

Cutting Performance of Nano-Polycrystalline Diamond

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Nano-polycrystalline diamond (NPD) obtained using direct conversion sintering process from graphite under high pressure and high temperature has a very fine texture composed of small diamond grains of several tens of nanometers without any binder materials or secondary phases. For this reason, NPD has significantly high hardness, no cleavage feature and high thermal stability. Because of its superior features, NPD is considered to be highly useful for cutting tools. Here, we describe some mechanical properties and several cutting performances of NPD. The Knoop indentation hardness tests showed NPD has very high hardness especially at high temperature; about 2 times higher than those of single-crystal diamonds (SCD) above 800°C. The data from wear tests using a diamond wheel indicated that the abrasive wear resistance of NPD is equivalent to those of the high wear-resistance directions on SCD, and 10–50 times higher than those of conventional polycrystalline diamond (PCD: sintered diamond with metal binder). The results of cutting tests for various work materials, such as Al-Si alloy, ceramics and cemented carbides, demonstrated that the NPD tool has a significantly higher cutting performance than conventional PCD and SCD tools. These results indicated that NPD has a high potential for wide applications in cutting and new processing fields.

Keywords: polycrystalline diamond, cutting tool, mechanical properties, hardness, abrasive, wear

1. Introduction

Single crystal diamond (SCD) is widely used as cutting tools and wear-resistant tools because of its highest hardness in all materials. However, the cleavage feature and the anisotropy of mechanical properties, which is peculiar to SCD, often causes practical problem according to applications.

On the other hand, commercially available polycrystalline diamond (PCD) containing metal binders or sintering aid such as Co has no cleavage feature and no anisotropy of mechanical properties. However, its mechanical properties degrade at high temperature due to the negative affect of metal binders containing at grain boundary.

We succeeded in the synthesis of single-phase polycrystalline diamonds using neither metal binder nor sintering aid by direct conversion sintering process from graphite under high pressure and high temperature^{(1),(2)}. This polycrystalline diamond consists of very fine diamond grains, which are several tens of nanometers, bonding directly each other with high bonding strength⁽³⁾. The nano-polycrystalline diamond (NPD) with no secondary phases has excellent high hardness superior to SCD⁽⁴⁾, and has neither cleavage feature nor anisotropy of me-

chanical properties⁽⁵⁾. For these excellent characteristics, NPD is expected as a new super hard material for cutting tools and wear-resistant tools. Currently we are developing mass-production technology for NPD in large size (8-10 mm) for commercial purposes (Fig. 1).

In this paper, we describe some properties such as high-temperature hardness, abrasive wear resistance and heat resistance, which are important in cutting tool applications. We also detail the results of cutting tests using NPD tools for various work materials. Then we discuss the cutting performance, based on the above material properties.

2. Synthesis Method and Characteristics of NPD

NPD specimens were synthesized by direct conversion sintering from high-purity isotropic graphite under pres-

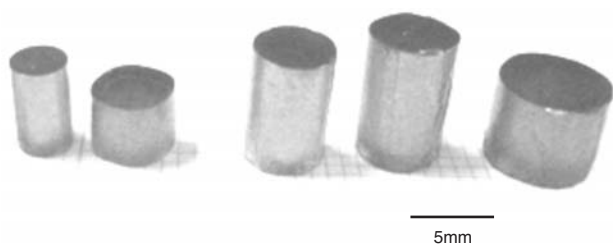


Fig. 1. Nano polycrystalline diamond (NPD)

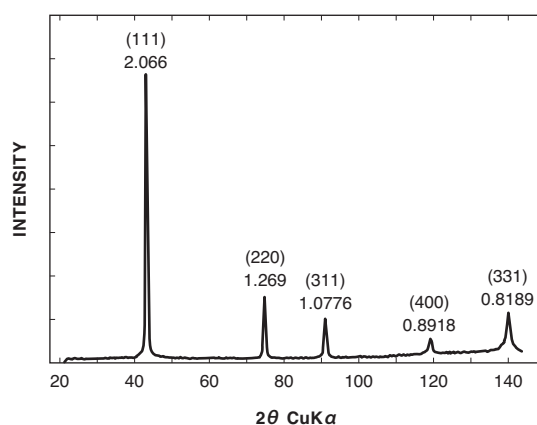


Fig. 2. X-ray diffraction pattern of NPD

tures of 14-18 GPa and temperatures of 2100-2300°C. The high pressure and high temperature conditions were generated with a multianvil apparatus and using a high melting point metal heater. **Figure 2** shows the X-ray diffraction pattern of NPD indicating that NPD has an isotropic polycrystalline pattern, and no secondary phases.

The typical transmission electron micrograph (TEM) of NPD shows NPD has a homogeneous fine structure consists of small granular crystals with 30-50 nm in average diameter (**Fig. 3**).

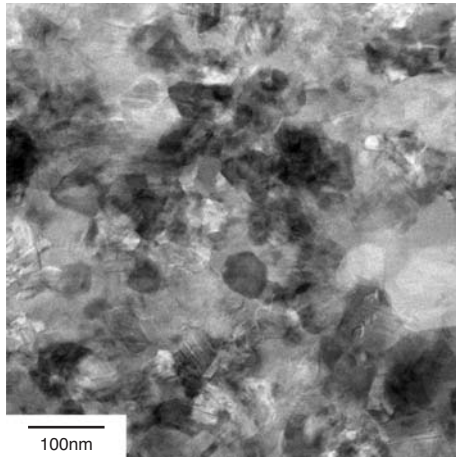


Fig. 3. TEM image of NPD

3. Mechanical Properties of NPD

Some basic properties which are important in cutting tool applications, such as hardness, wear resistance and heat resistance, were investigated to estimate the potential of NPD for cutting tools.

3-1 Hardness

The hardness of NPD was investigated by the indentation hardness test using Knoop indenters. The surfaces of specimens were finely polished to mirrored surfaces ($R_a \approx 20$ nm) using with a metal-bonding diamond wheel. After indenting the Knoop indenter on the polished surfaces with a load of 4.9 N or 9.8 N, the size of indentations was measured. We attempted to improve the precision and reliability of measured hardness values by always making comparative evaluation using the Knoop hardness in (001)<100> of a high-purity synthetic type IIa diamond crystal as reference.

Figure 4 shows the experimental results indicating the Knoop hardness of NPD has no anisotropy as SCDs do, and is equivalent to that of the highest direction of SCD at RT. In this hardness measurement test, we used the Knoop indenters which were made of synthetic high purity SCD (type IIa) by adjusting the tip orientation to the hard crystal orientation of the diamond (001)<110>⁽⁶⁾.

The high temperature hardness values on (001)<110> of synthetic type IIa SCD sharply decrease from 200°C because of thermally activated plastic deformation, and re-

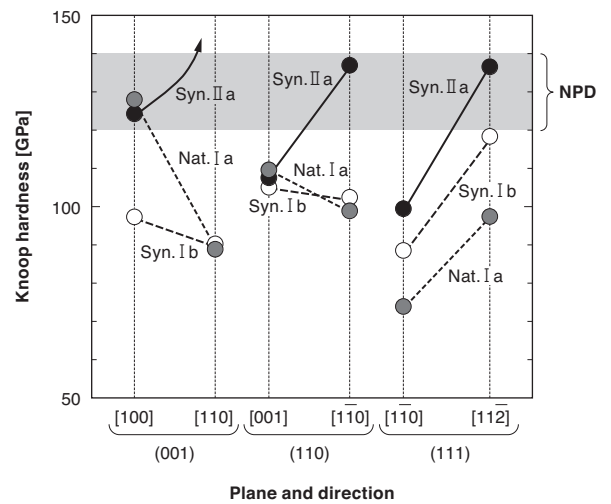


Fig. 4. Knoop hardness of NPD compared to those of various types of SCD

duce by nearly 50 % at temperatures over 300°C⁽⁷⁾. In the case of NPD, such a significant decrease of hardness is not observed. Consequently, high-temperature hardness tests were conducted by using special Knoop indenters made of NPD⁽⁸⁾. These NPD indenters made it possible to measure the high temperature hardness of NPD precisely, without breakage of the indenter at high temperatures.

Figure 5 shows the results of high-temperature Knoop hardness of NPD compared with synthetic type IIa SCD in an argon gas atmosphere. The decrease rate of hardness of NPD at high temperature is small, compared with the hardness of synthetic type IIa SCD. The Knoop hardness of NPD at 800°C is approximately twice as high as that of (001)<110> of synthetic type IIa SCD. In the case of SCDs, the plastic deformation at high temperatures induced by the slip deformation due to (111)<110> slip system, leading to a significant reduction of high temperature hardness.

The Knoop hardness of conventional PCD is around 50 GPa⁽⁹⁾, about 50% lower than those of NPD and SCD.

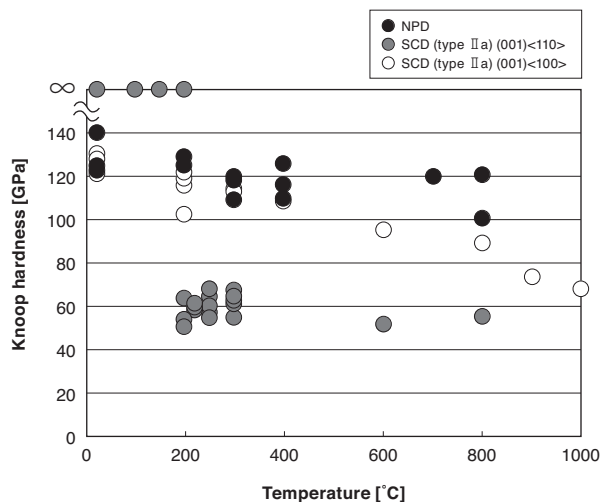


Fig. 5. Knoop hardness of NPD and synthetic SCD (type IIa)

Furthermore, the mechanical properties of PCD degrade at high temperatures of 600-700°C due to the metal binders. In the case of NPD, the blocking effect of dislocation movement (plastic deformation) at grain boundaries derives high-temperature hardness far beyond other diamond materials.

3-2 Wear resistance

The abrasive wear resistance of NPD against a diamond wheel was evaluated using a high speed polishing machine. The specimens were prepared to the size of 1×1×1 mm³, and pressed onto the metal-bonded diamond wheel, whose average grit size was 20 μm, rotating at a high speed of 2800 rpm at 3kgf/mm³ to measure abrasive wear loss. Since the surface condition of the diamond wheel varies drastically in the process of the abrasive test, the abrasive ability of the diamond wheel may be changed. To avoid such influence and maintain the test condition, we dressed the wheel using the grinding dresser for 10 seconds after every 15 seconds wearing test.

Figure 6 shows the abrasive wear rates of some NPDs with those of SCDs and PCDs. The abrasive wear resistance of NPD was approximately 20 times higher than that of synthetic type Ib SCD (001)<100> which is the low abrasive wear resistance direction, and equivalent to (112)<110> which is the high abrasive wear resistance direction, and 10-50 times higher than that of conventional PCDs. The high resolution scanning electron micrographs of wearing surface of specimens were shown in Fig. 7. The surface of every specimen was very rough, due to the high load and high speed test condition. Additionally, many abraded wounds and micro cracks are observed on the wearing surfaces, which were promoted by scratches and collision impact with the diamond grits of the metal-bonded diamond wheel.

On the wearing surface of NPD, many fine grain surface structures which are thought to be the high abrasive wear resistance crystallographic planes such as (111)<110> are observed standing slightly above the surrounding surface. In the case of SCD (type Ib), on the (001)<100> wear-

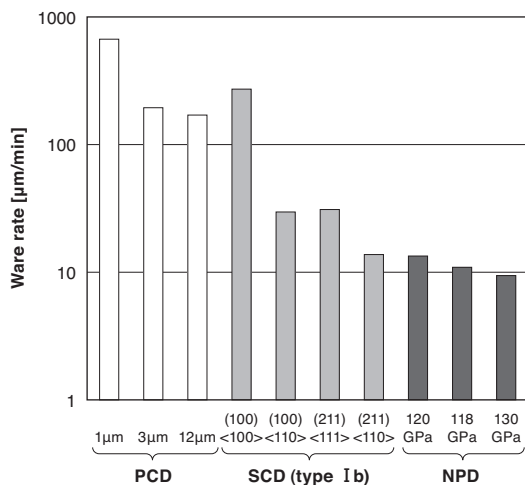


Fig. 6. Wear rate of NPD, PCDs (1μm, 3μm, 12μm) and SCD in various crystallographic orientations

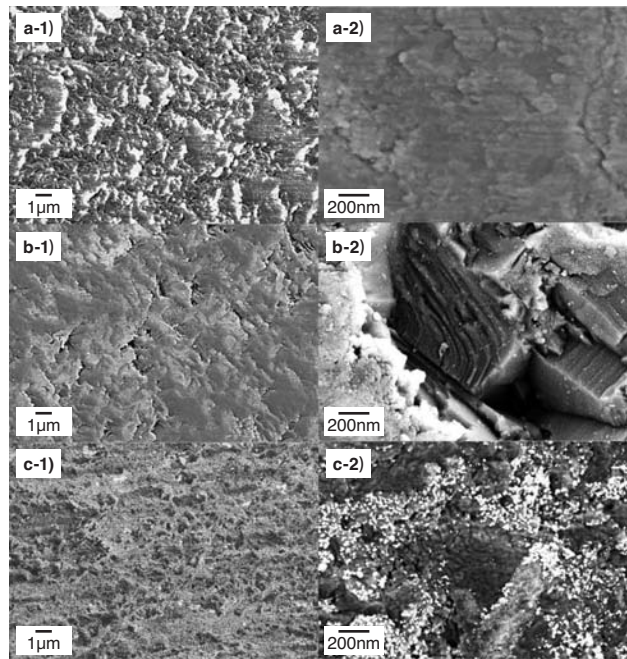


Fig. 7. High resolution SEM images of wearing surface (Enlarged images are shown in the right)
a: NPD
b: SCD (type Ib) (001)<100>
c: PCD

ing surface, the micro-cleavages appearing inside the cracks, and abrasive wear progresses on account of accumulation of micro-cleavages. On the wearing surface of PCD, micro cracks and traces of fallen grains are observed.

In the case of SCDs, the abrasive wear speed depends on the crystallographic orientation strongly. The anisotropy of abrasive wear property on mechanical process was investigated by Wilks⁽¹⁰⁾; our results on this work consist with their previous work. Therefore, on this test condition, the abrasive wear progresses due to the accumulation of micro cleavages, mechanical wear process is thought to be dominant, and the chemical wear process with the metal-bonded diamond wheel is negligible.

The abrasive wear resistivity of PCD depends on the size of diamond grains and the content of metal binders. It is commonly known that the abrasive wear resistance of PCD is deteriorated with smaller diamond grains and higher content of metal binders. The same tendencies were observed in this work. The conventional PCD consists of metal binders and continuous diamond structure which is formed of bonded diamond grains by catalysis of metal binders. The micro-cracks, which arise from thermal stress due to the thermal expansion difference between metal binders and diamond, progress by shock impact with the abrasive diamond grits of the wheel. The chemical wear due to bounding materials of the metal-bonded diamond wheel is thought to be negligible owing to the frequent dressing of the wheel. Therefore, the chemical attrition in this case seems to be due to graphitization of the surface of PCD grains promoted by catalytic action of metal binders in PCD such as Co. The wear due

to falling grains and graphitization was readily promoted, as the amount of grain boundaries containing Co binder increased with decreasing size of the diamond grains.

In contrast with SCD and PCD, NPD has no mechanical anisotropy and no metal binders, and its diamond grains bounded to each other directly and very strongly⁽⁵⁾. Therefore, the wear of NPD due to thermal degradation and falling diamond grain is negligible, and in every surface there are many diamond grains faced to hard planes such as {111} which are seen standing above the surrounding surface as shown Fig. 7 (a-2). As a result, NPD shows extremely high wear resistance equivalent to a high wear resistance direction of SCD.

3-3 Heat resistance

The surface of the specimens were finely polished to a mirrored surface of $Ra \approx 20$ nm using a metal-bonding diamond wheel. The heat resistance of NPD was estimated with Raman spectrometry and Knoop hardness tests on the polished surface, after heat treatment of 1000-1600°C in an inert atmosphere for one hour.

Both Raman spectra and Knoop hardness of NPD specimens were found to remain unchanged after heated up to 1400°C. The Knoop hardness of NPD after 1600°C heating declined by 8%. These results indicate that NPD has high heat resistance equivalent to SCDs.

It is known that diamond crystal is converted to graphite at temperature of 1500-1600°C in an inert atmosphere⁽¹¹⁾. The hardness of NPD and SCD after 1600°C heating degraded due to the deterioration of surface crystalline quality with graphitization.

In the case of PCD, the difference of the thermal expansion between metal binders and diamond leads micro-cracking at high temperatures above 600-700°C. Additionally, catalytic effect of metal binders in PCD seems to be activated at about 700°C, leading the conversion of diamond to graphite. Thus heating up the PCD over 600°C leads to drastic change in mechanical strength.

4. Cutting Performance

As discussed above, it was confirmed that NPD has excellent mechanical properties such as hardness, abrasive resistance and heat resistance, superior to SCDs and PCDs. Consequently, NPD is expected to be useful in cutting tool applications. Next, we made cutting tools from NPD and performed cutting tests with various work materials.

4-1 High-speed interrupted cutting of Al-alloy

NPD cutting tools having nose radius of 0.4 mm were prepared with high precision machining, and used to evaluate cutting performance on turning high-strength Al-alloy (17% Si-Al) cylinder with four U-shaped grooves, under high-speed cutting conditions as follows: the cutting speed (V_c) was 800 m/min, depth of cut (A_p) was 0.2 mm, and feed rate (f) was 0.1 mm/rev. The results are shown in Fig. 8. The wear resistance of NPD tools was approximately 10-20 times higher than that of conventional fine grain PCD tools. The tool wear of PCD progresses mainly due to thermal deterioration influenced by metal

binders such as Co, which give rise to micro-cracking, grain falling and graphitization as temperature rise at a cutting point. On the contrary, NPD is considered to have shown excellent abrasive wear resistance because it has no metal binders, excellent high thermal resistance and high bonding strength between diamond grains. It was also confirmed that when the NPD tool edge is made to be less than 0.1 μm , it provides high precision cutting surface with roughness of $Ra \approx 0.7 \mu\text{m}$, which is equivalent to that by SCD tools.

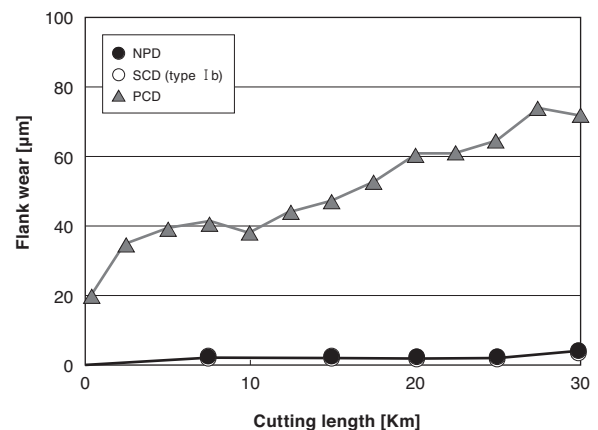


Fig. 8. Cutting performance of NPD, SCD (type 1b) and PCD cutting tools in cutting of Al alloy

4-2 Machining of ZnS ceramic Fresnel lenses

The sharp-edged NPD cutting tool that has a 45° pointed angle with 2 μm chamfer width was produced precisely (Fig. 9) to conduct a cutting test on Fresnel lenses made of ZnS ceramics. The ZnS lens that was 20 mm in diameter was rotated with 2000 rpm for a turning test. The cutting conditions were $A_p = 0.01$ mm and $f = 0.007$ mm/rev, and an ultra high-precision lathe was used.

Figure 10 shows the results of cutting tests, indicating the cutting performance of the NPD tool far beyond that of the SCD tool. In this turning test, the cutting surface roughness of the SCD tool deteriorated to 0.05 μm or higher, indicating the end of the tool life, after machining 5 lenses; in the case of the NPD tool, on the other hand, the roughness of cutting surface remained less than 0.02 μm after machining 20 lenses.

High resolution scanning electron microscope (SEM) observation of diamond tools after the turning test revealed the difference of tool wear between NPD and SCD. The SCD tool lost its edge configuration due to uneven abrasive wear. Since abrasive wear of SCD progresses due to the accumulation of micro-cleavages, there may be the anisotropic feature in the abrasive wear of SCD. This leads to an uneven abrasive wear of SCD.

On the contrary, the edge configuration of the NPD tool was retained even after machining 20 lenses. It is considered that satisfactory machining surface conditions

were constantly attained because NPD has high abrasive wear resistance in any direction and the tool edge shows little sign of losing shape.

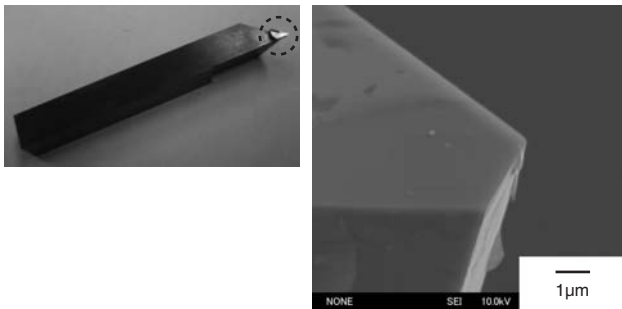


Fig. 9. NPD tool for high precision cutting

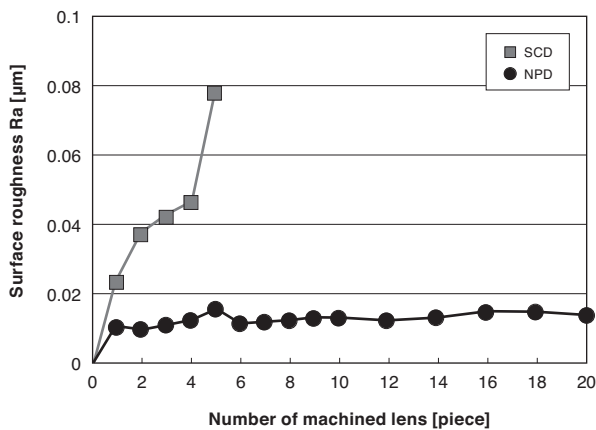


Fig. 10. Cutting performance of NPD and natural SCD cutting tools in high precision cutting of ZnS ceramics lens

4-3 Turning of cemented carbide

To confirm cutting performance of the NPD tool on cemented carbide for general-purpose metal molds (WC-7% Co, grain size: 2 μm), turning evaluation was made under the condition of $V_c = 20$ m/min, $A_p = 0.05$ mm and $f = 0.1$ mm/rev, with a 0.4 mm nose radius tool edge.

The results are shown in Fig. 11. On the SCD tool, breakage and chipping damages on the tool edge due to cleavages occurred in the early stage as after cutting to 20 m, while the NPD tool did not show remarkable damage after cutting to 280 m. It was ascertained that abrasive resistance of the NPD tool was more than five times higher than that of PCD tool B whose average grain size was 5 μm, and more than three times higher than that of PCD tool C whose average grain size was 30-50 μm.

Next, in order to confirm the potential of NPD for precision cutting compared with synthetic SCD (type 1b), an NPD tool with a V-shaped sharp edge (45° pointed angle) was prepared.

The cutting tests were demonstrated on ultra-fine-

grained cemented carbide (WC-12% Co, grain size 0.3 μm) by edge-face turning, under the condition of $V_c = 9.6$ -5.2 m/min, $A_p = 1.0$ μm and $f = 0.5$ μm, using an ultra precision lathe. Fig. 12 shows the cutting edge configurations before and after cutting tests. The SCD tool was degraded by chipping due to cleavages in the early stage, while the NPD tool worn only at the tip of the edge without remarkable chipping damage. The NPD tool was capable of machining V-shaped grooves more than double the distance that the SCD tool can groove.

Thus, it was confirmed that NPD has excellent wear and fracture resistance against common cemented carbide, compared with conventional PCD and SCD.

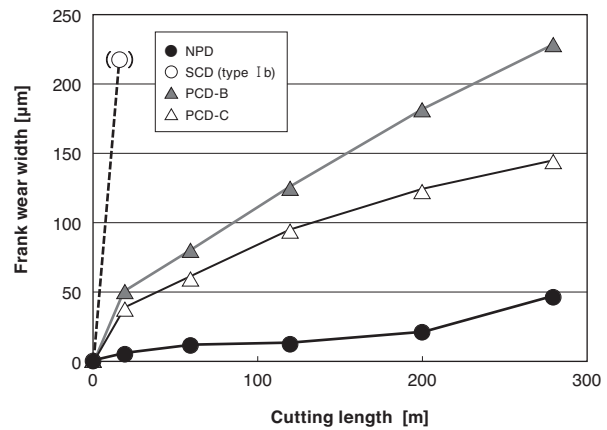


Fig. 11. Cutting performance of NPD, SCD (type 1b) and PCD (B: 5μm, C: 30-50μm) cutting tools in cutting of cemented carbide

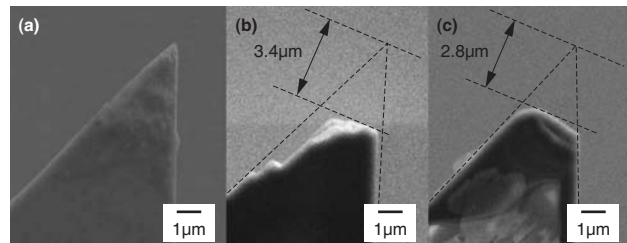


Fig. 12. Cutting edges after high precision cutting of cemented carbide
a) NPD : before cutting
b) SCD (type 1b) : after 400m cutting
c) NPD : after 800m cutting

4-4 High precision cutting of binder-less cemented carbide

A V-shape grooving test was conducted with an ultra-precision nano machine on binder-less fine-grained cemented carbide simulating a light guide plate. The binder-less cemented carbide used as mold material for glass forming is too hard (Vickers hardness of 23-25 GPa) to be cut precisely. Therefore, it has been machined with diamond grinding wheels and then polished. As it is very difficult to machine fine grooves with the diamond grinding wheel, it has been desired to be finely finished by high precision cutting.

The V-shaped precision NPD tool with a pointed angle of 90° and a rake angle of -20° was used for this test. The cutting conditions were $V_c = 10$ mm/min, $A_p = 0.3$ μm and $f = 2$ μm , and each groove was machined by 4 times to form 1.2 μm depth.

Fig. 13 shows the tool edges after 30 grooves machined. It indicates that the NPD tool has no remarkable damage, while the SCD tool is seriously damaged.

As described above, it was confirmed that the NPD tool has excellent cutting performances compared to the SCD tool even on binder-less cemented carbide.

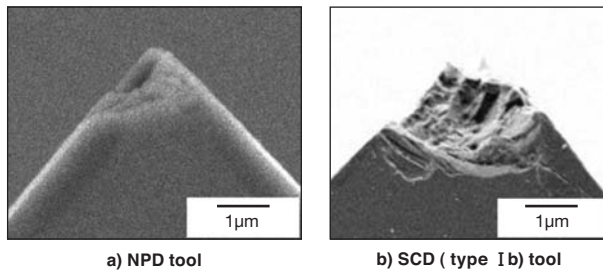


Fig. 13. Cutting edges after high precision grooving of binder-less cemented carbide
a) NPD tool
b) SCD (type Ib) tool

5. Conclusions

The mechanical properties of NPD derived from direct conversion sintering of graphite under ultra-high pressure and temperature (14-18 GPa, 2100-2300°C) were evaluated. It was confirmed that NPD exhibits excellent properties far superior to those of PCD and SCD in terms of hardness, abrasive resistivity and heat resistance which are important in cutting tool applications.

In addition, cutting tests were conducted using prototype NPD tools and various work materials under different conditions. Consequently, it was confirmed that NPD tools can be used in a wide variety of applications for which PCD and SCD have been previously used or conventional tools have not been able to be applied.

Moreover NPD cutting tools are expected to be used for large-area machining owing to their long tool life. The cutting capability of NPD tools also enables high-precision cutting of hard brittle materials, such as cemented carbide, which has been difficult with conventional SCD cutting tools.

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