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Effects of Binder Composition on the Mechanical Properties of CVDD/PCD Superhard Composite

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Abstract: ***This study prepared superhard composite consisting of CVDD (Chemical vapor deposition diamond) wrapped by PCD (Polycrystalline diamond) through high-temperature and high-pressure (HPHT) sintering, with Ni-Si-B as the binder for the PCD sintering. The effects of different Ni, Si, and B ratios on the properties and microstructure of the composite materials were investigated through hardness testing, wear ratio testing, and scanning electron microscopy (SEM). The results indicated that Ni and B can provide favorable liquid-phase sintering conditions during the sintering process of polycrystalline diamond, mitigating the detrimental effects associated with the brittleness of silicon carbide (SiC). However, as the Ni content increases, the hardness of the outer PCD layer decreases, and the overall performance of the CVDD/PCD composite declines. The composite exhibited optimal overall performance when the binder content of Si, Ni, B were 9.4wt%, 0.5wt%, 0.1wt%. This study provides a foundation for the research and application of CVDD/PCD composite in hard rock drilling.***

Keywords: Superhard composite, Polycrystalline diamond, CVD diamond, Mechanical properties.

1. Introduction

Polycrystalline diamond (PCD) is a composite material synthesized under high-pressure and high-temperature (HPHT) conditions. During synthesis, the catalytic action of metallic catalysts or phase transformations of ceramic binders facilitate the formation of diamond-diamond (D-D) bonds or diamond-metal-diamond (D-M-D) bonds between diamond particles [1,2]. Its performance was affected by the type and content of the binder [3]. Chemical vapor deposition diamond (CVDD) possesses higher hardness, excellent thermal stability, high thermal conductivity, and a low friction coefficient. Therefore, CVD diamond can serve as hard support points to improve drill bit performance [4-5].

The superhard composite material, formed with CVD diamond as the central core material encapsulated by an outer layer of polycrystalline diamond, combines the excellent properties of both components. Several researchers have investigated the composites with this structure [6-8]. However, studies concerning the influence of the binder composition on the properties remain limited. In this study, CVDD/PCD superhard composite with different binder ratios were prepared (Ni-Si-B as the binder). Their properties were tested, and the results showed that a suitable binder ratio contributes to stabilize the material properties.

2. Experimental

2.1 Sample Preparation

The diamond powders used in this study had particle sizes of 12-22 μm and 0-2 μm. And the binder consisted of silicon (Si), boron (B), and nickel (Ni) powders, all with an average particle size of 1 μm. The dimensions of the CVD diamond were 1×1×4 mm. The CVDD/PCD composite were prepared using a cubic press under sintering conditions of 5.0-5.5 GPa and 1400-1500 °C. The total binder content of each group of samples was 10 wt%, and the proportion of different components in the binder is shown in Table 1. The assembly of HPHT components and image of CVDD/PCD composite were shown in Figure 1.

Table 1: Binder ratios for each group

sample	Si/wt%	Ni/wt%	B/wt%
S_0	10	0	0
S_1	7.5	1.9	0.6
S_2	8.3	1.3	0.4
S_3	9.1	0.7	0.2
S_4	9.4	0.5	0.1

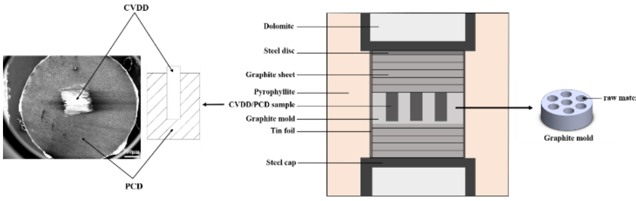

Figure 1: Schematic diagram of HPHT component assembly and CVDD/PCD composite

2.2 Material Performance Testing and Characterization

The formula for relative density (D) was shown in Equation (1), where D_b is the bulk density (g/cm^3) and D_T is the theoretical density (g/cm^3).

$$D = \frac{D_b}{D_T} \times 100\% \tag{1}$$

The formula for hardness (H_v) was shown in Equation (2), where P is the indentation load and d is the arithmetic mean value of the diagonal lengths of the indentation. An indentation load of 4.9 N and the test maintain for15s.

$$H_v = 1.8544 \frac{P}{d^2} \tag{2}$$

The formula for wear ratio was shown in Equation (3), where Ms is the mass loss of the standard silicon carbide grinding wheel, M_j is the mass loss of the CVDD/PCD composite, and K is the correction factor (1.06).

$$E_m = k\frac{M_s}{M_j} \tag{3}$$

3. Results and Discussion

Figure 2 showed the XRD patterns of the raw powder and the sintered sample. No peaks related to boron were detected in the phase composition patterns of sample S1 before and after sintering. This may be attributed to the boron doping concentration being too low, falling below the detection limit of the X-ray diffractometer. Under HPHT conditions, the binder Si reacts with C (diamond) to form SiC phase. Since the hardness of Si was much lower than that of SiC, unreacted silicon and residual graphite will reduce the mechanical properties of polycrystalline diamond. No silicon peaks or graphite peaks were observed in the XRD of sample S0 (Figure 2. (c)), indicating that silicon reacted completely with carbon to form SiC under HPHT conditions. So, the binder content was reasonable. Only the peak of elemental Ni appeared in the XRD of the sintered sample (Figure 2. (d)). Therefore, during the sintering process, Ni only acted to wet the surface of the diamond without reacting with other substances, remaining in the form of elemental Ni.

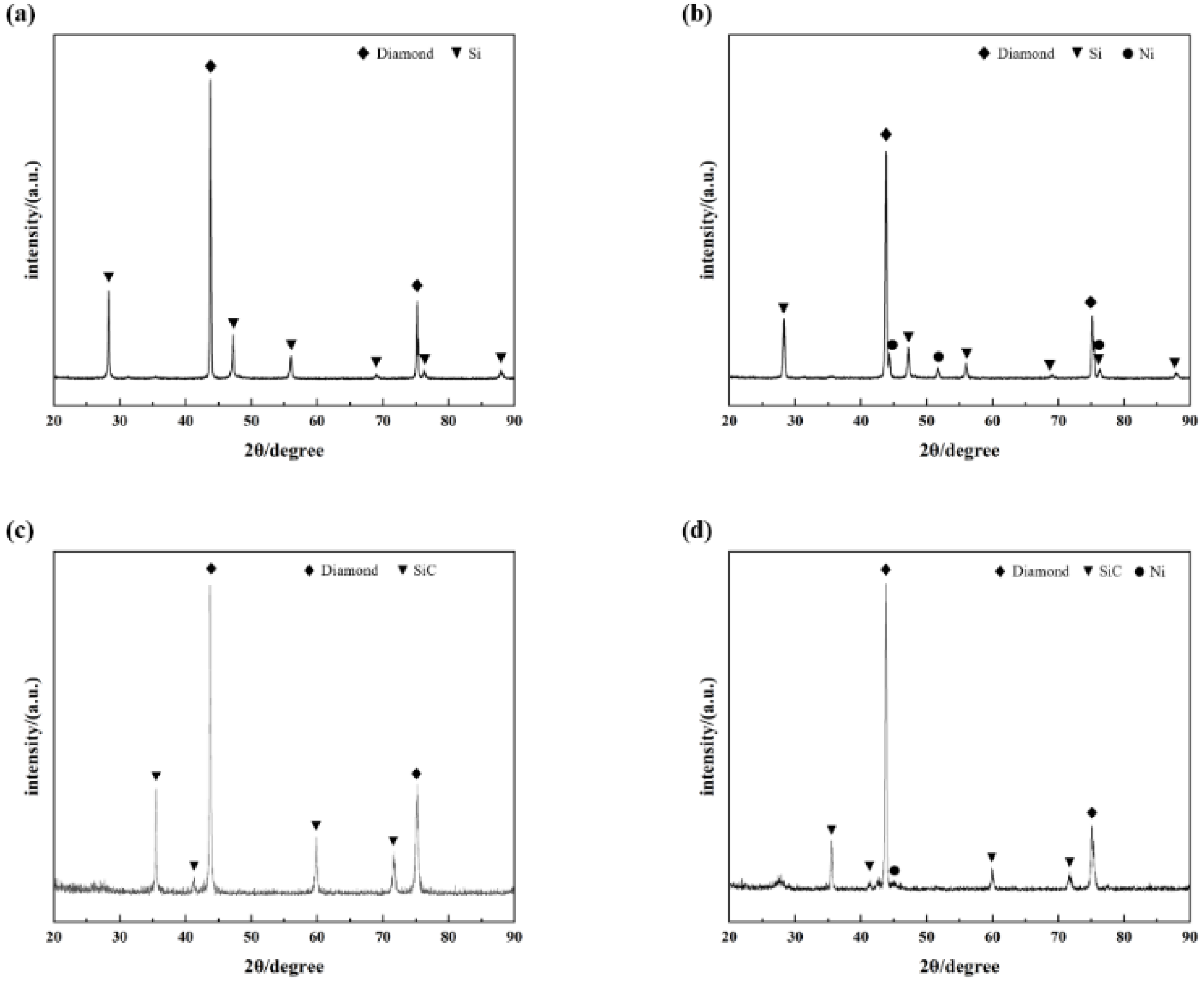

Figure 2: XRD patterns of the powder and the sample: powder (a) S0, (b) S1; sample (c) S0, (d) S1

Table 2: Mechanical properties and the main element ratios of each sample

sample	Relative Density (%)	Hardness (GPa)	Wear ratio ($\times10^4$)	Main element (wt%)			
				C	Si	Ni	B
S0	99.41	50.8	4.28	94.15	5.85	0	0
S1	93.14	-	-	93.37	4.68	1.36	0.60
S2	94.48	-	-	93.79	4.61	1.15	0.45
S3	99.24	45.2	1.01	94.08	5.20	0.72	-
S4	99.33	47.1	2.34	93.24	6.05	0.71	-

Table 2 showed the mechanical properties of the samples. The relative density value was that of PCD prepared under the same conditions, and it decreased with increasing Ni and B content. Although Ni and B can facilitate Si infiltration during sintering, excessive amounts of Ni and B were detrimental to improve the density of the sintered sample. The Vickers hardness indentations of samples S1 and S2 in test were unclear, possibly due to excessive Ni addition resulting in low sample strength. The wear ratio of the samples was closely related to their hardness. Therefore, wear resistance testing was conducted only on S0, S3, and S4. It can be seen that Sample S0 had the highest hardness and wear ratio. Overall, performance of the sample decreased with increasing proportions of Ni and B. To further analyze, the elemental composition of the main elements for each group was obtained through EDS surface mapping, as shown in Table 2.

The PCD of the composite primarily consist of C (diamond), Si, and Ni elements. During the sintering process, Si and Ni melt to form a liquid phase under HPHT conditions, causing the diamond particles to be immersed in the liquid phase and rearranged. The binder uniformly coats the diamond particles and forms a network structure, bonding the diamond particles together as an integrated whole through diamond - metal - diamond (D-M-D) bonding.

Ni and B were added to samples S1-S4, and the Si content was reduced to ensure that the total amount of binder remained unchanged. On one hand, the reduced silicon content resulted in a reduction in the SiC phase, preventing the formation of a well-connected network that wraps and connects the diamond particles. Furthermore, Ni, being a softer phase within the binder with lower hardness than silicon carbide, cannot provide effective support between diamond grains. Consequently, the sample hardness exhibited a trend of increasing as the Ni and B content decreased. Although SiC bonding phase have excellent heat resistance and hardness, their brittleness as ceramic phases is unavoidable. Therefore, we added a small amount of Ni and B to the binder. Nickel has excellent wettability, ductility and toughness, which can prevent sample damage caused by brittleness under impact. So, the brittle fracture was observed during the testing of sample S0, while no significant damage was observed in the other groups (added Ni) before or after testing.

Since S0 has a relatively high wear ratio, further research was conducted on its wear resistance. Five wear resistance tests were performed on sample S0, with the results of 42548, 35012, 50498, 28249 and 47891. This was due to the inherent characteristics of the CVDD/PCD superhard composite. When CVD diamond was used as the main contact point for grinding with the silicon carbide grinding wheel, it achieved a higher wear ratio value. However, the surrounding PCD has a lower hardness than CVD diamond, resulting in a correspondingly lower wear ratio. The performance difference determined that the PCD was worn away before the CVD diamond during the practical use of composite materials. Therefore, CVD diamond will continue to act as a support point on the drill bit, protruding slightly from the surface to perform rock crushing work. The SEM image after wear was shown in Figure 3(a). A layered structure perpendicular to the grinding direction appeared on the surface of the CVD diamond, indicating that brittle fracture occurred in the CVD diamond. Therefore, the control capability of the outer layer PCD over CVD was crucial. In the Figure 3(b), Si and Ni were continuously distributed at the interface between PCD and CVD, and the bonding between PCD and CVD diamond interfaces mainly relies on carbide binder phases between the two. This ensures that during operation, the CVD diamond does not suffer premature wear due to failure of the PCD retention, thereby enabling the CVDD/PCD superhard composite to achieve an effective self-sharpening effect in service.

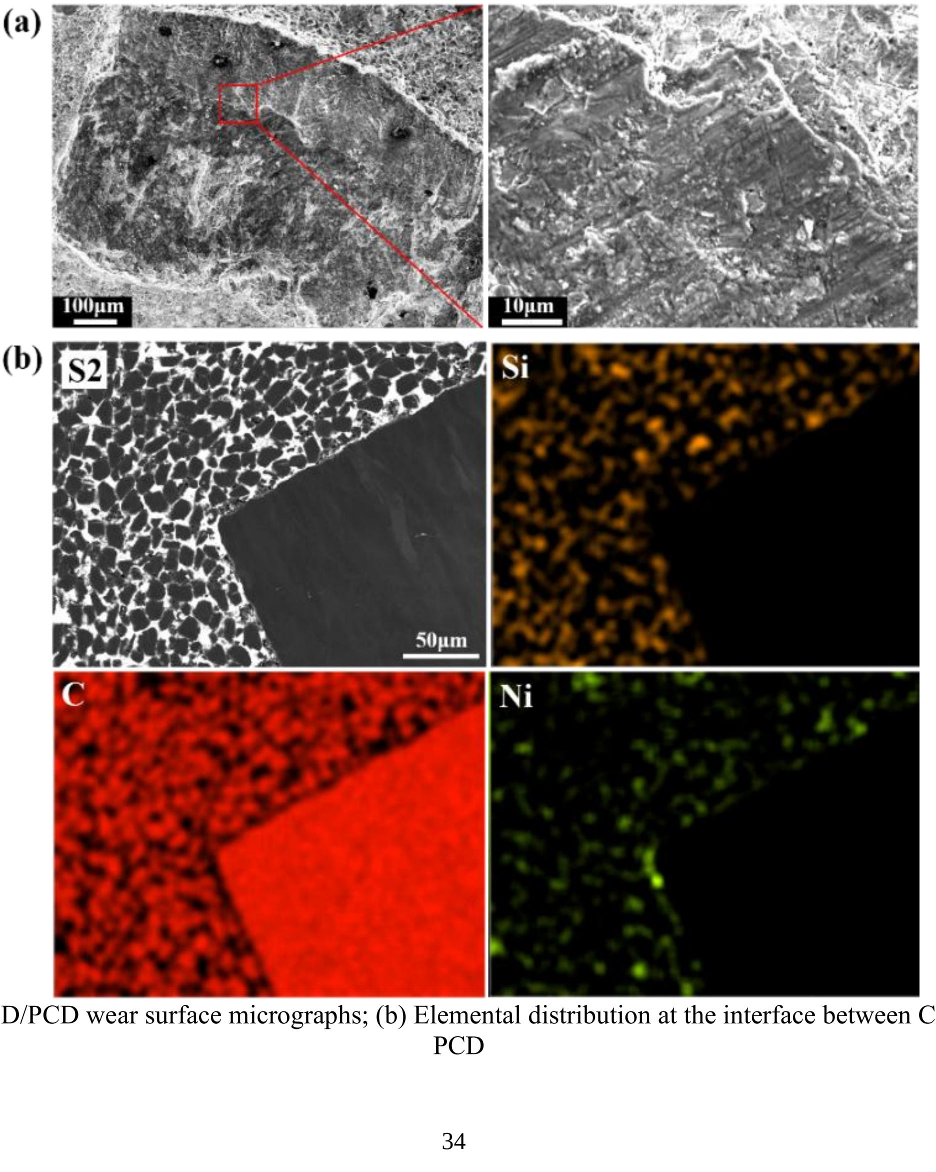

Figure 3: (a) CVDD/PCD wear surface micrographs; (b) Elemental distribution at the interface between CVD diamond and PCD

4. Conclusions

This study investigated the effect of binder content ratio on the performance of CVDD/PCD composite. The results demonstrated that the wear resistance of the CVDD/PCD superhard composite were superior to that of ordinary PCD, and a suitable binder ratio contributes to stabilize the material properties. The following conclusions can be drawn:

1) CVDD/PCD superhard composite with the Ni-Si-B binder system exhibited optimal hardness and wear resistance when the binder content of Si, Ni, B were 9.4wt%, 0.5wt%, 0.1wt%. The microhardness of the sample with this binder ratio was achieved 45GPa, and the density was over 99%. The sample with added nickel and boron did not exhibit brittle fracture.

2) When the Ni and B content was too high, the Si content decreases, and the diamond particles cannot be continuously wrapped by the SiC phase, resulting in a decrease in bonding strength and a decline in the overall performance of the composite material.

3) CVD diamond in CVDD/PCD superhard composite exhibited higher wear resistance than PCD polycrystalline diamond. Therefore, during operation, PCD wears out before CVD diamond. The composite material has self-sharpening properties, enabling it to maintain good grinding performance at all times.

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