

Assessment of Grinding Characteristics of CVD-SiC Through Indentation Testing

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Abstract—The primary focus of this study is to delve into the theoretical basis of CVD-SiC and to develop a predictive model for its grinding properties. This research involves a comparative analysis of the grinding properties of CVD-SiC through Vickers indentation and grinding experiments. The findings offer a practical calibration method for determining the optimal depth in ductile mode grinding for common applications. Additionally, the study discusses the influence of various parameters on the grinding efficiency and surface integrity of CVD-SiC, providing insights into the material's behavior under different operational conditions. The analysis further explores the correlation between indentation hardness and grinding performance, establishing a comprehensive understanding of how microstructural characteristics impact the overall machinability of CVD-SiC. This work contributes to the development of more efficient grinding techniques and improved material performance in industrial applications.

Keywords: CVD-SiC; Vickers Indentation; Grinding; Optical components; Material removal mechanisms.

I. INTRODUCTION

Silicon carbide ceramics produced through chemical vapor deposition (CVD-SiC) exhibit outstanding thermal resistance and exceptional physical shock resistance, making them ideal for applications such as reflection mirrors and optical element molds. These applications demand surfaces with low roughness and high form accuracy. Achieving such precise machining for CVD-SiC requires a thorough understanding of the material removal mechanisms involved. The inherent brittleness and hardness of CVD-SiC pose significant challenges in the machining process.

To address these challenges, extensive research has been undertaken with a focus on ductile grinding techniques. The evolution of nanoindentation technology, first introduced and developed by W.C. Oliver, has played a crucial role in these efforts. Nanoindentation is a classical mechanical testing method that has long been a standard for material characterization. This method allows for the comprehensive assessment of the mechanical properties of surface materials.

Hardness testing, in particular, is widely used in both industrial and research settings due to its simplicity, cost-effectiveness, and reliability in evaluating the basic properties of new and developed materials. The indentation process involves a small indenter that causes minimal damage to the bulk material, making it an ideal method for assessing machining properties. Indentation testing has been specifically applied to evaluate the fracture toughness of materials by measuring the cracks produced by a Vickers indenter under varying loads.

This study aims to expand on the understanding of CVD-SiC's grinding properties by employing indentation tests to predict and improve machining outcomes. By correlating the results of indentation tests with ductile mode grinding techniques, we can develop more effective and precise machining processes for CVD-SiC mirrors and optical components. This approach not only enhances the surface

quality and accuracy of CVD-SiC components but also contributes to the development of cost-effective manufacturing methods.

Furthermore, this research explores the relationship between microstructural characteristics and machining performance. The insights gained from these studies are crucial for advancing the application of CVD-SiC in high-precision industries, ensuring that the material's exceptional properties can be fully leveraged while maintaining manufacturing efficiency and effectiveness.

II. PRINCIPLE AND METHODS

Mechanical performance is a critical indicator of material quality and serves as the primary basis for design and calculation. Early experiments using load and displacement sensing indentation methods to measure mechanical properties were conducted by Tabor, who investigated the indentation behavior of various metals deformed by hardened spherical indenters. Building on this, Stillwell and Tabor explored the behavior of conical indenters, furthering the understanding of indentation mechanics.

In brittle materials, sharp indentation tests are used to initiate and control fracture tests. Hardness, a key mechanical property, is typically defined as the ratio of the applied load to the projected contact area of the indentation. The indentation depth is the sole characteristic length in this context, influencing the assessment of material properties.

The Vickers indentation test, a widely used method for evaluating hardness, involves a diamond indenter with a pyramidal geometry. This test allows for precise measurement of hardness and is particularly useful for brittle materials like CVD-SiC. The test's geometry, as shown schematically in Fig. 1, ensures consistent and reproducible results, facilitating the analysis of mechanical performance.

This study leverages the Vickers indentation test to evaluate the mechanical properties of CVD-SiC, aiming to enhance our understanding of its grinding characteristics. By

correlating indentation results with the material's behavior under ductile mode grinding, we seek to optimize machining processes for CVD-SiC components. This approach not only improves surface quality and form accuracy but also contributes to the development of cost-effective manufacturing techniques for high-precision applications. The insights gained from these experiments are essential for advancing the use of CVD-SiC in industries requiring exceptional material performance and precise machining capabilities.

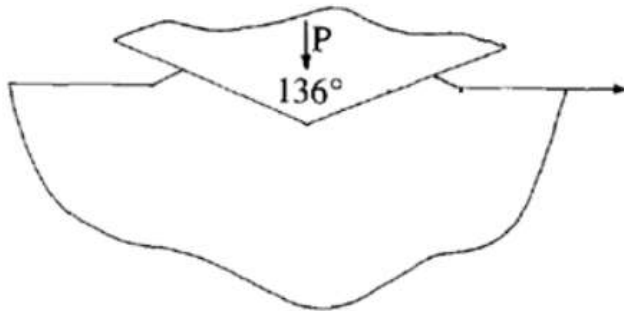


Fig. 1. Schematic of Geometry of the Vickers Test Experiment

III. EXPERIMENT SETUP AND METHOD

The workpiece selected for this study is depicted in Fig. 2. Indentation experiments were conducted using a micro Vickers hardness tester, which primarily consists of three components: the indenter system, an optical microscope, and a precise sample mobile worktable. The indentation load was varied during the experiments to obtain a range of results.

For the grinding experiments, a CNC grinding machine was utilized. The surface characteristics and quality of the material post-experimentation were analyzed using non-contact surface detection equipment, specifically Mitaka and Zygo systems. These advanced surface detection tools provided accurate and detailed measurements of the material's surface, ensuring reliable and comprehensive experimental results.



Fig. 2. CVD-SiC workpiece and Micrograph of Surface

This methodology allowed for a thorough investigation of the grinding properties of CVD-SiC. By using variable indentation loads and precise surface detection techniques, we were able to correlate the hardness and indentation results with the grinding performance of the material. The integration of advanced optical microscopy and accurate mobile worktables

ensured high precision in the indentation experiments, while the CNC grinding machine facilitated controlled and replicable grinding processes.

Overall, the experimental setup and methodology provided a robust framework for assessing the mechanical performance and grinding characteristics of CVD-SiC, contributing valuable insights into the optimization of machining processes for this advanced material.

IV. RESULTS AND DISCUSSION

The nanoindentation process is a quasi-static method where the indenter is gradually pressed into the material being measured. During these tests, various loads were applied to study the material's response under different conditions. The corresponding surface profiles and images were captured using a Zygo measuring instrument, a white light interference microscope, which provided detailed and accurate measurements of the indentation marks. This setup allowed for a comprehensive analysis of the material's mechanical properties and surface characteristics.

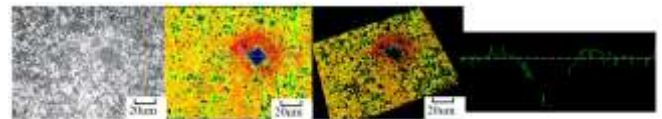


Fig. 3. Shows the experimental results obtained using the Zygo measuring instrument

The plastic deformation zone arises from the non-elastic flow of the specimen material beneath the indenter, and its size expands with increasing load. As depicted in the right image of Fig. 3, a clear pile-up behavior is observed. Additionally, in the colorful image, the red area indicates the region affected by the pile-up effect. This observation underscores the influence of load on the extent of plastic deformation and pile-up behavior in the material.

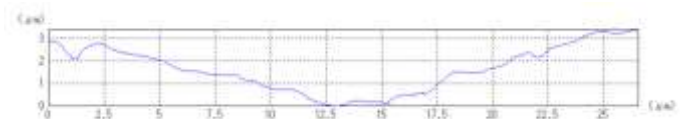


Fig. 4. Illustrates the surface crack profile resulting from an 850gf load, as acquired using the Mitaka PF-2SA system

V. EXPERIMENT RESULTS AND ANALYSIS

5.1 Material Removal Rate Analysis

The material removal rate (MRR) of sapphire under various polishing conditions is depicted in Fig. 1. Samples 1, 2, and 3 exhibit relatively low MRRs. These samples utilize the 1# pad, with polishing slurries containing silica and alumina particles, akin to traditional loose abrasive polishing methods.

Figure 4 represents an indentation profile captured by the Mitaka measuring instrument, wherein several measured shapes closely resemble the shape of the indenter. In these experiments, a Vickers indenter with a 136° face angle was utilized. It was observed that as the indentation load increased, the profiles more closely resembled the shape of the indenter. This transition suggests a shift in material properties from

elastic to plastic deformation due to variations in indentation load and depth.

Consequently, these findings have implications for the grinding process. By analyzing the similarity between the indentation profiles and the indenter shape, insights can be gained into the properties of the material. This understanding enables the determination and application of appropriate grinding grit sizes and depths in the grinding process, facilitating more precise and efficient machining of the material.

By measuring both the indentation load and the diagonal of the indentation on the specimen, hardness can be calculated according to the ASTM Standard Test Method C1327, expressed as:
$$HV = 1.8544 \left(\frac{P}{d^2} \right) \quad (1)$$

Where P is the applied load in Newtons(N) and d is the mean diagonal of the indentation in millimeters(mm).

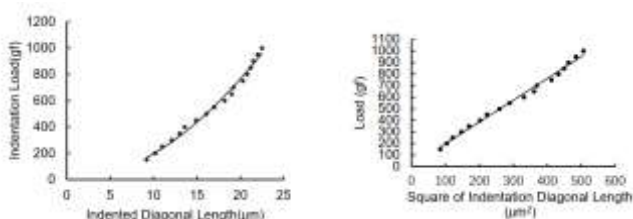


Fig. 5. Plot of the Indentation Load vs. Indented Diagonal Length on CVD-SiC

Figure 5 depicts a plot of the indentation load against the square of the mean indentation diagonal. It is evident from the plot that the curve representing load versus indented diagonal length during the experiments is nonlinear. However, another curve exhibits linearity. The slope of this linear curve, represented as (P/d^2) , provides a measure of the hardness value as per Equation (1).

The agreement between the results of the indentation experiments and the hardness equation is demonstrated, indicating the effectiveness of the methodology in determining material hardness.

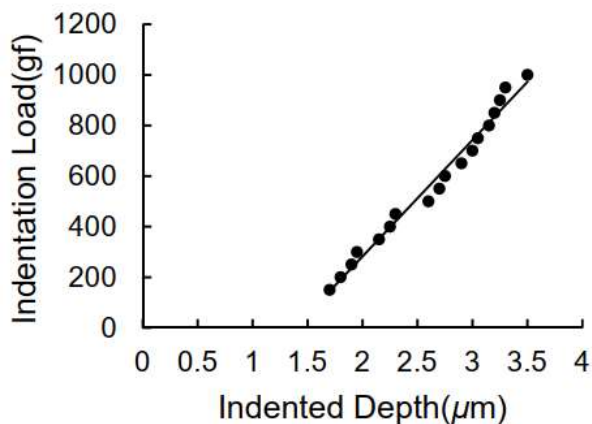


Fig. 6. Indentation Load-Indented Depth curve for CVD-SiC

The results for the material are plotted in Fig. 6 as pressed loads vs. indented depth. The indentation morphology of the

CVD-SiC after a Vickers indentation test with different pressed loads under a laser microscope is shown in Fig. 6.

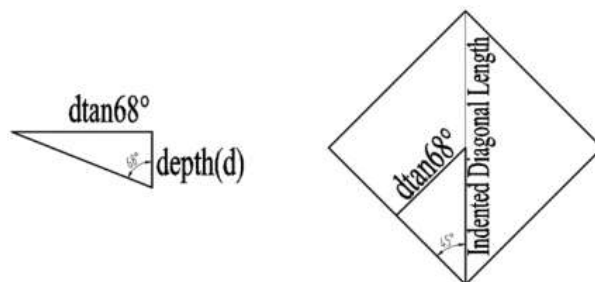


Fig. 7. Schematic Diagram of Corresponding Dimension

Indentation areas function may be obtained that describing the geometry of the indenter.

$$A = 4 \tan^2(68^\circ) d^2 \quad (2)$$

Where A is indentation projected contact area and d is the indented depth.

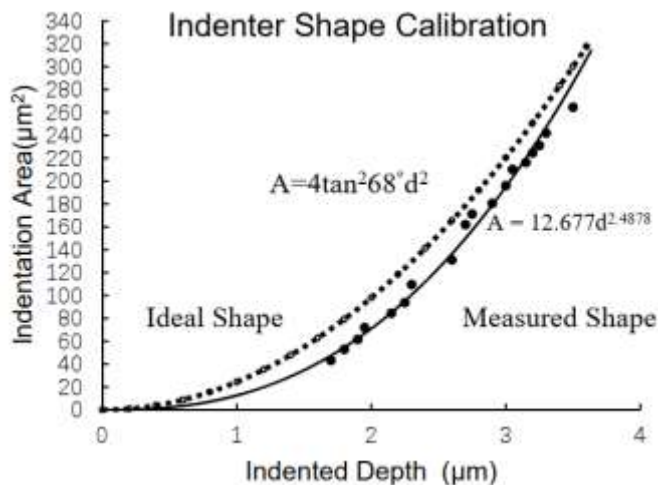


Fig. 8. Indenter Shape Calibration

The results of indenter shape calibration are shown in Fig. 8. These data were obtained from individual indentation. Determination of the indenter geometry is also important to the measurement of elastic properties.

To validate the assumptions derived from the indentation experiments, a grinding experiment was conducted. The critical depth obtained from the indentation results was approximately 3 μm. To verify this assumption, grinding wheels with grit sizes corresponding to the experimental conditions were selected. Traverse grinding was performed using a CNC grinding machine equipped with metal-bond diamond tools with grit sizes of 30 μm and 8 μm.

The effects of various grinding conditions, such as grinding wheel rotation speed, feed rate, and grit size, on form accuracy, surface roughness, and grinding-induced damage were investigated. The surface characteristics and quality of the machined samples were analyzed using Zygo and Mitaka measuring instruments. These tools provided detailed measurements, allowing for a comprehensive assessment of

the impact of different grinding parameters on the material's surface properties.

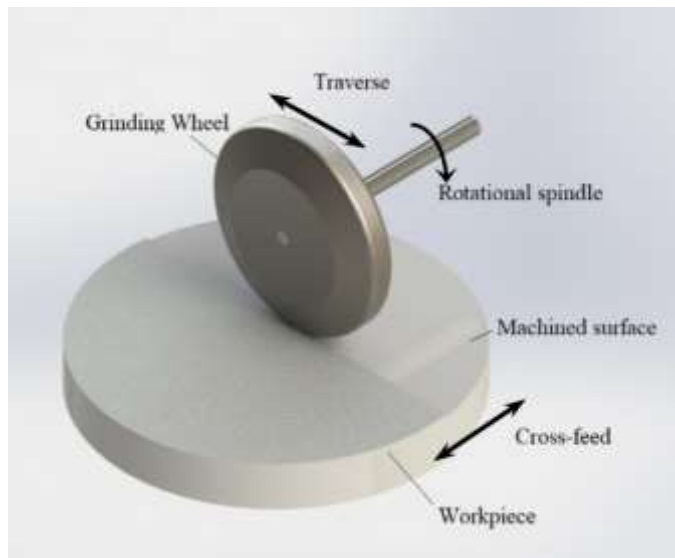


Fig. 9 Schematic of Grinding Experiment

TABLE 1. Main parameters of grinding experiment

Experimental Conditions	Values
Workpiece Material	CVD-SiC (ø18mm×25mm)
Grinding Wheel Abrasive	Diamond
Grinding Wheel Type	SD400#, SD1500#
Grinding Depth	8µm, 2µm
Grinding Wheel Rotational Speed	3000 rpm
Grinding Feed Speed	300mm/min
Scan Method	XY-YX scan
Scan Pitch	0.08mm, 0.04mm

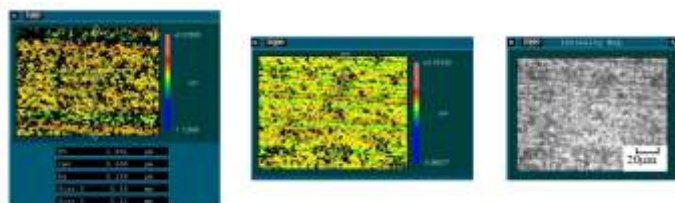


Fig. 10. Grinding Experiment Results with SD400# Obtained by Zygo



Fig. 11. Grinding Experiment Results with SD1500# Obtained by Zygo



Fig. 12. Machined Surface Profiles with SD 400# and SD1500# Wheels Obtained by Mitaka

Figures 10 and 11 display surface roughness and surface topography after grinding, respectively. Additionally, Figures 12 depict surface profiles and photographs. These results demonstrate that surface roughness is indeed influenced by both the grit size and grinding depth.

VI. CONCLUSIONS

This paper presents the results of both indentation tests and grinding experiments, which serve as valuable tools for assessing the grinding properties of substrates coated with silicon carbide via chemical vapor deposition. The findings indicate that CVD-SiC material exhibits elastoplastic behavior, thereby adjusting its machining adaptability according to fabrication conditions. This offers a straightforward approach to evaluating ductile-regime parameters.

During Vickers indentation on the surface of CVD-SiC, the material's deformation process can be delineated into three stages: plastic deformation, crack propagation, and micro-crushing. As the applied load decreases during Vickers indentation testing, the indentation undergoes a brittle-plastic transition. The plastic deformation zone of the indentation enlarges, and the crack depth increases with rising load. At low loads, the material demonstrates noticeable elastic rebound, resulting in an uneven indentation area. However, as the load increases, the indenter profile becomes more pronounced, indicating predominant plastic deformation.

The identification of optimal surface conditions hinges on discerning critical conditions, with the critical depth of brittle-plastic transition serving as a crucial parameter. This study introduces a practical method for determining machining properties through indentation experiments.

REFERENCES

- [1] Oliver, W.C., Pharr, G.M. An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments. *Mater Res* 7(6), 1546-1583 (1992).
- [2] Tabor, D. *Proc. R. Soc. A* 192, 247 (1948).
- [3] Stillwell, N.A., Tabor, D. *Proc. Phys. Soc. London* 78, 169.
- [4] Kasuga, H., Ohmori, H. Investigation on mirror surface grinding characteristics of SiC materials. *Journal of Ceramic Processing Research* 10(3), 351-354 (2009).
- [5] Gogotsi, Y., et al. Nanoindentation and mechanical properties of ceramic coatings. *Journal of Materials Research* 15(12), 2737-2745 (2000).
- [6] Wang, Y., et al. Microstructure and mechanical properties of chemical vapor deposited silicon carbide (CVD-SiC) coatings on silicon substrates. *Thin Solid Films* 515(24), 8689-8695 (2007).
- [7] Zhang, Y., et al. Effect of diamond grinding parameters on the surface integrity and subsurface damage of SiC ceramics. *International Journal of Machine Tools & Manufacture* 46(12), 1484-1491 (2006).
- [8] Nakayama, K., et al. Influence of grain boundary phase on the grinding of SiC. *Journal of the European Ceramic Society* 28(1), 125-133 (2008).
- [9] Wang, H., et al. Study on the grinding characteristic of silicon carbide ceramics with diamond wheel. *Key Engineering Materials* 329, 431-436 (2007).
- [10] Yang, J., et al. Investigation on the surface topography and subsurface damage of SiC ground by diamond wheels with different grain size. *Journal of the European Ceramic Society* 29(3), 387-395 (2009).