The influence of sintering parameters on the mechanical properties of vitrified bond diamond tools

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A B S T R A C T

The aims of this study were to investigate the influence of sintering parameters on the mechanical properties of vitrified bond diamond tools. All of the sintered specimens were examined using a thermal analyzer, an X-ray diffraction analyzer, an X-ray photoelectron spectrometer analyzer, a Raman spectrometer analyzer and a scanning electron microscope. The mechanical properties of the sintered specimens including the radial shrinkage percentage, sintered density, hardness and grinding ratio were also recorded. Experimental results showed that the introduction of nitrogen at a sintering temperature of 500 °C could prevent diamond grit from reacting with oxygen and prevent diamond crystals from forming a graphite structure. The microstructure of the sintered specimen 710-90 indicated that the vitrified matrix and diamond grit had excellent coverage and wetting. After the grinding ratio tests, the surface of the vitrified matrix of specimen 710-90 revealed a brittle failure pattern, indicating that the diamond grit could be easily pulled from the vitrified matrix when the diamond grit was worn and the cutting force increased. Therefore, the diamond grit always kept its sharpness and the workpiece maintained the preferred surface roughness. In this study, specimen 710-90 had the best mechanical properties and highest grinding ratio (57.5).

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1. Introduction

The unique physical and mechanical properties of diamonds make them the preferred choice in a range of applications, including those in electronics, precision optics, precision machinery, automobiles, biomedical, national defense, earthworks and aerospace [1–6]. Diamond tools are a group of composite materials composed of diamond grit and a bonding matrix. Familiar bonding materials include metals, resins, vitrified glass, and electroplating. As vitrified bond diamond tools can be densified by pressureless sintering at low temperatures, pores of different sizes can be introduced into the tool structure [3,4,7,8]. Such a structure can possibly reduce the frictional heat generated during working by reducing the contact area and by allowing for the flow of cooling fluid into the pores [8–10].

The vitrified matrix should provide a suitable brittleness, thus when the diamond grit is worn and the cutting stress increases, the bond can break and release the worn diamond grit from the matrix. In other words, vitrified bond diamond tools should possess an excellent capacity for self-dressing. To improve the bonding

force between the diamond grit and vitrified matrix, a suitable matrix toughness and hardness are required. A good wetting ability, gradual chemical reactions, and similar thermal expansion coefficients between the diamond grit and the matrix are also required [9,10]. Uniform pore distribution within the vitrified matrix is very important for chip removal and reduction of frictional heat generated during the cutting process. Additionally, reduction of the sintering temperature and introduction of a protective atmosphere are considerably important during the sintering process in order to prevent the graphitization of diamonds due to high temperatures, which would result in decreased performance of the diamond tools [11–14].

In this study, a borosilicate glass and diamond grit were selected to make vitrified bond diamond tools, and various sintering parameters were used. All of the sintered specimens were examined by thermal analysis, X-ray diffraction (XRD) analysis, X-ray photoelectron spectroscopy (XPS) analysis, Raman spectroscopy analysis, and various mechanical performance tests. The aims of this study were to clarify the influence of sintering parameters on the mechanical properties of vitrified bond diamond tools and to provide optimal sintering parameters for vitrified bond diamond tools.

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Table 1
Composition of glass powder used in this study.

SiO$_2$	B$_2$O$_3$	Na$_2$O	CaO	ZnO
>40.0%	10.0–40.0%	3.0–10.0%	3.0–10.0%	3.0–10.0%
Al$_2$O$_3$	K$_2$O	MgO	BaO	LiO$_2$
3.0–10.0%	0.1–3.0%	0.1–3.0%	0.1–3.0%	0.1–3.0%

Table 2
Nine sintering parameters for the specimens.

Isothermal holding duration (min)	690 °C	710 °C	730 °C
60	690-60	710-60	730-60
90	690-90	710-90	730-90
120	690-120	710-120	730-120

2. Experimental procedure

The composition of the specimens used in this study had a 1:3:1 volume ratio of diamond grit, glass, and paraffin wax. The paraffin wax was used to lubricate each specimen during the die-pressing process. The mean particle size of the diamond grit was 30.7 μm (GE, FM 30–40) and the glass powder was 16.7 μm (China Glaze, CT-1124). Table 1 shows the composition of the glass powder. The diamond grit and glass powder mixture were first mixed with 20 vol% paraffin wax, which was dissolved in heptane, and then blended in a plastic jar using tetragonal zirconia polycrystal balls. The milled powder slurry was heated at 60 °C for 90 min to remove the heptane, and granules were formed by sieving the dried mixture by using a 100-mesh sieve. After the granule-forming process, the powders were then die-pressed into the specimens. Round-shaped specimens, having 3 mm thickness and outer diameters of 12 mm and 30 mm, respectively, were die-pressed with a pressure of 30 MPa. The specimens were then placed on a high-density graphite plate and sintered in a tube furnace with various temperatures and isothermal holding times. Table 2 shows nine sintering parameters for the specimens.

The thermal profile was composed of heating at 5 °C/min to 250 °C, a temperature which was then maintained for 30 min to burn off the paraffin wax, followed by a continuing rise in temperature of 3 °C/min to 620 °C, a temperature which was then held for 60 min. This schedule was followed directly by heating at 1 °C/min to 690 °C, 710 °C, or 730 °C, with each of those temperatures held for 60, 90, or 120 min. All the specimens were furnace-cooled. An air atmosphere was maintained until isothermal holding at 500 °C. Then, at that temperature, the atmosphere was changed to nitrogen to protect the diamond grit from oxidation.

The thermogravimetric analyzer (TGA, TA Instrument Thermal Analysis Q500) carried out thermal analysis for the glass powder and diamond grit. A heating rate of 5 °C/min, from room temperature (R.T.) to 1000 °C was executed. A differential scanning calorimetry test (DSC, HT-DSC, Netzsch Instrument 404) was carried out for the glass powder in order to find out its glass transition temperature. A heating rate of 5 °C/min from R.T. to 900 °C was executed. Sintered specimens were also examined by XRD analysis. The X-ray diffractometer (Shimadzu, XRD-6000) used 30 kV voltage and 30 mA current, diffraction angles of 20°-100°, a scanning speed of 5°/min, and a scanning interval of 0.03°.

Raman spectra for diamond grit were analyzed for C—C bond vibrations signals of the sintered specimens to investigate whether the diamond crystals changed their structure after the high temperature sintering process. The wavelength of the excitation laser of the Raman spectra was 780 nm (Lambaba Solution, P2). Chemical bonding between the diamond grit and the vitrified matrix was investigated using X-ray photoelectron spectroscopy (XPS). The target of the XPS (Ulvac-Phi, PHI 5000 Versa Probe) used Al mono, and the X-ray energy was 187.85 eV. Secondary electron images (SEIs) of the sintered specimens and grinding test specimens were examined using a scanning electron microscope (SEM, Jeol, JSM-6390LV). The working voltage of the SEM was 5 kV.

The radial shrinkage percentages for the sintered specimens were calculated by averaging five measurements taken using a digital vernier caliper. Sintered density was calculated using the Archimedes method and each specimen was measured five times and then values were averaged. The hardness of the sintered specimen was measured using a micro Vicker's hardness tester (Makazawa, HM-221) under a load of 2kgf for 10 s. The average of five tests was recorded. Grinding ratio tests for the sintered specimen were performed using a lathe at a spindle speed of 1800 rpm. A cemented carbide spindle with a diameter of 4.5 mm (WC87%–Co13%, density 14.18 g/cm^3) was used for the workpiece; the depth of cut was 0.01 mm/pass at a feeding speed of 0.0125 mm/s, and water was used as the cooling medium. A 3D surface roughness measuring instrument (Kosaka, SEF-3500) was used to measure the surface roughness of the workpiece after the grinding tests. Center line average roughness (Ra) was used and 3D scanning images of the workpieces were illustrated.

3. Results and discussion

3.1. Thermal analysis

TGA analysis of the diamond grit showed no weight loss from 25 °C to 600 °C; however, a rapid weight loss was observed between 630 °C and 725 °C due to the fact that diamonds react with oxygen [15–18]. TGA analysis of the glass and paraffin wax showed a weight loss between 180 °C and 350 °C. A differential of TGA data exhibited that the volatilization temperature of paraffin was around 204 °C. DSC analysis showed that the glass transition temperature (Tg) for the borosilicate glass powder used in this study was approximately 716 °C.

3.2. XRD analysis

The specimens sintered at different temperatures for the same isothermal holding time of 90 min (690-90, 710-90, and 730-90), and those with the same sintering temperature of 710 °C but different isothermal holding times (710-60, 710-90, and 710-120) were examined by XRD. All XRD patterns showed three diffraction peaks, the 2θ angles were 44.3°, 75.5°, and 91.7°, respectively, which approach the diffracted angles of (111), (220), and (311) planes of diamond [18,19]. Therefore, within the experiment range of this study, no phase other than diamond was observed after changing the sintering temperature and isothermal holding time.

3.3. Raman spectroscopy analysis

Fig. 1(a) shows the Raman spectra for specimens 690-90, 710-90, and 730-90, respectively. A sharp peak at 1333.4 cm^{-1} can be observed which belongs to the crystalline diamond C—C bond D-band signal. This indicated that the analyzed object was a diamond with the SP^3 structure [20,21]. The amorphous carbon generated on the diamond surface and the changed diamond crystal was possibly due to the increase in sintering temperature or isothermal holding times. Fig. 1(b) shows that the Raman spectra of the specimens with different isothermal holding times (710-60, 710-90, and 710-120) were similar; no G-band signals belonging to the SP^2 graphite structure were detected. It is particularly noteworthy that the intensity of D-band signal decreased with the increase in sintering temperature or isothermal holding,