



Contents lists available at ScienceDirect

International Journal of Refractory Metals & Hard Materials

journal homepage: www.elsevier.com/locate/IJRMHM

Effects of added nano titanium on the microstructure and mechanical properties of vitrified bond diamond tools

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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 applications involving precision machinery, electronics, national defense, and aerospace [1–7]. Since pores can be introduced into the matrix structure easily, vitrified bond diamond tools provided with an excellent capacity for chips removal and cooling. For this reason, such tools possess an outstanding grinding ratio and the workpiece maintained the preferred surface roughness [6–10]. However, synthetic diamond grits usually contain active metal components that can cause the diamonds to be easily catalyzed and transformed from sp^3 structures into sp^2 graphite via sintering, consequently decreasing the cutting performance of vitrified bond diamond tools. The improve technique of above phenomenon at present including: reduction of the sintering temperature, decrease isothermal holding, introduce of a protective atmosphere and added elements for protect diamond grits [11–15]. However, reducing the sintering temperature and decreasing the isothermal holding result in weakened bonding between the diamonds and vitrified matrix, in addition to lowering the grinding ratio of the tools. As for the use of a protective atmosphere during the sintering process, this approach can increase the cost of manufacturing the tools [6,7,11–13].

A high temperature sintering process can cause the thermal degradation of diamond grits, which would result in decreased cutting performance of the diamond tools while simultaneously causing uncontrolled expansion and numerous irregular pores in the composites. Previous study indicates Ti powder was used as oxygen getter due to its excellent affinity with oxygen. The results showed that diamond grits received good protection from oxidation during sintering due to the prior reaction of Ti powder with oxygen. In addition, TiO_2 , an oxidation product of Ti, could enter into the vitrified matrix, and decrease the volume changes that the tools during sintering process and strengthen the matrix [14].

The main focus in this study was investigating the addition of various volume ratios of nano Ti to vitrified bond diamond tools. The sintering temperature was 710 °C and isothermal for 90 min. Air and

nitrogen, respectively, were used during the sintering. All of the sintered specimens were examined using an X-ray diffraction (XRD) analyzer, a Raman spectrometer, and a scanning electron microscope (SEM). Observations of the microstructure and tests of the mechanical properties of the sintered specimens were carried out. More specifically, the effects of the additions of nano titanium on the microstructure and mechanical properties of the vitrified bond diamond tools were investigated. Finally, according to the analytical results, an appropriate amount of nano Ti to add to vitrified bond diamond tools is suggested.

2. Experimental procedure

Table 1 shows the composition of the glass powder (China Glaze, CT-1124) used in this study. The mean particle sizes of the glass powder and diamond grits (GE, FM 30–40) were 20.5 μm and 30.7 μm , respectively. The mean particle size of the nano Ti (Bojun Co., WU-Ti-001, density 0.19 g/cm^3) was 40 nm. Fig. 1 shows SEM images of the glass powder and diamond grits. The composition of the specimens used in this study had a 1:3:1 volume ratio of diamond grits, glass, and paraffin wax. The compositions and designations of the specimens are shown in Table 2, with the added amounts of nano Ti ranging from 0 to 50 vol%.

The glass powder was first added with a 20 vol% paraffin wax, which was dissolved in heptane, and then blended in a plastic jar. The powder slurry was heated at 40 °C to remove the heptane, and then mixed with various proportions of glass powder, diamond grits, and nano Ti in a dried condition according to the experimental design. After the mixing process, the powders were then die-pressed into the specimens. Round-shaped green specimens; having 3 mm thickness and outer diameters of 12 mm was die-pressed with a pressure of 30 MPa for 3 min.

The green specimens were sintered in a tube furnace. The thermal profile was composed of heating at 5 °C/min to 250 °C, with that temperature then maintained for 30 min to burn off the paraffin wax, followed by further heating at 3 °C/min to 620 °C, with that temperature then maintained for 60 min. This schedule was followed directly by heating at 1 °C/min to 710 °C, with that temperature then maintained

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Received 3 January 2018; Accepted 15 March 2018

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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%

for 90 min. All the specimens were furnace-cooled. An air was maintained until 500 °C for the clean of the tube furnace, then specimens sintering at atmosphere. Only the specimen designated as 0 N was sintered in a nitrogen atmosphere at the temperatures above 500 °C. This was done to investigate the effect of using nitrogen to protect the diamond grit from oxidation during the sintering process.

A thermogravimetric analyzer (TGA, TA Instrument Thermal Analysis Q500) was used to carry out thermal analyses of the glass powder and diamond grits. A heating rate of 5 °C/min, from room temperature to 900 °C was executed. A differential scanning calorimetry (DSC, HT-DSC, Netzsch Instrument 404) test was carried out for the glass powder in order to find out its glass transition temperature. A heating rate of 5 °C/min from 25 °C to 900 °C was executed. The sintered specimens were also examined using an X-ray diffractometer (XRD). The XRD (Shimadzu, XRD-6000) used a voltage of 30 kV, current of 30 mA, diffraction angles of 20° to 100°, a scanning speed of 5°/min, and a scanning interval of 0.03°. A Raman spectrometer (Lambaba Solution, P2) was used to analyze the C–C bond vibration signals of the diamond grits in the sintered specimens to investigate whether the diamond crystals changed their structure after undergoing the sintering process. The wavelength of the excitation laser of the Raman spectrometer was 780 nm. 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 30 kV.

The radial shrinkage percentages for the sintered specimens were calculated by averaging five measurements taken using a digital Vernier caliper. The sintered density of each specimen was calculated using the Archimedes method, with each specimen being measured five times and the values then being averaged. The hardness of each sintered specimen was measured using a micro Vickers hardness tester (Makazawa, HM-221) under a load of 19.6 N for 10 s. The average of five tests was recorded. Grinding ratio tests for the sintered specimens 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/cm³) 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 Laboratory Ltd., SEF-3500) was used to measure the surface roughness of the workpiece after the grinding tests. Center line average roughness

Table 2
Compositions and designations of specimens.

Specimen number	Diamond (vol%)	Glass powder (vol%)	Paraffin wax (vol%)	Ti (vol%)	Ti (wt%)
0 N	20	60	20	0	0
0 T	20	60	20	0	0
6 T	18.8	56.4	18.8	6	0.6
10 T	18	54	18	10	1.0
20 T	16	48	16	20	2.1
30 T	14	42	14	30	3.6
40 T	12	36	12	40	5.4
50 T	10	30	10	50	7.9

(Ra) was used and 3D scanning images of the workpieces were illustrated.

3. Results and discussion

3.1. Thermal analysis

The TGA results for the diamond grits showed no weight loss from 25 °C to 600 °C; however, a rapid weight loss rate of 1.39%/°C was observed between 633 °C and 695 °C due to the fact that diamonds react with oxygen, with the total weight loss being close to 100% at the temperature of 850 °C [13–15] (Fig. 2(a)). The TGA results for the glass and paraffin wax showed an endothermic peak at the temperature of 203 °C, with that peak indicating the volatilization temperature of the paraffin. The DSC test results showed that the glass transition temperature (T_g) for the borosilicate glass powder used in this study was approximately 716 °C (Fig. 2(b)).

3.2. X-ray diffraction analysis

The sintered specimens (0 T, 10 T, 30 T, and 50 T) to which different volume ratio percentages of nano Ti were added were examined using the XRD (Fig. 3). All the X-ray diffraction patterns showed three diffraction peaks, the two theta angles were 43.9°, 75.3°, and 91.5°, respectively, which approach the diffracted angles of (111), (220), and (311) planes of diamond crystal (JCPDS 65-0537) [13,16] (Fig. 3).

The sintered specimen with an added amount of 50 vol% nano Ti (50 T) was observed to exhibit a rutile TiO₂ phase diffraction pattern. The two theta angles were 27.4°, 36.1°, 41.3°, and 54.4°, respectively, which approached the diffracted angles of (110), (101), (111), and (211) planes of rutile TiO₂ (JCPDS 89-0552). The diffraction peaks of rutile TiO₂ revealed a broad pattern. These results suggested that TiO₂ phase belong to nano scale, and composed of irregular polycrystalline [17,18]. The sintered specimen with an added amount of 30 vol% nano Ti (30 T) was also observed to exhibit a rutile TiO₂ phase diffraction

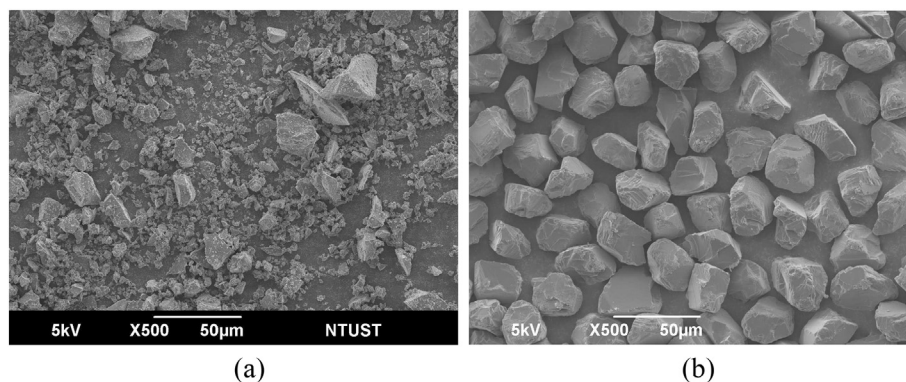


Fig. 1. SEM images of the: (a) glass powder and (b) synthetic diamond grits used in this study.

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