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Investigation of the Physical and Mechanical Properties of Silicon Carbide Prepared on an Industrial Scale

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ABSTRACT

In this study, an industrial polycrystalline SiC tile was successfully sintered by pressureless sintering at 2150°C for 1 hour. The physical and mechanical properties of silicon carbide including density, hardness, bending strength, and fracture toughness were evaluated. The results indicated that the mentioned properties were 3.08 $g.cm^{-3}$, 2503 HV0, 249.3 MPa, and 1.23 $MPam^{0.5}$, respectively. The mechanical properties of samples showed that strength, hardness, and fracture toughness were low, indicating samples are inappropriate for industrial applications. It seems that the use of pressure-assisted sintering such as spark plasma sintering or sintering with the use of sintering aid materials such as alumina and yttrium oxide in the structure of the specimen, will improve the physical and mechanical properties of the sample.

1. INTRODUCTION

The striking mechanical properties of silicon carbide are due to the strong covalent bond between carbon atoms and silicon [1]. Silicon carbide is a good semiconductor, which is also used in electronic applications [2]. Silicon carbide usually has two alpha and beta phases, and each of these two phases is synthesized in different ways, which has cubic, hexagonal, and rhombohedral crystal structures. The arrangement type of atoms is different in these structures. Its hexagonal structure is divided into structure of 2H, 4H, and 6H structures and so on. The 2H structure is the most unstable and the 4H structure has the lowest energy. Typically, the alpha powder microstructure has a 6H co-axial grain that has lesser sintering capability than beta powder, and requires higher temperatures for sintering. In contrast, the Beta phase has co-axial fine-grains, which can be converted to an alpha phase if heated at temperatures above 1950°C, and forms a plate-shaped blade and will strengthen the silicon carbide in the case of coarsening. The sintering temperature of the silicon carbide is about 2500°C [3]. Various additives such as iron, aluminum, boron, magnesium or lithium are used to reduce the temperature of the sintering.Moreover, oxides such as alumina, boron oxide, or yttria are also used in the sintering process. The research has shown that the presence of carbon as a sintering aid will increase the density. The presence of additives such as alumina and yttria (10% by weight in total) will increase the density and sinterability of silicon carbide [4-7]. The purpose of this research is to sinter the industrial SiC tile (150×100×50mm) by pressureless sintering. Furthermore, the physical and mechanical properties of the sintered tile were studied.

2. EXPERIMENTAL PROCEDURES

In this study, an Alpha silicon carbide powder was blended into a planetary ball mill with a mean particle size of 0.7 microns, coupled with the sintering aids such

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as carbon and boron carbide, for 8 hours. It was then pressurized at a pressure of 50MPa. The specimen was sintered at 2150°C for 1 hour and the physical and mechanical properties were evaluated after the sample was cut. The Phase composition was determined by XRD of specimens using a Philips-PW3710 operating at 40kV and 30mA using Cu Kα radiation (λ = 0.15406nm). The ASTM B962 [8] standard was used to determine the density, water absorption, and apparent porosity, and the ASTM C 1327-15 [9] standard was used to determine hardness. The hardness of the specimens was evaluated according to Vickers Hardness Test (MVK-H21, Akashi) as well as (under 100gr load and a load time of 15 seconds) standard. Each test was repeated 5 times for each sample and the mean of the obtained values was reported to increase the accuracy of the test. In addition, the C1161-13 [10] standard was used to determine the bending strength of the samples, and the standard C1421 [11] was used to determine the fracture toughness of the specimens. Ultimately, the field emission scanning electron microscope (FESEM-TESCAN) was used to test the microstructure of the sample.

3. RESULTS AND DISSCUSSION

Fig. 1 illustrates the XRD of the sintered specimen. XRD analysis reveals that all the characteristic peaks related to SiC have appeared. The uniform phase composition of silicon carbide (reference code: 01-072-0018) formed, and there was not unreacted phase remained in the sample, which means that the reaction of sintering aids and SiC completely occurred during the sintering process. The results of the calculation the density, apparent porosity, and water absorption for the SiC sample are given in Table 1. The relative density of the sample is reported with respect to the density of silicon carbide 3.21g.cm^{-3} [12]. The density of the three samples was measured by the archimedes method for density measurement, then the obtained density was as much as 3.081g.cm^{-3} by averaging. As the results show, the density of the sample is lower than its ideal amount, which can be attributed to the presence of porosity in the sample structure, the presence of phases of the sintering aids such as carbon that has a lower density than silicon carbide, and also the lack of pressure during the sintering [5].

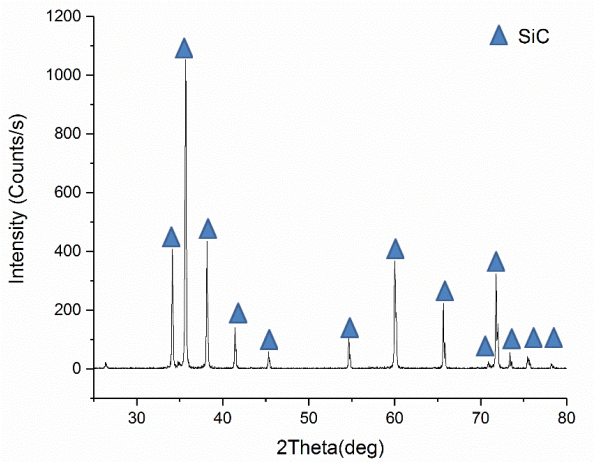

Figure 1. XRD of SiC sintered specimen

The hardness results for the sample from the approved locations are shown in Table 2. The average hardness of this sample was 2503 Vickers. The hardness of a ceramic sample depends on factors such as grain size, porosity, and microstructure [13].

TABLE 1. The Density of silicon carbide samples

Sample	Dry Weight (g)	Wet Weight (g)	Buoyancy Weight (g)	Density (g.cm^{-3})	Relative Density (%)	Porosity Percent	Water Absorption (%)
1	5.2687	5.2717	3.5729	3.101	96.6	3.38	0.057
2	4.9734	4.9831	3.3409	3.028	94.3	5.65	0.195
3	4.7294	4.7314	3.2128	3.114	97	2.98	0.042

TABLE 2. Hardness results of carbide silicon tile sample

Test	Indent Diameter d1 (mm)	Indent Diameter d2 (mm)	Average Diameter (mm)	Hardness HV0.1 (Vickers)
1	0.0085	0.0087	0.0086	2507
2	0.0085	0.0085	0.0085	2566
3	0.0088	0.0085	0.0087	2449
4	0.0086	0.0083	0.00845	2597
5	0.0089	0.0087	0.0085	2394

A three-point test was used to evaluate the bending strength of the specimen, in which the dimensions of the specimen were 45×4×45mm and the fixture spacing was 40mm. The samples were carefully cut by the diamond blade, and then, they were grinded and polished to remove hair cracks. Five samples were examined at a loading rate of 0.5mm.min^{-1}. The test was performed the SANTAM-STM-20 model. The bending strength results are given in Table 3. The mean bending strength of the samples was 2449.3MPa.

TABLE 3. The results of the bending strength of Silicon Carbide tile

Sample	Thickness (mm)	Width (mm)	Length (mm)	Fracture Force (N)	Bending Strength (MPa)
1	3.05	3.98	40	118.2	191.55
2	3	4.03	40	153	253.1
3	2.95	3.95	40	169.2	295.33
4	2.95	4.02	40	129.5	222.10
5	2.98	4.02	40	169.2	284.38

The microstructure of the sample was examined by field emission scanning electron microscope. The results of this analysis showed that the carbon phase is present in the structure and, as mentioned in the introduction, this phase is used as a sintering aid in the process of sintering.The elemental analysis was carried out from dark points of the field to further evaluate and prove the presence of the carbon phase.

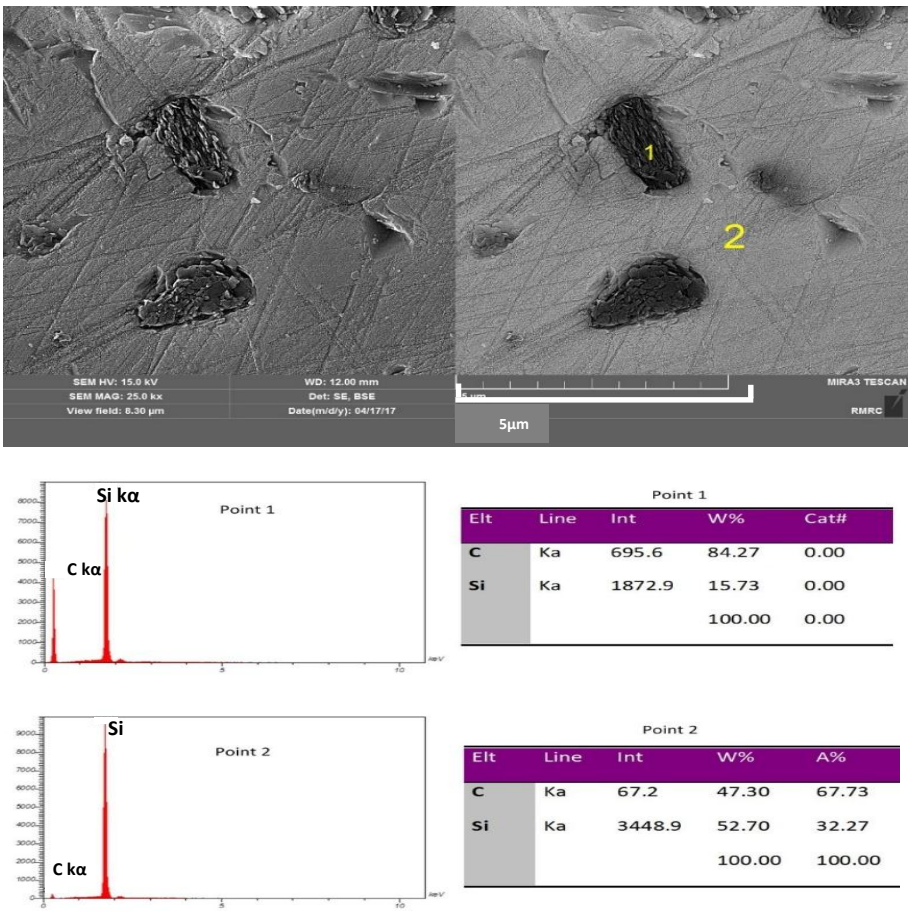

Figure 2. Elemental analysis of points 1 and 2

As shown in Fig. 2, point 1 represents 84% carbon phase of the elemental analysis of the field also shows the presence of carbon and silicon in the silicon carbide phase. As can be seen, the carbon phase appears with a sheet-shaped morphology. Moreover, the microstructure of the sample in terms of grain size and their distribution is shown in Fig. 3. The average grain size was obtained using MIP4 image analysis software. The average grain size was as much as 1.33µm. To confirm the result, another image analysis was carried out from the other part of the sample, which resulted in an average grain size of 1.27.

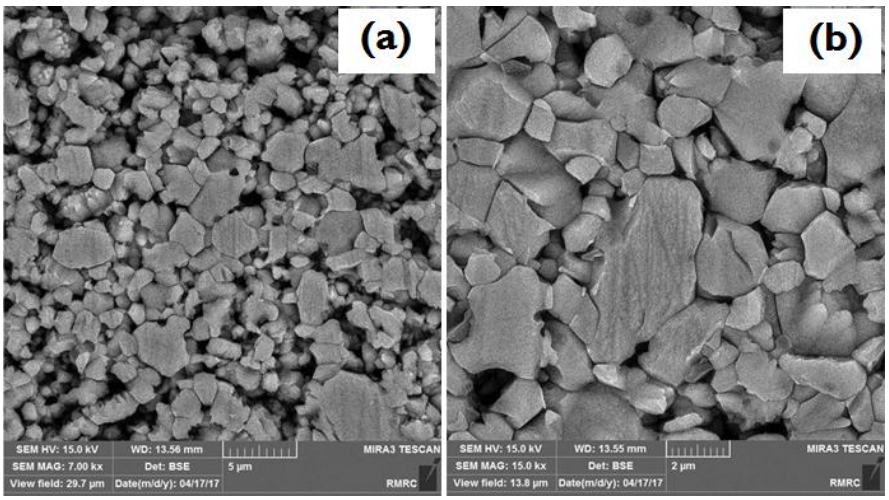

Figure 3. Scanning the electron microscope image of the sample in two different magnifications

Samples were prepared with dimensions of 6.35 mm × 6.35 mm × 45 mm. In order to measure the fracture toughness, and a groove with a 0.25mm width applied on these specimens using a shear plate with a thickness of 0.20, (Figure 4). Then, a force of 0.2mm.min^{-1} was applied in the test of bending strength using a three-point method. It is possible to determine the fracture toughness based on the ultimate strength of the fracture and the dimensions of the specimens as well as the conditions $a_0/w<0.42$ and $0.95<1$ through Equation (1)

$$K_{Ivb} = Y^*_{\min}\left[\frac{P_{\max}[S_0 - S_i]10^{-6}}{BW^{3/2}}\right] \quad (1)$$

In equation (1) which, Y^*_{min} is the stress intensity factor, which is calculated from Equation (2):

$$Y^*_{\min}(a_0/W, a_1/W) = \frac{0.7601 - 3.6364(a_0/W) + 3.1165(a_1/W) - 1.2782(a_1/W)^2 + 0.3609(a_1/W)^3}{1.000 - 3.1199(a_0/W) + 3.0558(a_0/W)^2 - 1.0390(a_0/W)^3 + 0.0608(a_1/W)} \quad (2)$$

In which, P_{max}, S_0-S_i, B, and W are the maximum force exerted on the sample, the difference between the two external holders (S_0) and the distance between the two internal holders (S_i), the sample width, and sample thickness, respectively. In addition, a_0, a_{11} and a_{12}, a_1, a_0/W, and t represent the size of the tip of the Chevron, the dimensions of the Chevron, which is a coefficient of sample thickness, the average of a_{11} and a_{12}, the size of the groove and the thickness of the groove, respectively.

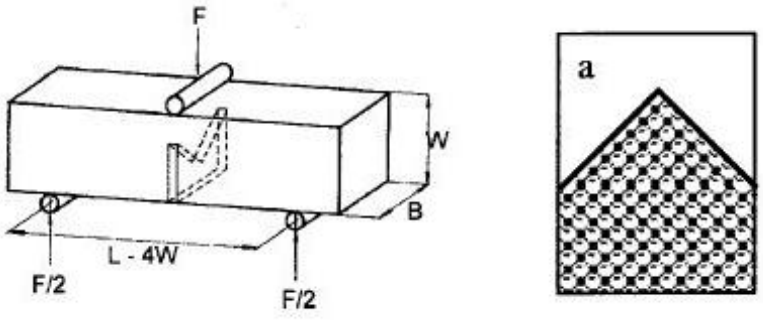

Figure 4. Schematic of Chevron groove

The results of the fracture toughness are presented in Table 4. As can be seen from the data in this table, the mean value obtained using this method is 1.23MPam$^{0.5}$, which indicates that the fracture toughness of this specimen is low, in comparison with [14-15].

TABLE 4. Fracture toughness of samples

Sample	a_0 (m)	$a_1=a_{11}=a_{12}$ (m)	a_0/W	B (m)	W (m)	S_0 (m)	P_{max}(N)	K_{IC} (MPa.m$^{1/2}$)
1	0.00254	0.0635	0.040	0.0635	0.0635	0.04	38.7	1.17
2	0.00243	0.0635	0.0382	0.0635	0.0635	0.04	40.7	1.22
3	0.00254	0.0632	0.040	0.0630	0.0632	0.04	32.4	0.98
4	0.00252	0.0632	0.040	0.0630	0.0632	0.04	32.9	1.01
5	0.00258	0.0635	0.040	0.0635	0.0635	0.04	46.1	1.39
6	0.00257	0.0635	0.040	0.0635	0.0635	0.04	53.5	1.61
7	0.00251	0.0633	0.040	0.0633	0.0633	0.04	41.7	1.26

4. CONCLUSION

The results of density, hardness, bending strength, and fracture toughness tests showed that this sample has a relatively low density as well as high water absorption and porosity. The mechanical properties of this sample also showed that its strength, hardness, and fracture toughness were low, indicating this sample is inappropriate for applications requiring the use of materials with high-strength fracture toughness. Moreover, microstructural studies indicated the presence of the carbon phase in the structure, which can be a reason for the reduction of mechanical properties. It seems that the use of high-quality primary powder, full sintering with additives such as yttria and alumina, and the use of as well as pressurized sintering methods such as SPS, will help to improve the quality of the samples.

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