for quantifying the peak features (peak-position, peak-width, peak-height and base line) with 0.001 $cm^{-1}$ order of the precision, for trying to investigate the damaging mechanisms in addition to quantify the material failure. Two-dimensional or three-dimensional measurements and the visual representation of the peak features by cultivative measurements have advantage of

Table 2 measurement condition in micro Raman spectroscopy

| description | value |
|---|---|
| Laser power | 10 mW |
| Wavelength of laser | 532 nm |
| NA of objective lens | 0.8 – 0.9 |
| Pinhole diameter at incident opening of the spectrometer | 20–25 μm |
| Exposure time | 30 sec |

enhanced reliability of the evaluating results. The depth-imaging were conducted by measurement with scanning the sample stage vertically in the areas with 20 μm in the depth and 40 μm in the length, involving both damaged region and nondamaged region.

## Result and discussion

Here, we show two examples of the depth-profile measurement by Raman spectroscopy. Fig. 2 (a) shows the microscopic image of the surface of BMG at depth-profile measurement by Raman spectroscopy. Spectroscopic data was obtained along the dashed line. The surface roughness Ra was 180 nm, and the depth of the dimples is 3μm, typically. (b) and (c) are the depth-profile of the peak-width and the peak-shift of the phonon mode at 417cm$^{-1}$, respectively. The pixel size was 0.2 μm in both vertical and horizontal direction. The dashed line indicates the surface level. Broaden peak-width was found at vicinity of the surface within 3μm from the surface. The distribution is concentrated on edges of dimples due to existing dense lattice degradation like fractures by stress or amorphization by heat. In middle region for 5 μm and 17 μm in the depth, sparse failures as twining on a prismatic plane was found. The vertical failures are not extended to 20 μm of the depth since the peak-widths at bottom end were as narrow as ones of intact bulk sapphire (2.2 cm$^{-1}$). The peak-shift was accompanied with broaden peak-width at the top surface, for both higher (compressive) and lower (tensile) wavenumber. Strong strain (stress) was concentrated on the fractures, and the strain field was extended vertically to the neutral plane at 7 μm-depth. The values of maximum residual strain (stress) was estimated as 1.4 × 10$^{-3}$ (500 MPa) when the calibration coefficient (-500 MPa/cm$^{-1}$) was used [5]. Weak strain was uniformly distributed in the region deeper than the neutral plane due to compensation of internal force in the material.

Fig.3 (a) shows the microscopic image of the surface of NTL at the depth-profile measurement along the dashed line involving both machined part (right side) and unmachined part (left side). Step-like surface geometry was formed at the

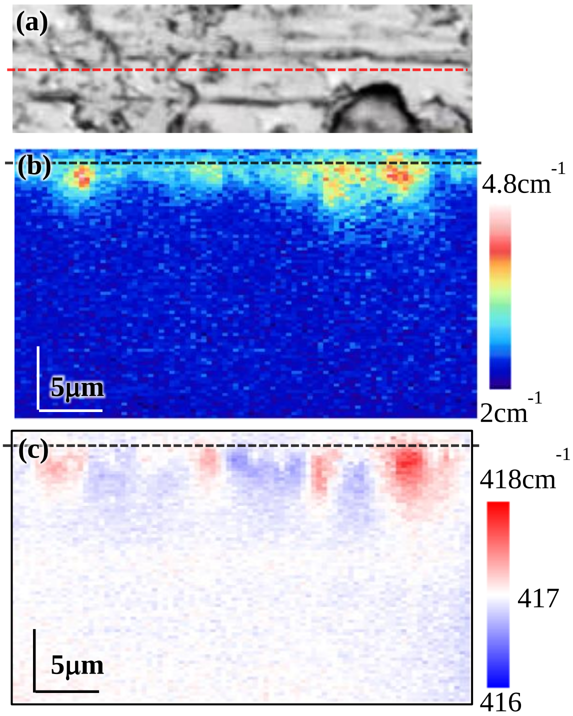



Fig.2 surface image of the surface of BMG (a), and depth profile of the peak-width (b) and peak-shift (c) at 417 cm$^{-1}$ of the phonon mode .

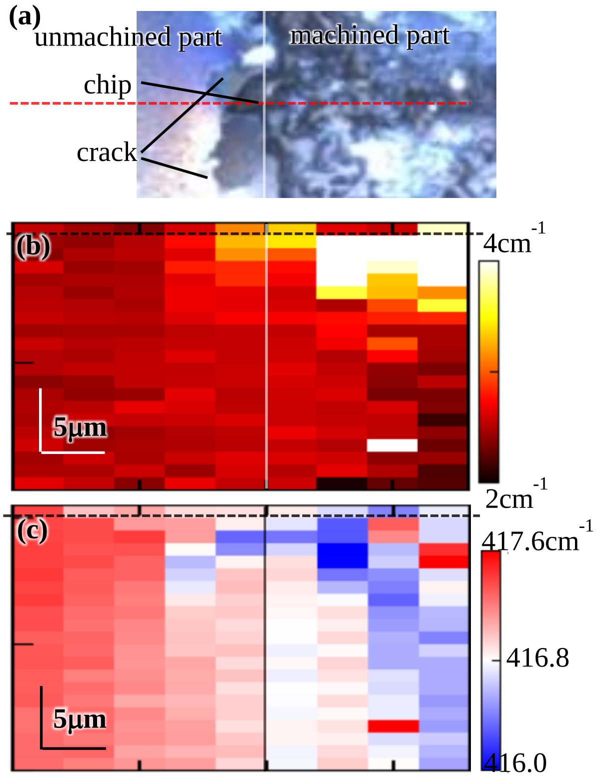



Fig.3 surface image of the surface of NTL (a), and depth profile of the peak-width (b) and peak-shift (c) at 417 cm$^{-1}$ of the phonon mode .

border, and the step-depth was 3 - 4.5 μm. The surface was fractured and the border was chipped, which were characteristics of nonthermal material removal in the laser ablation process. Lateral cracks were found in unmachined part at near the border. (b) and (c) are the depth-profile of the peak-width and the peak-shift of the phonon mode at 417cm$^{-1}$, respectively. The pixel size was 1 μm in vertical direction and 4 μm in horizontal direction. Broaden peak-width was concentrated at the fractured surface in machined part (Max. 6 cm$^{-1}$) and at the chipped surface in unmachined part (Max. 3.5 cm$^{-1}$). The distribution of the peak-shift was completely different from the one of the peak-width which is analogue to the lattice degradation. The strain field was distributed in the machined part and much widely extending for unmachined region. Tensile strain was induced in a region under the machined surface for more than 20 μm-below, due to the effect of thermal volume expansion by heat from deposited laser. The chipped part also had tensile strain. Strong compressive strain was induced in the left side of unmachined part with more than 10 μm apart from the border of the machined part, due to the reaction force of residual tensile strain. The elastic strain was neutralized at the border of machined area.

From these results, we confirm the usability of the depth measurement by 3D micro Raman spectroscopy as the inspection instruments of surface quality on various machining method. The depth-images are distinguishable in the material failure between grinding and laser machining, by different characteristics in the distributions of plastic strain (peak-width) and elastic strain (peak-shift). These data are also beneficial for the investigations of damaging mechanisms as a part of the machining mechanisms.

## Summary

We developed the method of inspections of the surface quality on sapphire by means of depth profile measurements of 3D micro Raman spectroscopy. This method can detect the lattice degradations and residual strain distribution with micron order of the spatial resolution. The difference of the distribution of the material failure by grinding and laser ablation can be recognized by this method.

Acknowledgement: This paper financially supported by Grant-in-Aid for Scientific Research (A)15H02213 and 16K06000 (JSPS) Japan.

## References

[1] T. Onuki et.al., Raman analysis of machining qualities on ground surfaces of sapphire wafers, Proc. of ISAAT2017, (2017), pp.1122-1126.

[2] T. Wermelinger et.al., 3-D Raman spectroscopy measurements of the symmetry of residual stress fields in plastically deformed sapphire crystals, Acta Materialia **55** (2007) pp.4657-4665.

[3] K. Wu et.al., Study on the potential of chemo-mechanical-grinding (CMG) process of sapphire wafer, International Journal of Advanced Manufacturing Technology, **91** (2017) pp.1539-1546

[4] K. Wu et.al., Study on the finishing capability and abrasives-sapphire interaction in dry chemo-mechanical-grinding (CMG) process, Precision engineering **52** (2018) pp.451-457

[5] T. Onuki et.al., Cross-sectional measurements of laser damage in sapphire using confocal micro Raman spectrometer, Proc. of ICPE2018, (2018).