



Effects of sintering conditions on the microstructure and mechanical properties of SiC prepared using powders recovered from kerf loss sludge

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Abstract. The effects of sintering conditions on the microstructure and mechanical properties of the sintered SiC prepared using the SiC powder recovered from the kerf loss sludge were investigated. The recovered SiC powders were consolidated by spark plasma sintering (SPS) and conventional sintering methods. The effects of sintering temperature, time and methods (SPS and conventional sintering) on the phase, grain size and density of SiC were systematically studied. The Vickers hardness of spark plasma-sintered (SPSed) samples was higher than that of conventional sintered samples due to small grain size. When holding time was increased from 10 to 30 min, the grain size and relative density of SPSed samples were also increased, which lead to the almost constant Vickers hardness by competing effects of grain size and relative density. When holding time was over 30 min, no appreciable change of the relative density and grain size were observed, which can lead to similar values of Vickers hardness. SPS process can be used to make SiC with high density and hardness at relatively low temperature compared with the conventional sintering process.

Keywords. Recovered SiC; sintering conditions; spark plasma sintering (SPS); conventional sintering.

1. Introduction

The photovoltaic (PV) industry uses a large amount of silicon, and also used in the solar cell manufacturing process, more than 50% of silicon is wasted as kerf loss sludge during the cutting of the silicon ingots when Si ingots are sliced into thin wafers [1–6]. The kerf loss sludge consists of silicon powders, abrasive silicon carbide (SiC) powders, metal from the saw wire and glycol-based solution [1,3,6]. The recovery of high purity Si or SiC from the kerf loss sludge was carried out using many methods, which include solidification, superconducting magnetic separation and flotation economic and environmental benefits [6–12].

SiC is one of the promising materials for wide range of applications, such as advanced engineering ceramics, aerospace, nuclear energy processing, ballistic protection and power semiconducting devices, because it has excellent mechanical strength/hardness, high thermal conductivity and good oxidation resistance, chemical and thermal stability and low density. The densification of SiC, however, is difficult due to the strong covalent nature of Si–C and the low self-diffusion coefficients, which result in the need of high temperature for dense SiC bulk [13–15]. Many efforts to obtain SiC bulk with

high density at relatively low temperature were reported. One way to obtain SiC with high density at relatively low temperature is spark plasma sintering (SPS) [15–23]. SPS consists of axial pressure and heated by a pulsed electric current passing through the die that contains the powder. Compared with other sintering methods, SPS can be used to obtain high density in a short time and at relatively low temperature. It was reported that very dense SiC can be obtained by SPS [17,19]. Ohyanagi *et al* [17] reported that nanostructured SiC with high relative density of 98% can be obtained without additives through SPS. Guillard *et al* [19] reported the effect of temperature, holding time and conditions of pressure application on density and microstructure of SiC, and the relative density of 92% was obtained in the sample, which was spark plasma sintered at 1850°C for 5 min under 75 MPa.

Another way to obtain SiC with high density at relatively low temperature is the addition of sintering aids such as oxide, boron and/or carbon-based materials [24–35]. If, oxide-sintering aids, such as alumina, zirconia and yttria are used, the liquid phase can be formed, which can lead to the enhancement of shrinkage and densification of SiC at relatively low temperature. However, there can be limits to the high temperature structural applications because of the

formation of the liquid phase at high temperature, which can degrade mechanical performance at high temperature [20,23]. On the other hand, boron or B_4C can enhance the diffusion rate of Si and C, and carbon can remove the SiO_2 layers, which can be present on the surface of SiC particles, and can lead to high density and enhancement of mechanical property of SiC [30,36]. Therefore, the use of boron and/or carbon as sintering additives seems to be a better way than that of oxides to achieve high density without the reduction in the mechanical performance at high temperature.

There were only a few reports on the sintering characteristics of SiC powders recovered from kerf loss sludge [37], although many approaches of recovering SiC powders from kerf loss sludge were carried out and reported. In this work, to investigate the sintering characteristics of the recovered SiC powders, the recovered powders were mixed with sintering aids (1 wt% B_4C and 3 wt% carbon), and were consolidated by SPS. The effects of sintering conditions on the microstructure and mechanical properties of the SiC were investigated, and these properties were compared with samples sintered by a conventional pressure-less sintering method.

2. Experimental

SiC powders with purity of 99.3% were recovered from kerf loss sludge, and the details of the recovery process and information of SiC powders were previously reported [37]. B_4C (Terabor®) and phenol resins (KNG100; carbon source, Kolon Chemical Co.) were used as sintering aids. The SiC powder recovered from kerf loss sludge, 1 wt% B_4C , 6 wt% phenol resins (~3 wt% carbon) and ethanol (95%, Samchun Chemical Co.) were mixed and ball-milled with SiC balls for 24 h, and then the mixture powders were dried at 70°C for 12 h. The dried powders were sintered by SPS and conventional pressure-less sintering method. SPS was performed at various temperatures (1650, 1700, 1750 and 1800°C) for different holding times (10, 30 and 60 min) under uniaxial pressure of 50 MPa. SPS was conducted in vacuum (10^{-3} Torr) with a heating rate of 100°C min⁻¹.

The conventional sintering was carried out at various temperatures (2070, 2100, 2130 and 2160°C) in Ar atmosphere for 1 h. Higher temperatures were used for the conventional sintering to obtain microstructure and densification similar to those of SPSed bulk. Phase analyses were performed using an X-ray diffractometer (D8 Advance, Bruker). Microstructural characterizations were carried out using a field emission scanning electron microscope (FESEM, SU-70, Jeol). Elemental compositions of sintered samples were obtained by energy-dispersive X-ray spectroscopy (EDS). The density and Vickers hardness of samples were obtained by Archimedes method (Voyager pro, Ohaus) and Vickers hardness tester (430SVD, Wolpert Group), respectively.

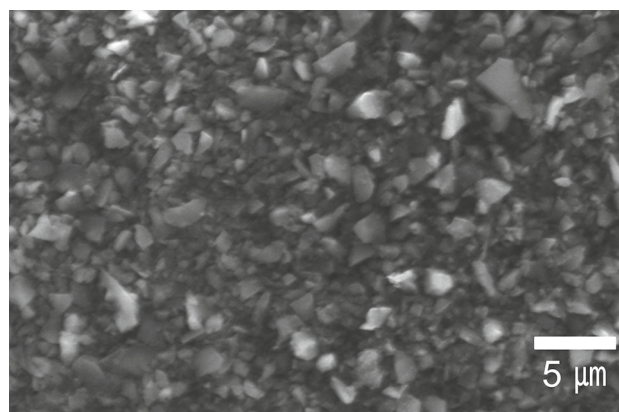


Figure 1. SEM image of SiC powder recycled from kerf loss sludge.

3. Results

Figure 1 shows an SEM image of SiC powders recovered from kerf loss sludge. The size of the particles, which was measured by SEM was 1–3 μ m. The laser particle size analyzer, which can count agglomerate as a single particle was used, and the average diameter (D50) of 8.8 μ m was measured [37], which is different from the sizes obtained from SEM.

Figure 2 shows microstructures of chemically etched surface of SiC with 1 wt% B_4C and 3 wt% carbon sintered by SPS (figure 2a–d) for 10 min and (figure 2e–h) conventional sintering (CS) for 1 h at different temperatures. It was reported that the densification is generally improved with increase in temperature [38]. The grain size of 1–3 μ m of the SPS samples showed no significant change from the initial particle size, and porosity decreased with increasing sintering temperature. On the other hand, the grain size of the CS samples increased from 5 to 9 μ m with increasing sintering temperature. In all SEM images, the dark grey regions are observed. It was reported that graphite and B-rich phase (which mainly consists of B and carbon) were present at the grain boundaries and the amount of Si in B-rich phase was negligibly small [37]. The amounts of B-rich phase and graphite can affect the density and mechanical properties of the SiC. In this study, the same amount of B_4C and carbon (1 wt% B_4C and 3 wt% carbon), however, were added as the sintering additive in all the samples, and hence, the effects of B_4C and C on the density and mechanical properties of SiC were not considered. Zhou *et al* [16] reported that the localized heating, which was higher than the measured temperature, can be generated by the electrical discharge during the SPS process and can lead to the grain growth. The grain size of SPSed samples in this study, however, were smaller and the grains were less elongated than those of the conventional sintered samples, because of the faster heating rate and shorter holding time of SPS than those of the conventional sintering. This indicates that the grain growth, which results from the localized heating did not take place during the SPS process in this study. These

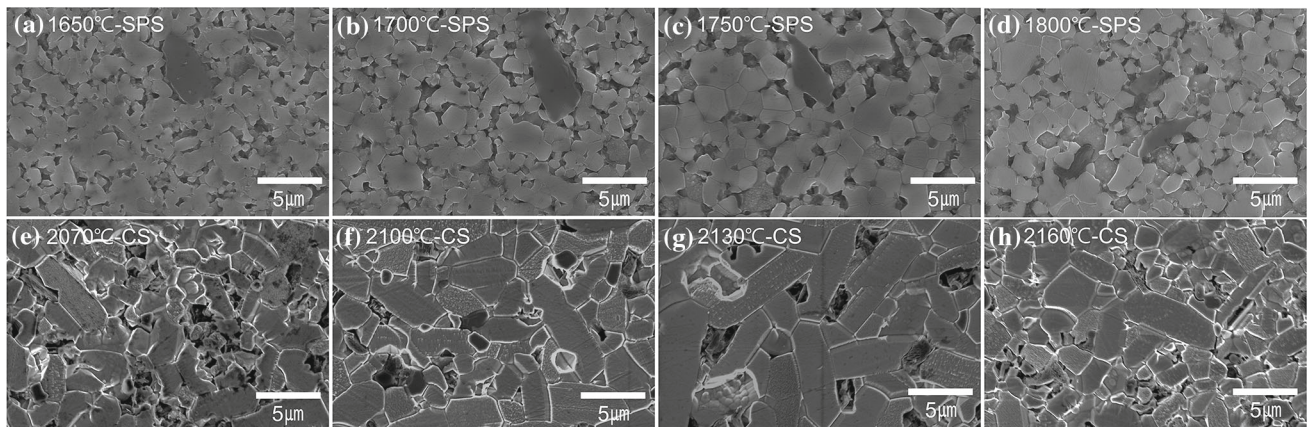


Figure 2. SEM images of B_4C and C-added SiC samples sintered at different temperatures for 10 min by (a–d) spark plasma sintering (SPS) and (e–h) conventional sintering (CS).

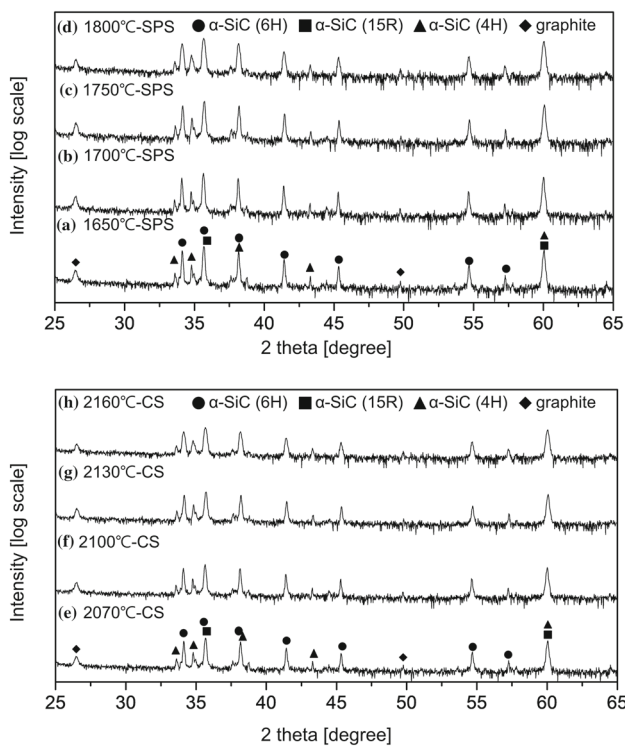


Figure 3. XRD patterns of B_4C and C-added SiC samples sintered at different temperatures for 10 min by (a–d) (SPS) and (e–h) conventional sintering (CS).

results are consistent with previous studies, which reported that nanostructured-SiC bulk can be obtained through SPS of commercial powders [15,20,23].

Figure 3 shows the X-ray diffraction (XRD) patterns of the SiC samples sintered with B_4C and carbon by SPS (figure 3a–d) and conventional sintering (figure 3e–h) at different sintering temperatures. The XRD patterns indicate that the major phase of all the samples was α -SiC composed of 4H, 6H and 15R. XRD peaks from graphite phase were also

Table 1. Results of Rietveld refinement quantitative phase analyses of B_4C and C-added SiC samples sintered at different temperatures for 10 min by spark plasma sintering and conventional sintering.

| Sample | 4H-SiC (%) | 6H-SiC (%) | 15R-SiC (%) | C (%) |
|----------|------------|------------|-------------|-------|
| SPS-1650 | 6.8 | 89.3 | 1.9 | 2.0 |
| SPS-1700 | 7.0 | 88.8 | 2.2 | 2.0 |
| SPS-1750 | 7.1 | 88.0 | 2.4 | 2.5 |
| SPS-1800 | 7.5 | 87.8 | 2.1 | 2.7 |
| CS-2070 | 5.7 | 89.0 | 2.5 | 2.8 |
| CS-2100 | 6.8 | 87.9 | 2.8 | 2.5 |
| CS-2130 | 9.1 | 84.1 | 3.5 | 3.3 |
| CS-2160 | 13.6 | 80.7 | 3.0 | 2.7 |

observed in all the SiC samples sintered by SPS and conventional sinterings. Any secondary peaks except those from graphite were not observed in all the samples. Peaks from the B-rich phase were not identified in the XRD patterns possibly because of the amount of this phase, which was below the detection limit of XRD. Campos *et al* [39] and Tsakiris *et al* [40] reported that the boron can be incorporated into the SiC via solid solution or can also persist as dispersed phase, and the B_4C detection limit of XRD is ~ 5 wt%. With increasing sintering temperature, the transition from 6H to 4H was observed in both the samples sintered by SPS and conventional sintering, which was also observed in the previous study reported by Yoshimura *et al* [41], who reported that 6H polytype of SiC tends to transform to 4H polytype with increasing sintering temperature in 0.4 wt% B_4C and 1.8 wt% C-added SiC. Rietveld analysis was used for the quantitative phase analysis of the SPS and CS samples, and the results are shown in table 1. In the case of SPS and CS samples, the phase transition from 6H to 4H occurred with increasing sintering temperature, but the degree of the phase transition of SPS samples is smaller than that of CS. A large number of elongated SiC grains were observed in CS samples with increasing sintering temperature. The change of phases and

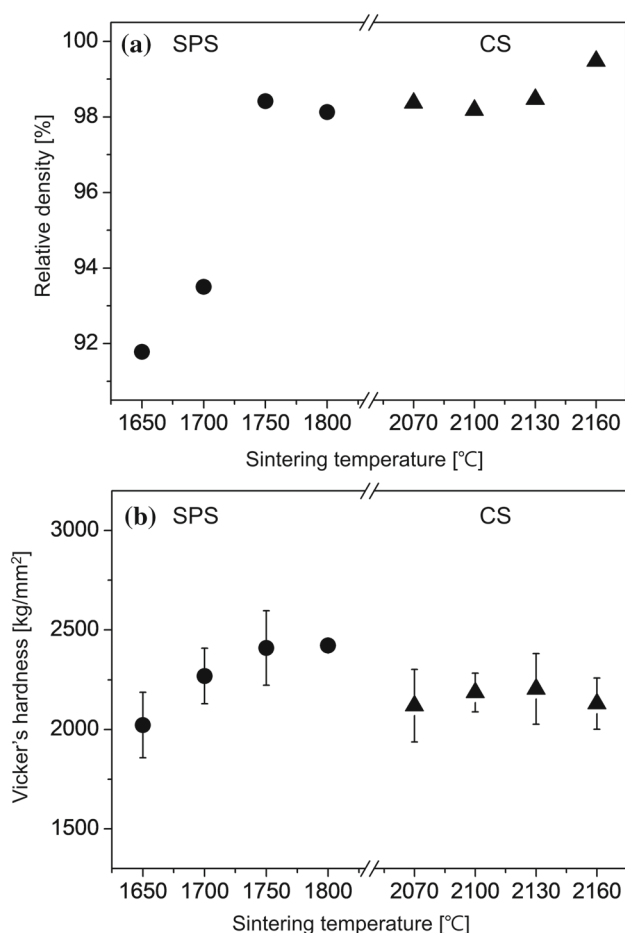


Figure 4. (a) Relative density and (b) Vickers hardness of B₄C and C-added SiC samples sintered at different temperatures for 10 min by spark plasma sintering (SPS) and conventional sintering (CS).

grain morphology, which were identified and observed from XRD and FESEM, respectively, indicates that the growth of elongated grains can be closely related to the phase transition from 6H to 4H. Figure 4a shows the relative densities of B₄C and C-added SiC samples sintered by SPS and conventional sintering at different temperatures. The theoretical density of the samples was 3.144 g cm^{-3} , which was calculated with the mixture rule (the theoretical densities of the SiC, B₄C and carbon used in the calculation were 3.21 , 2.52 and 2.267 g cm^{-3} , respectively) [42,43]. The relative densities of samples sintered by SPS were increased with increasing sintering temperature. Since sintering is a thermally activated process, diffusivity and viscosity are sensitive to temperature, which can be expressed as exponential functions of temperature [44]. The relative densities of samples sintered by a conventional sintering were also increased with increasing sintering temperature, but the amount of change is small compared to that of SPSed samples. Similar relative densities (98–99%) were obtained from SPSed samples at 1750 °C and conventional-sintered at 2070 °C. Tamari *et al* [38] and Zhou *et al* [45] reported that SPSed and conventional-sintered commercial SiC with high relative densities (98–99%) were

obtained at 1800 and 2000 °C, respectively, which is consistent with the results in this study. These results can be related with the characteristics of the heating in SPS such as self-heat generation through microscopic electric discharge between particles, leading to high speed mass and heat transfer [46,47]. And faster kinetics, such as surface diffusion, diffusion through the melt, and/or time-independent process such as plastic deformation can result in the rapid densification in SPS [38,48,49].

Figure 4b shows the change of Vickers hardness of B₄C and C-added SiC samples sintered by SPS and conventional sintering. With increasing sintering temperature, the increase of Vickers hardness was observed in the samples sintered by SPS. Vickers hardness is related to relative density and grain size. Hayun *et al* [22] reported that the hardness of the SPS-processed silicon carbide changes from 20 ± 2 to $32 \pm 0.7 \text{ GPa}$ with the relative density increasing from 89 to 99.4%, indicating that the hardness can increase with increasing relative density. And the effect of grain size on the hardness can be explained using Hall–Petch relation [50,51].

If the grain size is reduced, the Vickers hardness can be increased according to the Hall–Petch relation. The grain size and the relative density observed in the samples sintered by SPS were increased with increasing sintering temperature. Therefore, the increase in relative density can be responsible for the increase in Vickers hardness with increasing sintering temperature. In the case of the conventional sintering performed in this study, similar Vickers hardness values were obtained in the samples sintered at all the temperatures. As the sintering temperature was increased, the degree of increase in the hardness by the increase in density and the degree of decrease in the hardness by the increase in grain size can be similar to each other. In this case, there can be no significant changes in the hardness values, which can come from the competing effects of relative density and grain size on Vickers hardness. Figure 4b shows that the Vickers hardness values of the SPSed samples at temperatures >1750 °C are higher than those obtained from the conventional sintering at much higher temperatures (>2070 °C), which can result from the small grain size of SPSed samples.

Figure 5a shows XRD patterns and microstructures of B₄C and C-added SiC SPSed samples at 1700 °C for 10, 30 and 60 min (figure 5b–d). The XRD patterns of all the samples showed that major phase is α -SiC composed of 4H, 6H and 15R and graphite. No noticeable difference of XRD patterns was observed in the samples sintered for different holding times. As shown in figure 5b–d, the grain size was increased and the porosity was decreased with increasing holding time, but the grain size was similar in the samples sintered for 30 and 60 min. And the dark grey regions, which were also shown in the SEM images in figure 2 and these were called B-rich phase and graphite, were observed in all the samples, and were confirmed by EDS analyses (results are not shown).

Figure 6a shows the change in relative density and figure 6b shows Vickers hardness of B₄C and C-added SiC SPSed samples at 1700 °C for 10, 30 and 60 min. The relative

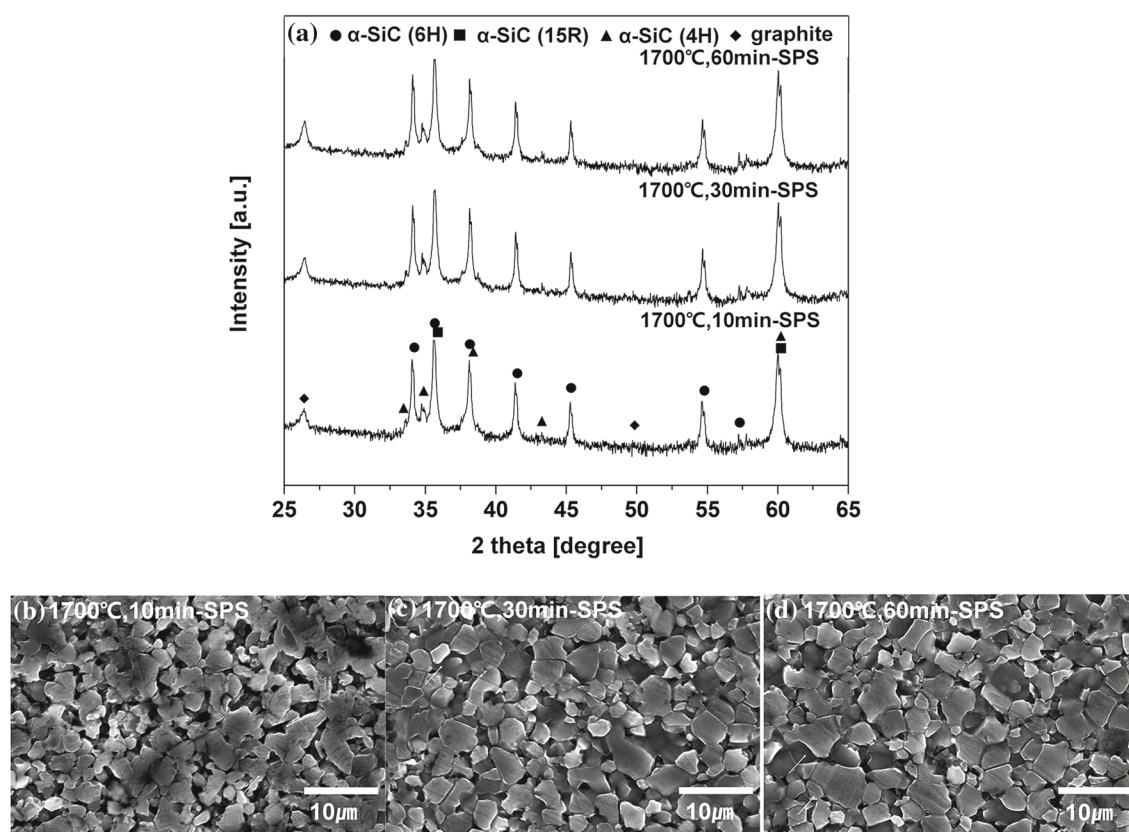


Figure 5. (a) XRD patterns and microstructures of B_4C and C-added SiC samples, which were SPSed at 1700°C for (b) 10, (c) 30 and (d) 60 min.

densities were increased with increasing holding time, but similar relative density was observed in the samples sintered for 30 and 60 min, which is consistent with the SEM images in figure 5b. Zhao *et al* [52] and Yamamoto *et al* [53] reported that the grain size and the density of SiC sintered by SPS were increased when holding time was changed from 1 to 30 min, which are consistent with the results of this study. Figure 6b shows Vickers hardness of B_4C and C-added SiC samples sintered by SPS at 1700°C for 10, 30 and 60 min. When holding time was increased from 10 to 30 min, the grain size and the relative density were increased. And hence, the competing effects of grain size and relative density can lead to almost constant Vickers hardness. On the other hand, when holding time was increased from 30 to 60 min, there was no appreciable change in the relative density and grain size, which can lead to similar values of Vickers hardness.

In this study, SiC powders recovered from kerf loss sludge were sintered under various sintering conditions, and their microstructures and mechanical properties were investigated. High density and hardness values, which are similar to those of SiC made with commercially available powder [54–56] were obtained, which shows that the recycled powder can be used to fabricate commercial products.

4. Conclusion

To investigate the sintering characteristics of the SiC powders recovered from kerf loss sludge, a mixture of recovered SiC powders and sintering aids (1 wt% B_4C and 3 wt% carbon) was consolidated by SPS and pressureless conventional sintering, and the effects of sintering conditions on the microstructure and mechanical properties were compared. With increasing sintering temperature, the increase in grain size and density, and the decrease in porosity were observed in the samples sintered by both SPS and conventional sinterings. The grain size of SPSed samples, which are smaller than that of conventional sintered ones was obtained at lower sintering temperature and for shorter sintering time than the case of conventional sintering. When sintering temperature was increased, the Vickers hardness of SPSed samples was increased due to the increase in relative density, while the samples sintered by conventional sintering have similar values of Vickers hardness due to the competing effects of grain size and relative density on Vickers hardness.

With increasing holding time of SPS from 10 to 30 min, grain size and density of SPSed samples were increased, which resulted in the similar Vickers hardness values due to the competing effects of grain size and relative density. When holding time was over 30 min, grain size and density

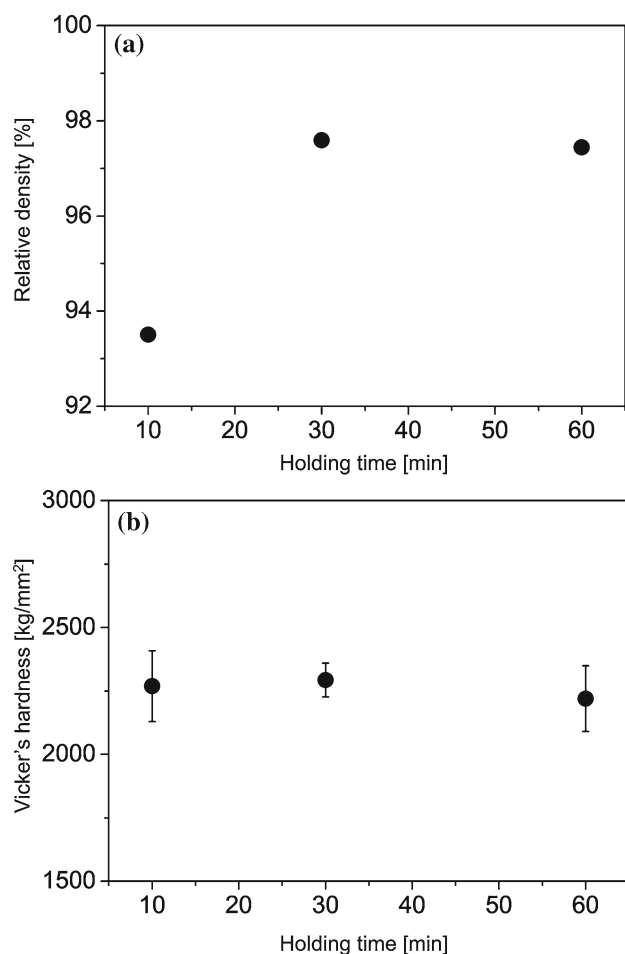


Figure 6. The change of (a) relative density and (b) Vickers hardness of B_4C and C-added SiC samples, which were spark plasma-sintered at $1700^\circ C$ as a function of holding time at sintering temperature.

of SPSed samples were not significantly changed, which can lead to similar values of Vickers hardness. This is the first report, to the best of our knowledge, on the effect of sintering methods/conditions on the microstructure and mechanical properties of SiC prepared using powders recovered from kerf loss sludge. And it was also confirmed that SPS can be used to make SiC bulk with high density and hardness at relatively low temperature compared with the conventional sintering process.

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