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Review:

Characteristics and Mechanism of Catalyst-Referred Etching Method: Application to 4H-SiC

Pho Van Bui, Yasuhisa Sano, Yoshitada Morikawa, and Kazuto Yamauchi[†]

Department of Precision Science and Technology, Graduate School of Engineering, Osaka University
2-1 Yamada-Oka, Suita, Osaka 565-00817, Japan
[†]Corresponding author, E-mail: yamauchi@prec.eng.osaka-u.ac.jp

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A novel abrasive-free planarization method named catalyst-referred etching (CARE) was developed. A polishing pad is coated with a catalytic material to promote chemical etching of the work substrate. During processing, the topmost areas of the work substrate, which are in contact with the catalyst surface, are selectively etched off. Atomically highly ordered surfaces are obtained for many types of work substrates. In this paper, the removal characteristics and mechanism of CARE for single crystalline 4H-SiC are reviewed.

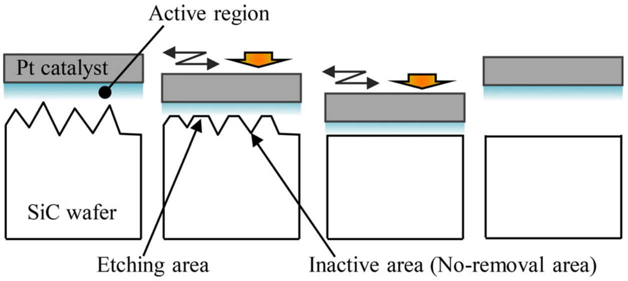

Fig. 1. Schematic outline of CARE concept.

Keywords: catalyst-referred etching, chemical planarization, SiC, interface reaction, first-principles calculations

1. Introduction

Silicon carbide (SiC) is one of the most attractive substrate materials for next-generation power devices because of its excellent physical properties such as a wide band gap, high breakdown voltage, highly saturated electron velocity, and high thermal conductivity. However, it is very difficult to flatten and smoothen the SiC surface because of its mechanical hardness and chemical inertness [1–3]. To achieve theoretically expected properties of SiC power devices, an atomically well-ordered surface is essential.

Chemical mechanical polishing (CMP) is widely used as a final polishing method for SiC; abrasives softer than the work material are generally used. Colloidal silica abrasives and a soft pad made from urethane foam are used for the planarization of SiC substrates. Chemicals in the slurry enhance the polishing rate by supporting the mechanochemical reaction between the abrasives and work surface. Up to now, epi-ready surfaces have been delivered commercially. However, micro-scratches and crystallographic damage are accidentally induced during CMP, degrading the device performance.

Recently, an abrasive-free polishing method based on a chemical etching reaction was developed in our group and was named the catalyst-referred etching (CARE) method [4–13]. A schematic outline of the CARE concept is shown in **Fig. 1**. A catalytic pad is utilized to perform etching exclusively on the pad surface. Therefore, the topmost part of the work surface, which is more in contact with the pad surface than other parts, is preferentially removed. The pad surface also acts as a reference pad that is statistically copied onto the work surface, similar to the pad in the CMP method. The chemical-oriented etching mechanism of CARE thereby smoothens the work surface effectively. During CARE, platinum and hydrofluoric (HF) solution are generally employed as catalyst and etchant, respectively.

Hara et al. [4] first proposed the CARE method for flatting SiC substrates. Arima et al. [5] proposed electroless etching through oxidation of the SiC surface by holes injected into its surface. The oxide layer was then removed in HF. However, the proposed etching reaction could not explain the high concentration of F^- terminations on the processed SiC surface [6]. In later works, intensive experimental studies were performed to understand the removal characteristics. Okamoto et al. [7–9] clarified the dependence of the material removal rates (MRRs) on different conditions.

Bui et al. [10] clarified surface structures of a CARE-processed surface by using high-resolution transmission electron microscopy (TEM). Using density functional theory (DFT) simulations, Bui et al. [11, 12] proposed an etching reaction via HF dissociative adsorption on a step-edge Si atom of the topmost SiC surface. Based on the proposed mechanism, recently, Isohashi et al. [13, 14] experimentally demonstrated that pure water also etches 4H-SiC via CARE, similar to the HF solution.

In this study, we review the removal characteristics of CARE for 4H-SiC with HF and water. The removal mech-

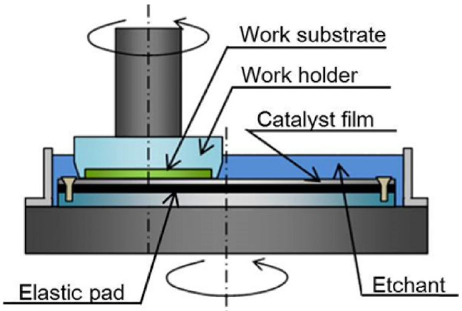

Fig. 2. Schematic drawing of CARE apparatus. Work substrate is placed on catalyst plate in wafer holder and pressed onto catalyst film. Substrate and catalyst pad independently rotate on each axis.

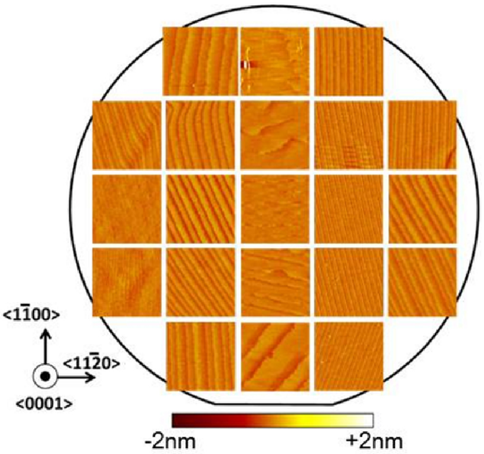

Fig. 3. AFM images in 10-mm intervals over entire area of 2-inch SiC wafer. View area is 2×2 μm^2.

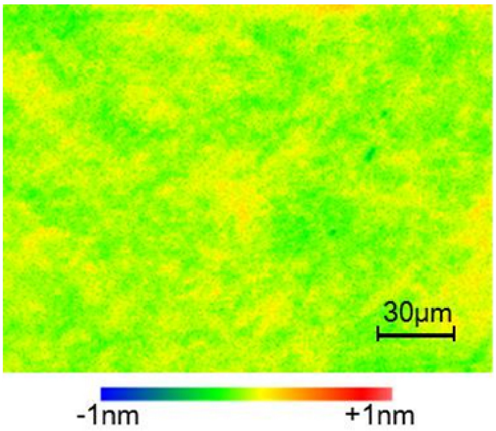

Fig. 4. Typical optical microscopic interferometer image of CARE-processed SiC. View area is 187×140 μm^2.

anism of CARE with HF clarified by density functional theory (DFT) is also reviewed and discussed.

2. CARE Apparatus and Processed Surfaces

The CARE method employs nearly the same apparatus as conventional CMP methods. **Fig. 2** shows a schematic drawing of a CARE apparatus. The work substrate is placed in the wafer holder and pressed onto the polishing pad, on which a ~100-nm-thick catalytic metal is deposited. The pressure between the work substrate and pad surface is controlled with an air bag placed behind the work substrate. The work substrate and pad independently rotate on each axis at nearly the same rotational speed. They are fully immersed in the etchant.

A 2-inch n-type on-axis-cut 4H-SiC wafer with a resistivity of 0.02–0.03 Ωcm was processed under CARE conditions. The rotational speed, processing pressure, concentration of the HF solution, and processing time were 10 rpm, 100 hPa, 25 mol/ℓ, and 1 h, respectively. The processed surfaces were observed with atomic force microscopy (AFM, SII Nanotechnology, Inc., Chiba, Japan, SPA400 + SPI3800N) and optical interferometry (ZYGO Corp., Middlefield, CT NewView 200 CHR).

The AFM observations were carried out in 10-mm intervals over the entire area of the 2-inch wafer (**Fig. 3**). Each view area is 2×2 μm^2. The average roughness of the entire wafer is 0.092 nm rms. Periodic step-and-terrace structures were observed during all measurements. The step has a height of one bilayer, showing that the processed surface is geometrically and crystallographically highly ordered at the wafer scale. **Fig. 4** shows a typical image of an optical interferometer measurement with an area of 187×140 μm^2. The rms roughness is less than 0.1 nm. The removal rate is proportional to the atomic step density because CARE proceeds only at the step edge because of the step-flow etching phenomenon. The removal rate is not high, that is, ~10 nm/h, because the wafer is cut along the $< 0001 >$ axis and has a low step density. In the case of an off-axis-cut wafer with an angle of 4°, which is utilized for device substrates, the step interval decreases to the nm level and the removal rate increases to $>$ 100 nm/h [8, 9].

Based on first-principles simulations, as the first step of the removal process of CARE for SiC with HF solution, Bui et al. investigated the dissociative adsorption of the HF molecule at the back bond of the surface [11, 12]. A simulation was performed with a slab model containing a stepped 3C-SiC(221) surface and Pt(111), as shown in **Fig. 5**. By employing the 3C-SiC model consisting of four bilayers in a unit cell, the calculated adsorption energies and activation barriers were expected to be similar to those of 4H-SiC(0001) surfaces because of comparable local atomic configurations and surface energies [11, 12].

The Pt(111) surface was selected based on a previous study showing a high-intensity Pt(111) peak in the X-ray diffraction pattern of deposited Pt [15]. A stepped Pt surface was employed because of its high activity, which is ideal for the identification of the low-energy reaction pathway. A vacuum region with a thickness of ~20 Å in a direction normal to the Pt(111) surface was employed to separate the successive slabs. The C atoms at the edge of the step and dangling bonds in the bottom layers of the

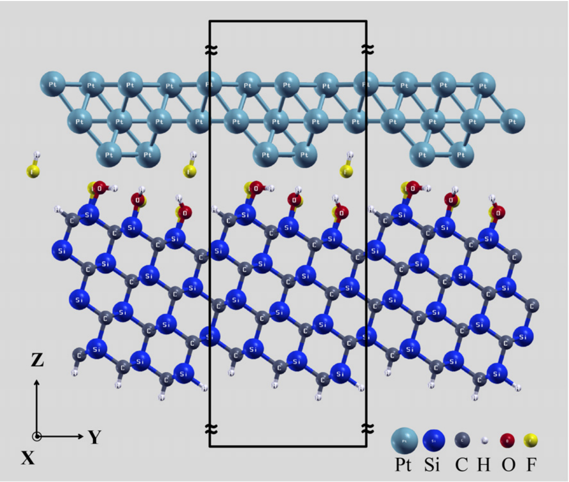

Fig. 5. Side view of 3C-SiC(111) model with step-and-terrace structure. Box indicates a unit cell.

SiC model were terminated by H atoms.

Additionally, Si atoms of the topmost surface were terminated by Si-OH or Si-F. During the simulations, the adsorbates, the two topmost bilayers of SiC and terminated atoms, and the bottom layer of Pt were allowed to fully relax, while the position of the remaining atoms was fixed to maintain the substrate and Pt(111) structures. The calculations were performed with the Simulation Tool for Atom Technology (STATE) program package, which has been successfully applied to investigations on metals, semiconductors, and organic materials [6, 16, 17].

The first-principles simulations were based on a generalized gradient approximation and the Perdew-Burke-Ernzerhof functional [18]. The ion cores were replaced with Troullier-Martins-type norm-conserving pseudopotentials for the Si atoms and ultrasoft pseudopotentials for the C, H, O, F, and Pt atoms [19]. The valence wave functions and charge densities were expanded in the plane wave basis sets with cutoff energies of 25 and 225 Ry, respectively.

A $3 \times 2 \times 1$ uniform k-point mesh was used for the entire surface of the Brillouin zone. To calculate the reaction pathways, the climbing image nudged elastic band method was adopted [20, 21]. Optimization was performed iteratively until the residual forces acting on all atoms at the saddle point were reduced to $< 10^{-3} E_h/a_0$, where $E_h = 27.211$ eV and $a_0 = 0.529$ Å.

Calculations were carried out with and without a Pt catalyst. The results are shown in **Fig. 6**. In this figure, the energy diagram along the reaction pathway from the initial state (IS) to the final state (FS) via the metastable state (MS), and the atomic configurations at these states, are represented for the cases with and without a Pt catalyst. In the case without Pt, the water molecule is dissociatively adsorbed onto the step-edge Si to form fivefold coordination (MS), leading to the weakening of the back bond.

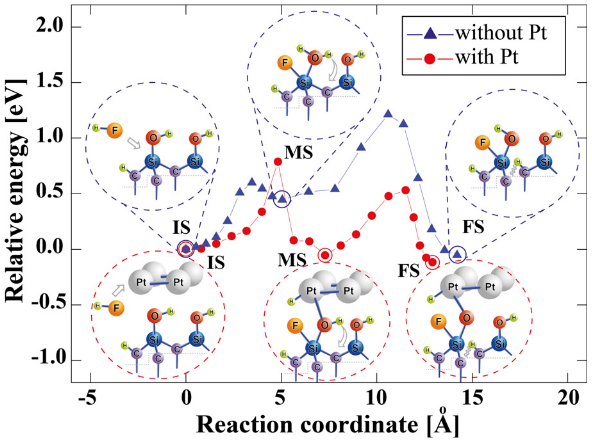

Fig. 6. Reaction pathway of CARE with HF solution. In initial state, HF molecule is placed between Pt and Si-C surfaces. Fivefold coordination forms at target Si through a dissociative adsorption of the HF molecule. In the intermediate state, the back-bond of the Si is weakened; in the final state, H^+ transfers from OH on Si to the back-bond to break.

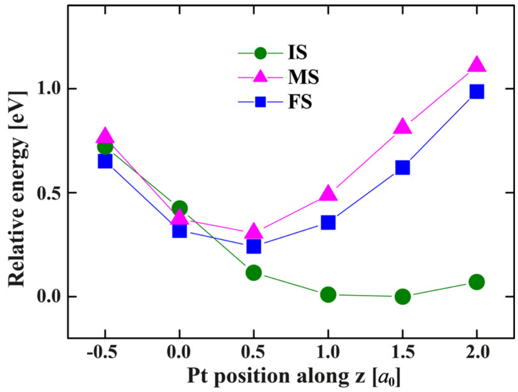

Fig. 7. Relative energies at IS, MS, and FS when the stepped Pt(111) surfaces are moved toward the SiC surfaces along the z-direction [12].

Subsequently, H^+ transfers from OH on Si to the second-layer C to break the Si-C back bond (FS). The reaction barrier is 1.2 eV, which is high enough for the reaction not to proceed at room temperature. In the case with a Pt catalyst, the barrier is reduced to 0.8 eV, which is sufficiently low to proceed at room temperature in spite of similar reactions. These are types of indirect dissociative adsorption of an HF molecule onto a Si-C back bond. The role of the Pt catalyst is to dissociate the HF molecule and stabilize the MS from 0.5 to −0.05 eV. The latter effect is more significant for the reduction of the reaction barrier.

To understand the stabilization mechanism, the total energies at the IS, MS, and FS are plotted in **Fig. 7** by changing the distance between the Pt and SiC surfaces from −0.5 to 2.0 a_0. The zero value is defined at the distance where the energy diagram shown in **Fig. 6** was calculated. The results show that the total energy of IS increases with decreasing distance owing to Pauli repulsion. By contrast, MS and FS stabilize with decreasing distance and approach a minimum around 0–0.5 a_0. The origin of this stabilization is reflected by the interface bonding between Pt and O on the targeted Si.

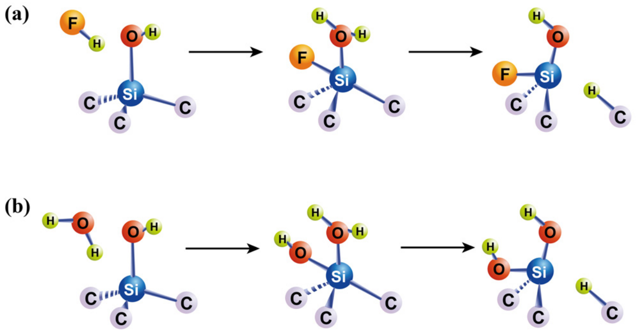

Fig. 8. Schematic of possible reaction pathways of (a) CARE with HF, which is theoretically considered to be a type of indirect dissociative adsorption of HF molecule that breaks the Si-C back-bond, and (b) CARE with pure water, which is expected to occur owing to similarity of F and OH.

The energy barrier to form a hypervalent silicate at MS, which leads to back-bond weakening, is significantly reduced owing to this mechanism. After the hypervalent silicate is formed, back-bond breaking proceeds rather easily, with and without a Pt catalyst. However, owing to the relatively high energy of the MS of the model without Pt, the total activation barrier is high, in contrast to that of the model with Pt. The key role of Pt enabling the reaction at room temperature is the stabilization of the MS by appropriate bonding with O that is terminated on the targeted Si.

3. CARE with Pure Water

Based on the discussion above, the mechanism of 4H-SiC via CARE with HF solution is an indirect dissociative adsorption of the HF molecule onto the back bond. In the reaction pathway, the targeted Si forms a fivefold coordination at the MS through an additional adsorption of F^-, resulting in a weakening of the back bond. It is generally known that Si can form hypervalent silicates with Lewis bases, such as F^- or OH^-, which are considered intermediate states in a variety of chemical reactions owing to their high nucleophilicities [22–26]. Therefore, OH^- is considered to play a role similar to that of F^-, as shown in **Fig. 8(b)** (**Fig. 8(a)** represents CARE with HF solution as a reference), that is, to induce an indirect dissociative adsorption of H_2O, an indirect hydrolysis.

First, Isohashi et al. [13, 14] applied the method to the 3-inch n-type 4H-SiC(0001) on-axis and 4°-off-axis samples at a rotational speed of 30 rpm and an applied pressure of 800 hPa to understand the fundamental etching characteristics. The processing time was 1 h. The processed surface was observed with AFM. **Figs. 8(a)** and **(b)** show AFM images of the preprocessed and processed samples of a 4H-SiC(0001) on-axis wafer, respectively. The view area is $2 \times 2\ \mu m^2$.

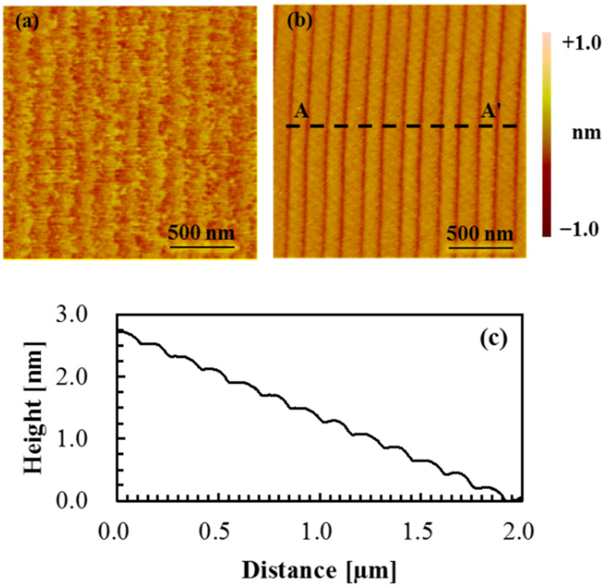

Fig. 9. AFM images of (a) preprocessed and (b) processed surfaces of 4H-SiC(0001) on-axis-cut wafer. (c) Cross-sectional image obtained along dashed line between A and A' in (b) [14].

The surface morphology was drastically changed after processing, and a straight step-and-terrace structure with a step height of approximately 0.25 nm was observed (**Fig. 9**), which corresponds to the height of a bilayer of Si and C. This result shows that pure water can etch 4H-SiC, similar to the HF solution, with the assistance of a Pt catalyst. The highly ordered surface indicates that the removal proceeds via a step flow and is based on a chemical phenomenon similar to the case of CARE with HF solution [14].

The material removal rates (MRRs) of the on-axis and 4°-off-axis wafers were 1–2 and 19 nm/h, respectively. The MRR for CARE with HF solution is known to be roughly proportional to the atomic-step density corresponding to the off-angle against the (0001) surface [8]. Thus, the MRRs obtained using pure water exhibited similar characteristics.

During the semiconductor fabrication process, the epitaxial layer is grown on the wafer to produce a high-crystallinity layer. However, the epitaxial growth induces surface waviness owing to step bunching, which can significantly degrade the performance of the device [27–33]. The processing time was 3 min. **Figs. 10(a)–(c)** show AFM images obtained from a $2 \times 2\ \mu m^2$ area of the preprocessed surface and surfaces processed at 2 and 3 min, respectively. The waviness appears to have been removed after 2 min, as evident in the topmost part of **Fig. 10(b)**.

Finally, the bunching was removed to yield a sufficiently smooth surface with a roughness of 0.051 nm rms within 3 min (**Fig. 10(c)**) [14]. Based on the results described above, CARE with pure water can etch the 4H-SiC wafer and atomically smoothen the surface. This method uses no chemical or slurry, unlike conventional mechanical and chemical polishing methods, and thereby ensures higher compatibility with processes used in semiconductor fabrication.

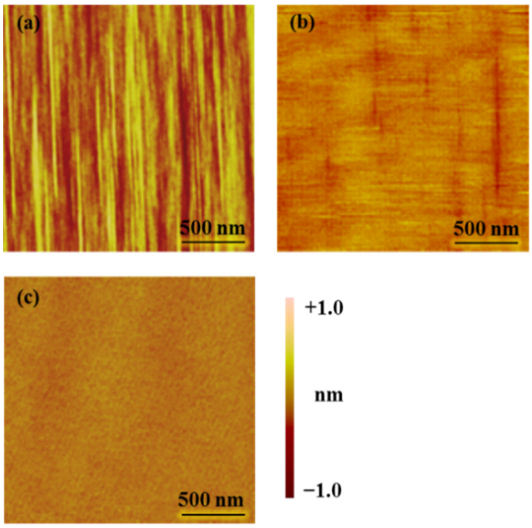

Fig. 10. AFM images of (a) epitaxially grown surface, (b) 2-min CARE-processed surface, and (c) 3-min CARE-processed surface of 4H-SiC(0001) 4°-off-axis-cut wafer [14].

4. Conclusion

The removal characteristics and mechanism of CARE for 4H-SiC with HF were reviewed. The results show that the main reaction of CARE is a catalytically induced indirect dissociative adsorption of the HF molecule, and that the processed surface is highly geometrically and crystallographically ordered. The CARE method using pure water was also reviewed. The results show that the water molecule plays a role similar to that of the HF molecule owing to the high nucleophilicity of OH^-. The obtained surfaces are of nearly the same quality as those obtained from CARE with HF solution. CARE with pure water is a novel chemical polishing method that is expected to contribute to sustainability and environmental harmony.

Acknowledgements

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Name:
Pho Van Bui

Affiliation:
Ultra-Precision Machining Laboratory, Osaka University

Address:
2-1 Yamada-oka, Suita, Osaka 565-0871, Japan

Brief Biographical History:
2016 Ph.D. degree (Engineering), Osaka University
2016- Visiting Scientist, Osaka University

Main Works:
- Density functional theory calculations and modeling the dynamics of etching and catalytic processes at surfaces and interfaces
- Experimental surface observation and study of catalysts

Membership in Academic Societies:
- Vietnamese Theoretical Physics Society

Name:
Yasuhisa Sano

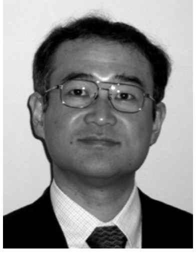
Affiliation:
Ultra-Precision Machining Laboratory, Osaka University

Address:
2-1 Yamada-oka, Suita, Osaka 565-0871, Japan

Brief Biographical History:
1993-2004 Assistant Professor, Osaka University
2003 Ph.D. degree (Engineering), Osaka University
2004- Associate Professor, Osaka University

Main Works:
- Precision machining
- Processing semiconductor substrates
- X-ray optics

Membership in Academic Societies:
- Japan Society for Precision Engineering (JSPE)
- Japan Society of Applied Physics (JSAP)



Name:
Yoshitada Morikawa

Affiliation:
Computational Physics Laboratory, Osaka University

Address:
2-1 Yamada-oka, Suita, Osaka 565-0871, Japan

Brief Biographical History:
1994 Ph.D., The University of Tokyo
2004-2009 Associate Professor, The Institute of Scientific and Industrial Research, Osaka University
2009- Professor, Osaka University

Main Works:
- Physics, quantum simulation
- Surfaces and interfaces

Membership in Academic Societies:
- Physical Society of Japan (JPS)
- Surface Science Society of Japan (SSSJ)

Name:
Kazuto Yamauchi

Affiliation:
Ultra-Precision Machining Laboratory, Osaka University

Address:
2-1 Yamada-oka, Suita, Osaka 565-0871, Japan

Brief Biographical History:
1991 Ph.D., Osaka University
2003- Professor, Osaka University

Main Works:
- Precision machining and metrology
- Surface science and technology
- X-ray optics

Membership in Academic Societies:
- Japan Society for Precision Engineering (JSPE), Fellow (2016-)
- Japan Society of Applied Physics (JSAP)
- Japan Society for Synchrotron Radiation Research (JSR)
- Surface Science Society of Japan (SSSJ)
- International Society for Optical Engineering (SPIE), Fellow (2017-)
- Optical Society of America (OSA), Fellow (2017-)