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Hydrothermal-Reaction-Assisted Laser Machining of Cubic Boron Nitride

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Although the high hardness and chemical stability of cubic boron nitride (cBN) complicate its machining, the mass decrease of cBN in a steam environment at high temperature has been reported. In this study, we investigated hydrothermal-reaction-assisted laser drilling of cBN in various environments. A single-crystalline cBN grain, binder-containing sintered cBN, and binderless sintered cBN were irradiated with an Ar ion laser in water and steam, and in gas atmospheres. The cBN reacted with water, and NH_4^+ or NH_4^+-N and boric acid were produced. Hydrothermal-reaction-assisted machining was not effective for binder-containing sintered cBN, but was effective for single-crystalline cBN and binderless sintered cBN.

I. Introduction

Cubic boron nitride (cBN) is a material offering many interesting properties, such as chemical inertness, low density, thermal conductivity, and high hardness, that are inferior only to diamond. As a hard material for machining applications, cBN is superior to diamond because of its low solubility in ferrous metals and higher resistance to oxidation.[1] cBN has been synthesized at high pressure and high temperature. Furthermore, sintered cBN is now widely used in machining tools. Much research on cBN film deposition by chemical vapor deposition has also been reported,[2] and its applications as a coating of machining tools has been expected. Machining is essential for application to machining tools, but the high hardness and chemical stability of cBN complicate the machining process. However, the mass decrease of cBN in a steam environment at high temperature has been reported,[3] and this suggests that the acceleration of cBN laser machining can be expected in steam or in water.

Much research on ceramic machining using lasers in water has been reported, such as on laser-enhanced etching,[4] ablation rate enhancement in water by excimer laser irradiation,[5] and prevention of recast layer and crack formation.[6]

However, laser machining of ceramics using a laser in combination with hydrothermal reaction has not been reported until now. In this study, we investigated hydrothermal-reaction-assisted laser machining by laser irradiation of single-crystalline cBN, binder-containing sintered cBN, and binderless sintered cBN in various environments.

II. Experimental Procedure

(1) Thermogravimetric Analysis

Single-crystalline cBN (SBN-M, Showa Denko K.K., Tokyo, Japan) synthesized at high pressure and high temperature was used (Fig. 1). Grain size was approximately 400 μm. Thermogravimetric analysis was performed in steam (90% RH at 363 K), air (40% RH at 293 K), and argon heated to 1673 K to reveal the influence of the atmosphere on cBN. Heating rate was 10 K/min. Gas flow rate was set at 0.3 L/min.

(2) cBN Sample

The cBN grain was electrodeposited on a copper disk (10 mm in diameter), because the grain size of cBN is approximately 400 μm, which is too small to handle directly. After the electrodeposition, cBN was lapped with diamond powder (grain size: 20 μm). Therefore, the crystal plane was disregarded. Two types of sintered cBN, binderless sintered cBN (Sumitomo Electric Industries Ltd., Osaka, Japan: cBN > 99.9 vol%), and binder-containing sintered cBN (AMB90, Element Six Ltd., Tokyo, Japan: cBN approximately 90%; the rest was aluminum binder, cBN grain size: 8–10 μm), were used.

(3) Laser Irradiation

A laser beam that is not absorbed by water is required to process samples immersed in water, because the laser beam must penetrate through water and reach the cBN. Furthermore, sufficient power for heating in water is required. Therefore, a multiline argon ion laser beam (Innova Sabre TSM-20, Coherent Inc., Santa Clara, CA; maximum power: 24 W, beam mode: TEM_{00}) with wavelengths of 455–529 nm was used. The laser beam was focused by a convex lens, with a focal length of 25 mm, to a spot size of approximately 80 μm.

Irradiation was examined in water and steam, as well as in various gas atmospheres (air, vacuum (3–5 kPa), argon (101 kPa), and oxygen (101 kPa)). Figure 2 shows the experimental setup. The cBN samples were placed in a chamber, as shown in Fig. 2(a), in the case of gas atmospheres. The setup for irradiation in water is shown in Fig. 2(b). Water was injected from the nozzle in order to remove bubbles and misty particles produced during the irradiation process. Furthermore, a glass window was located at the water surface to prevent laser beam dispersion by the undulation of the water surface. The cBN sample was placed 2 mm below the glass window. Steam atmosphere was created

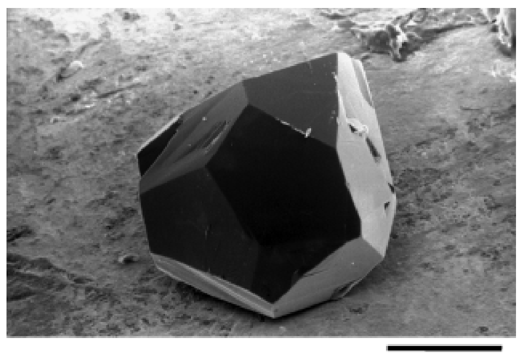

Fig. 1. Scanning electron microscope image of cubic boron nitride before plating.

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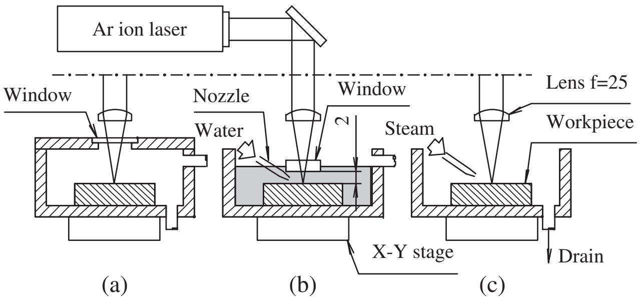

Fig. 2. Experimental setups for Ar ion laser machining in various atmospheres.

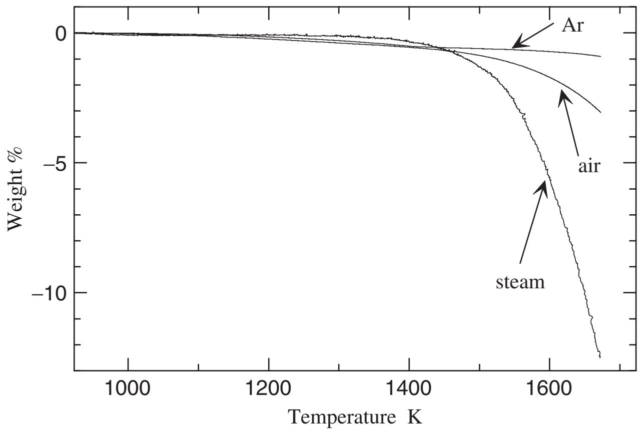

Fig. 3. Results of thermogravimetric analysis of cubic boron nitride.

by steam injection, as shown in Fig. 2(c). Water was boiled at atmospheric pressure and the steam was conducted through the nozzle.

Contamination on the binder-containing sintered cBN was analyzed with energy-dispersive X-ray spectroscopy (EDS; JSM-6301F, JEOL, Tokyo, Japan). After the laser irradiation, gold was evaporated on the cBN to prevent charging, and then the cBN was analyzed. Acceleration voltage was set at 15 kV.

III. Results and Discussion

(1) Thermogravimetric Analysis

Figure 3 shows the mass decrement with heating. In argon, the mass decrement was below 1%, even with heating to 1673 K. In air, the mass was decreased slightly from around 1550 K, and was approximately 3% at 1673 K. On the other hand, in steam, the mass decreased from 1450 K, and the mass decrement was 13% at 1673 K in steam. The mass decrement in steam was much faster than those in air and argon. This result revealed that cBN reacts with steam, and therefore the acceleration of cBN laser machining is expected in steam.

(2) Laser Machining

(A) Scanning Electron Microscope (SEM) Observation: SEM micrographs of laser-irradiated cBN are shown in Fig. 4 (laser power: 24 W, irradiation time: 30 s). In air, the irradiated area becomes slightly dented, and the center of the dented area is smooth. In vacuum, there are contaminants around the irradiated area, and pits can be recognized around the irradiated area. In argon, there is fibrous contamination in the irradiated area. The contaminants could be easily removed by rubbing with tweezers. In oxygen, no contamination is seen, and a deep hole is drilled. In water, the diameter of the hole is smaller than that in the other cases, and no contamination is observed. In steam, a deep hole is formed, and radial slots are recognized around the irradiated area.

The surface profile of the irradiated area was measured with a profile micrometer (VF-7500, Keyence, Osaka, Japan), and the depth is shown in Fig. 5. The depth of the hole machined in steam was more than 180 μm, and the depth could not be measured accurately because it was beyond the limit of the profile micrometer.

Laser irradiation with various laser powers was examined in each atmosphere (irradiation time: 30 s). The cBN was drilled with laser powers above 15 W in air and oxygen, but in the other

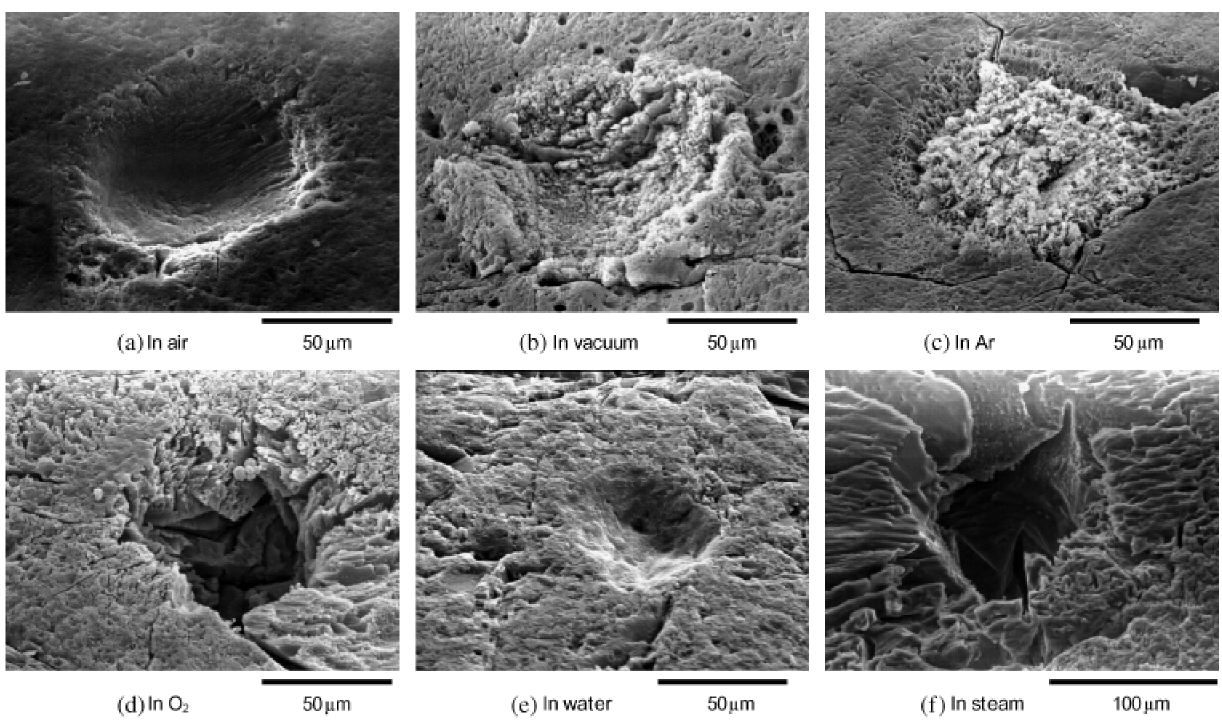

Fig. 4. Scanning electron microscope micrographs of cubic boron nitride after Ar ion laser machining in various atmospheres. Laser power: 24 W, irradiation time: 30 s.

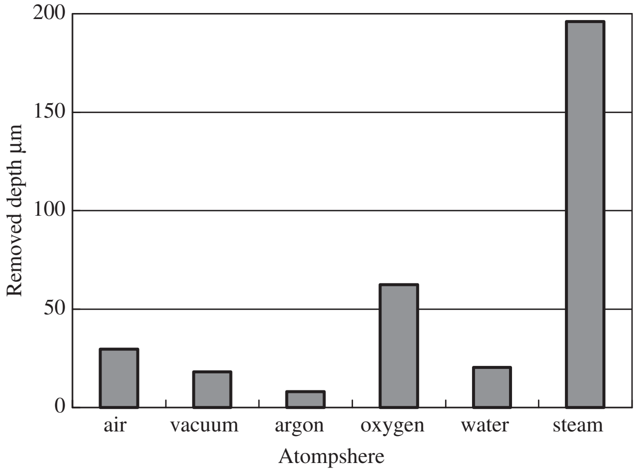

Fig. 5. Depth of holes in cubic boron nitride formed by Ar ion laser machining in vacuum (3–5 Pa), air, argon (101 kPa), oxygen (101 kPa), water (293 K), and steam (373 K). Laser power: 24 W, irradiation time: 30 s. The depth of the hole in the steam was beyond the limit of the measuring device and was more than 180 µm.

environments, no change was recognized below 20 W. In water and steam, cooling by water and steam should prevent the decomposition of cBN at low laser power.

We hypothesized that the diameter of the hole drilled in streaming water was small because cBN was strongly cooled by the water. In order to reveal the effect of cooling by water, the laser was irradiated in static water without water injection or the glass window. As a result, a deeper and larger hole was drilled (approximately 100 µm in diameter, 60 µm in depth). The dispersion of the laser beam owing to undulation of the water surface enlarged the diameter. However, the depth was three times deeper than that in the case of steaming water. Low heat conductivity in steam enables deep drilling. Furthermore, heating around the laser-irradiated area may induce the cBN to react with steam, causing the generation of the slots.

Humidity in the atmosphere could be the cause of the difference in the drilled depth in air, argon, and vacuum. The cBN samples were irradiated in high (15.6 mg/L, 90% RH at 20°C) and low (4.3 mg/L, 25% RH at 20°C) humidity air, in order to evaluate whether the difference was caused by the moisture in gas or the presence of oxygen. No change was recognized; therefore we concluded that the decomposition was influenced not by humidity, but by the amount of oxygen.

(B) Dissolved Product Detection: The reaction of cBN with water is expected to produce, for example, ammonium and boric acid in water. Water used in laser irradiation was examined. A large amount of injected water made the detection of reaction products impossible, because they are produced only in small amounts in water. Therefore, the laser was irradiated into static water, and then the water was tested by the indophenol method and the azomethine-H method. The water inspection kit (Product No. WAK-NH_4 and WAK-B, Produced by Kyoritsu Chemical-Check Lab., Co., Tokyo, Japan, kyoritsu-lab.co.jp) was applied for the test. The results were both positive. These results showed that cBN reacted with water, producing NH_4^+ or NH_4^+-N and boric acid.

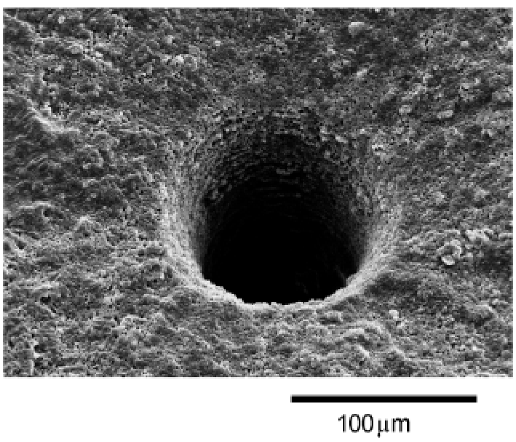

Fig. 6. Scanning electron microscope micrograph of binderless sintered cubic boron nitride after Ar ion laser irradiation in steam. Laser power: 24 W, irradiation time: 30 s.

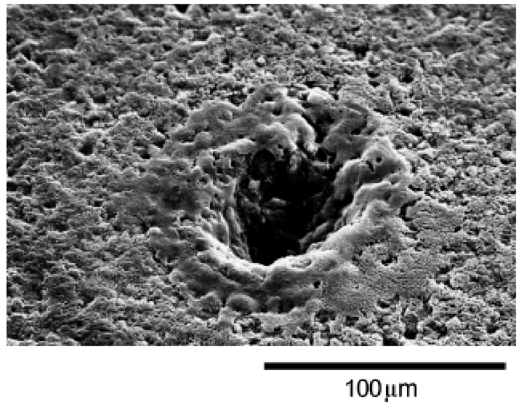

Fig. 7. Scanning electron microscope micrograph of sintered cubic boron nitride after Ar ion laser irradiation in steam. Laser power: 24 W, irradiation time: 30 s.

(C) Cemented cBN: An SEM micrograph of the binderless sample drilled in steam is shown in Fig. 6. The depth of the holes was 180 µm in steam and 100 µm in air .The diameter of holes was approximately 100 µm in both atmospheres. Polycrystalline cBN was drilled faster in steam than in air, similar to the monocrystal. No changes were observed around the hole, unlike the case of monocrystal cBN (slots were seen in Fig. 4(f)).

There was no difference between drilling in steam and in air for binder-containing cBN. Figure 7 shows the results for a laser incident on the sample in steam. The diameter and depth of the hole were 50–60 and 20–25 µm, respectively. Convexity around the hole of the material indicates local melting and resolidification. EDS analysis determined the irradiated area to be aluminum binder. It was considered that contaminants in the hole prevent the hydrothermal reaction and, as a result, no difference is observed between machining in steam and in water.

IV. Conclusion

Hydrothermal-reaction-assisted laser machining by laser irradiation of cBN was investigated. The drilled depth in steam was six times deeper then that in the case of air. The cBN reacted with water, producing $NH4^+$ or NH_4^+-N and boric acid. The hydrothermal-reaction-assisted machining was not effective for binder-containing sintered cBN but effective for single-crystalline cBN and binderless sintered cBN.

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