

# Influence of High Sintering Pressure on the Microhardness and Wear Resistance of Diamond Powder and Silicon Carbide-Based Composites

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The work reported on here involved the development of several samples of “diamond-SiC” composite produced under sintering pressures of up to 9.0 GPa at temperatures of up to 1973 K. The average size of the diamond micropowder crystals used was 40/28  $\mu\text{m}$ . The sintering process was carried out in a 2500-ton hydraulic press equipped with an anvil-type high-pressure device having a toroidal work surface and a central concavity diameter of 20 mm. The microhardness and wear resistance of the samples were found to be dependent on the sintering pressure. The experimental results indicated that the maximum microhardness and minimum wear resistance coefficients of each compact were attained when the pressure applied during sintering exceeded 6.5 GPa. Based on the established values of pressure, this study served to identify the types of devices applicable for the manufacture of composite material inserts for a variety of rock drilling applications.

**Keywords:** *diamond powder, sintering, silicon carbide, composite*

## 1. Introduction

The use of thermally polycrystalline diamond materials has become increasingly common in drilling bores and cutting tools for application in both the oil and metal-mechanical industries. These materials are produced by high-pressure high-temperature sintering of diamond micropowder with the addition of a variety of additives<sup>1-3</sup>, which become synthesized in the diamond-silicon carbide system. Silicon, the most commonly employed additive, acts as an activator of the process<sup>4,5</sup>.

At pressures of over 3.0 GPa and temperatures exceeding 1573 K, the diamond micropowder undergoes a process of impregnation by the melted silicon. In the regions outside the boundaries of contact between the diamond particles (pores), the liquid phase reacts, causing the formation of silicon carbide<sup>3</sup>. This results in the production of a body composed of a material whose structure is formed by the interpenetration of the diamond micropowder and silicon carbide particles<sup>6</sup>. This structure and the simple type of bond formed between the silicon carbide and diamond powder during sintering render the material extremely hard, a property that remains unaltered even after heat treatment at tem-

peratures of up to 1473 K. This characteristic allows for the use of standard welding and soldering technologies in the manufacture of tools with this type of composite material, and also permits them to work efficiently under the high temperature conditions generated when operating the tool.

The composition of the composite material varies according to the sintering pressure applied and the particle size of the diamond powder used. Hence, the properties and the areas of application of the composite material are also variable. However, for commercial reasons, the particularities of these materials' production processes are treated as industrial secrets. Within this context, therefore, the purpose of the present work was to determine the dependence of the mechanical properties of composite materials (microhardness and wear resistance) on the sintering pressure used in the manufacture of the “diamond-SiC” system.

## 2. Materials and Methods

The sintering process was carried out in a 2500-ton hydraulic press<sup>7</sup> equipped with an anvil-type high-pressure device having a toroidal work surface<sup>8</sup> and a central con-

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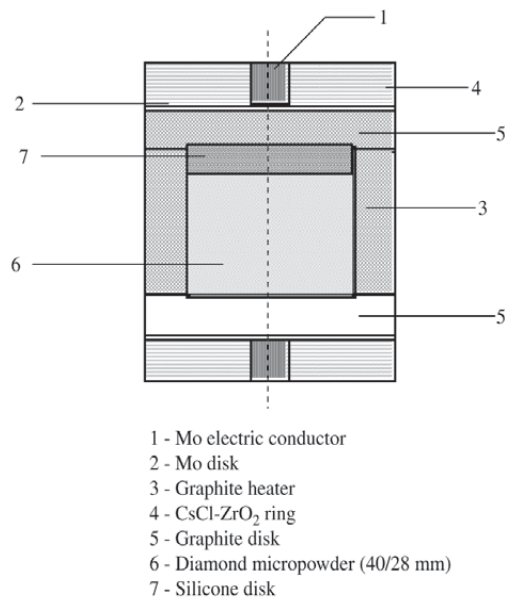
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cavity diameter of 20 mm. With this setup, it is possible to use a 16 mm diameter reaction cell. Figure 1 shows a schematic drawing of the reaction cell.

The high-pressure device was precalibrated in order to obtain results with greater precision. The pressure was calibrated at ambient temperature as a function of the phase transformations of the Bi I – II (2.55 GPa), PbSe (4.23 GPa) and Bi VI-VII (7.7 GPa). The pressure calibration graph was constructed based on the correlation between the pressure in the main cylinder of the press and the pressure inside the compression chamber. The temperature was measured using a Pt/Pt-10%Rh thermocouple as a function of the electric current applied to generate heat inside the compression chamber. The influence of the pressure exerted on the e.m.f. of the thermocouple was not taken into account.

The experiments were conducted in the following sequence:

- Cold compaction of the mixture at 3.0 to 9.0 GPa.
- Heating of the cell to a temperature range 1573 - 1713 K for 60 s.
- Turning off of the heating system.
- Gradual reduction of the pressure to ambient pressure.
- Disassembly of the cell and removal of the sample.
- Machining of the tip of the sample.
- Determination of the Knopp microhardness at the sample's tip using a diamond indenter with a 9.8 N load.
- Testing to determine the wear resistance coefficient. For this step, an abrasive  $\text{Al}_2\text{O}_3$  cutting disk was used,



**Figure 1.** High pressure cell.

and the samples and disk weighed before and after each test.

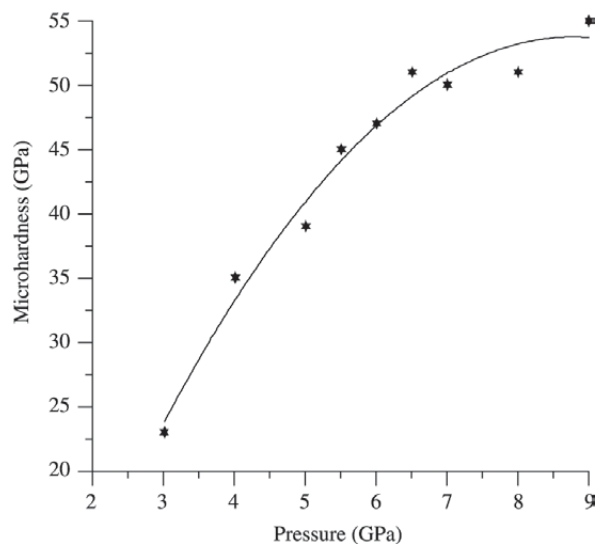
- Calculation of the wear resistance coefficient, based on the correlation between the mass loss of the sample ( $\Delta M_c$ ) and that of the abrasive disk ( $\Delta M_D$ ). The relation (1) shown below was used for this calculation.

$$n = \frac{\Delta M_c}{\Delta M_D} \rightarrow \left( \frac{mg}{kg} \right) \quad (1)$$

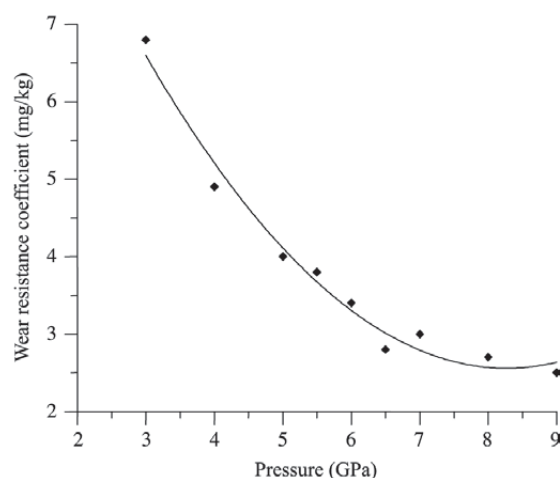
### 3. Results and Discussion

The high-pressure high-temperature sintering experiments yielded thirty-two 12 mm diameter and 5 mm high samples of the composite. Upon conclusion of each experiment, the samples were tested for microhardness and wear resistance coefficient, the results of which are illustrated in Fig. 2 and 3.

Our analysis of the influence of the sintering pressure indicated that, at values of over 6.5 GPa, the mechanical properties investigated displayed no significant variations. The microhardness reached a maximum value of  $52 \pm 3$  GPa, tending to remain constant with increased pressure. The same behavior was displayed by the coefficient of wear resistance, which tended to remain constant at  $2.9 \pm 0,3$  mg/kg at pressures of over 6.5 GPa. It should be noted, however, that these results are valid specifically for the particle size and sintering



**Figure 2.** Dependence of the Knopp microhardness of the diamond-SiC composite on the sintering pressure.



**Figure 3.** Dependence of the diamond-SiC composite's wear resistance coefficient on the sintering pressure.

times employed in this study. One can therefore assume that, with particle sizes and sintering times other than those utilized here, the material's microhardness and wear resistance may display a different behavior.

It has been demonstrated<sup>9</sup> that the value of the specific surface of diamond grains, as well as the mean diameter of the pores generated during the sintering process of diamond powder, do not vary significantly in response to pressures of over 6.0 GPa. This observation corroborates our own findings, validating the results obtained from this investigation. Increased pressure in the compression chamber of the high-pressure device caused the porosity of the compacted micropowder to decrease, reducing the carbide phase content and increasing the diamond phase of the composite samples produced. This fact was confirmed by our X-ray analyses, which showed that, in the samples sintered at pressures of over 6.5 GPa, the SiC content remained unchanged and was approximately equal to 23.5% (in mass).

A drop in the high pressure from 6.5 GPa to 3.0 GPa caused the SiC content in the structure of the sintered diamond-silicon carbide composite to increase from 23.5 (mass%) to 35.0 (mass%), while the contact area between the diamond grains decreased. These changes in the composite's structure reduced the microhardness and increased the wear resistance coefficient of the diamond-silicon carbide composite.

#### 4. Conclusions

Based on the results and discussion presented herein, the following conclusions can be drawn:

- It was shown that a correlation exists between the structure and properties of the "diamond-SiC" composite material.
- Based on the conditions employed in this study, a minimum value was established for the sintering pressure at which it is possible to produce a two-phase (diamond and SiC) polycrystalline composite material with excellent mechanical properties.
- This minimum pressure (6.5 GPa) will serve as a reference for the selection of the type and dimensions of high-pressure device compression chambers.

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