Spark Plasma Sintered Silicon Carbide Ceramics with Nanostructured Ferritic Alloy as Sintering Aid

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Abstract:

Nanostructured ferritic alloy-silicon carbide composites (NFA-SiC) were sintered at 1950°C-2100°C by spark plasma sintering (SPS). The influence of NFA addition on the sintered NFA-SiC composites has been investigated based on densification, phase, microstructure, and mechanical property. Addition of 2.5 vol% NFA can lead to full densification for the NFA-SiC composites even though it also introduces secondary carbon-rich and iron-rich phases. With the increase of the sintering temperature, the Vickers hardness for the pure SiC and NFA-SiC composite samples is enhanced. However, the NFA-SiC composites have lower hardness than the pure SiC samples. The flexural strength for the pure SiC is also higher than that of the NFA-SiC composites. Nanohardness results demonstrate that the reaction products between SiC and NFA lower the mechanical properties of the NFA-SiC samples.

Keywords: composites; sintering; fracture; microanalysis; mechanical characterization

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1. Introduction

Silicon carbide (SiC) is a well-recognized engineering material [1] for mechanical strength as well as physical and chemical stabilities up to very high temperatures. The exceptional stability under high radiation exposure [2-4] makes it an alternative material in harsh radiation environments, such as in fission and fusion nuclear reactors, due to the low induced activation and low after-heat properties. At high temperatures, the impurities in SiC show extremely low diffusivities, which makes it quite different from traditional nuclear materials. Thus, SiC is being used as fission product barriers in the TRISO fuel [5]. SiC coating layer for fission fuel particles is effective in retention of solid fission products at temperatures exceeding 2000 K [6, 7]. As a result, SiC-based ceramic composites are expected to be promising structural materials in fission energy systems.

From the processing point of view, SiC is a covalently bonded compound [8]. It is difficult to densify SiC without sintering additives based on the conventional methods. Although sintering additives [9, 10], including boron-based compound, yttrium oxide, and aluminum oxide, are used to promote SiC densification and its mechanical properties [11, 12] by introducing a liquid phase at grain boundaries, these additives deteriorate the irradiation performance (e.g., neutron absorption), cause swelling [13], and limit the nuclear applications. In order to achieve full density for SiC but avoid these drawbacks, sintering SiC with nanoparticles without any additives [10, 13] by using spark plasma sintering (SPS) has been reported with improved mechanical properties. Densification of SiC ceramics with additives [9, 14] is also demonstrated by using SPS.

SPS [15] densifies green samples in a graphite die by using pulsed DC electric current and simultaneous pressure. Compared with conventional pressure-assisted sintering, the sintered samples by SPS have improved characteristics because of the effects of spark plasma, spark impact pressure, Joule heating, electromagnetic field, electromigration, surface current,

and rapid heating. The externally applied pressure is generally 30-200 MPa. The heating rate is $100\text{-}1000^{\circ}\text{C}\cdot\text{min}^{-1}$, which is much higher than that of electric resistance rapid heating [16]. High heating rates reduce the duration of surface diffusion and favor the sinterability of hard-to-sinter powders. Densification is intensified by grain-boundary diffusion while grain growth is hindered [17]. This method has attracted considerable attention as a rapid sintering method. It is also capable of producing highly dense and homogeneous sintered components [18].

Nanostructured ferritic alloys (NFAs) are defined as a mixture of mechanically alloyed and particle-hardened material with nanograined Fe-Cr alloy matrix and Y-Ti-O-Cr-Fe enriched nanoclusters [19-21]. Due to the excellent creep and irradiation resistances, NFAs have been considered as primary candidate materials for both fission and fusion reactors [22]. A small amount of NFA addition to SiC can potentially enhance the creep and irradiation resistances of SiC. Furthermore, due to the low melting point of NFA, the sintering of SiC can be accelerated, which would make SiC easier to achieve full density.

However, it has been shown that reactions between Fe and SiC happen during the fabrication of Fe-SiC composites, both in experiment [23-25] and simulation [26]. At high sintering temperatures, SiC decomposes in the presence of Fe. The decomposed Si and C then interact with Fe and form iron silicide (e.g., Fe_3Si , FeSi and FeSi_2) [27] and iron carbides (e.g., Fe_3C) [28]. Considering this problem, one possibility to achieve high density NFA-SiC composites is by fast sintering of SPS to decrease the potential reactions between them. Our work about the SPS sintering of pure NFA and NFA-SiC composites with different compositions (97.5 vol% NFA-2.5 vol% SiC and 95.0 vol% NFA-5.0 vol% SiC) has been reported recently [29].

In this work, pure SiC and NFA-SiC composites (2.5 vol% NFA-97.5 vol% SiC, and 5.0 vol% NFA-95.0 vol% SiC), in which the NFA was also considered as a sintering aid, were

fabricated between 1950°C and 2100°C by SPS. The density, phase, and microstructure were studied. Mechanical properties including Vickers hardness, flexural strength, and nanohardness were characterized.

2. Materials and methods

Commercial SiC powder (SiC UF-15, H. C. Starck, Goslar, Germany) and lab-made NFA (Fe-9Cr-2W-0.4Ti-0.2V-0.12C-0.3Y₂O₃) powder [21, 22] were used as raw materials. The particle sizes for the SiC and NFA were measured by a laser light scattering particle size analyzer (LA-950, Horiba Scientific, Tenyamachi, Japan). The corresponding average particle sizes were 1.24 µm and 14.28 µm, respectively. The volume ratios for the NFA and SiC powders in the two NFA-SiC composites were 2.5:97.5 and 5.0:95.0, respectively. Ball milling of the SiC and NFA powders was initially carried out for 60 mins before the sintering process. Subsequently, the powders were poured into a cylindrical graphite die for the SPS process. The die had 20 mm diameter and the height of the powders was controlled at about 5 mm. Sintering for both the pure SiC and the NFA-SiC composites was performed at 100 MPa pressure, 100°C/min heating rate, and 10 min holding time. The peak temperatures were 1950°C, 2000°C, 2050°C, and 2100°C, respectively.

The density of the sintered samples was measured by a gas pycnometer (AccuPyc II 1340, Micromeritics, Norcross, GA). In order to estimate the relative density, the sintered sample theoretical density was calculated by considering the theoretical densities of SiC (3.21 g•cm⁻³) and NFA (8.23 g•cm⁻³) and their contents. The phase composition was measured by X-ray powder diffraction (XRD, PANalytical B.V., EA Almelo, Netherlands). The microstructure was characterized by electron scanning microscopy (SEM, Quanta 600 FEG; FEI Company, Hillsboro, OR). The elemental composition was measured by an energy dispersive X-ray spectroscopy module (EDS, Bruker AXS, MiKroanalysis, GmbH, Berlin, Germany), which was attached to the SEM. The Vickers hardness was obtained from 15

indentations with a load of 98 N by using a hardness tester (LV700AT, LECO, St. Joseph, MI). The nanoindentation was carried out by using a nanoindenter (TI 950 Triboindenter, Hysitron, Inc., Minneapolis, MN) with a 3-sided pyramidal diamond Berkovich probe of 100 nm. The load was kept at 8000 μ N.

3. Results

3.1. Density

Fig. 1 shows the relative density of the sintered pure SiC and NFA-SiC samples at different sintering temperatures. The 2.5 vol% NFA-97.5 vol% SiC samples have the highest relative densities (near 100%) at the sintering temperatures of 1950°C and 2000°C. The 5.0 vol% NFA-95.0 vol% SiC samples have the lowest relative densities (91-94%) at the same sintering temperatures. The pure SiC samples have the relative densities (94-99%) in-between the two composite samples in the sintering temperature range of 1950°C-2000°C. This means that the addition of 2.5 vol% NFA benefits the densification of the NFA-SiC composites, while higher NFA content of 5.0 vol% NFA hinders the densification of the NFA-SiC composites. As an example, the 2.5 vol% NFA-97.5 vol% SiC sample has the highest relative density of ~100% at 2000°C, while the 5.0 vol% NFA-95.0 vol% SiC sample has the lowest relative density of ~92% at the same temperature.

3.2. Phase

Fig. 2 shows the XRD patterns of the pure SiC and NFA-SiC samples at different sintering temperatures. All the pure SiC samples show only the 6H-SiC and 4H-SiC phases, consistent with the raw powders. This means that the SPS sintering at 1950°C-2000°C does not alter the phase structure of SiC due to its high thermal stability. However, the NFA-SiC composites have additional phase peaks. These additional phases are identified as graphite

carbon and iron/chromium silicides, which are introduced by the chemical reactions between SiC and NFA.

3.3. Microstructure

Fig. 3 shows the SEM images of the fracture surfaces of the pure SiC and NFA-SiC composite samples at different sintering temperatures. These samples exhibit differences in morphology due to the different compositions. The pure SiC samples have relatively smooth fracture surface, while the two NFA-SiC composites have rougher fracture surface. Thus, the addition of NFA to the SiC matrix modifies the microstructure of the sintered NFA-SiC composites. The pure SiC samples have the densest fracture surface throughout the sintering range of 1900°C-2100°C, and it is difficult to observe any pores from the fracture surfaces. Even at the lowest sintering temperature of 1950°C, the pure SiC sample (with some small pores) is still denser than the NFA-SiC composite samples as shown in Figs. 3a, 3e, and 3i. When the sintering temperature increases to 2100°C, it is difficult to see any pores on the pure SiC fracture surface. This further explains why the pure SiC samples have increasing densities with the sintering temperature as shown in Fig. 1.

The fracture surface morphologies shown in Figs. 3(e-h) for the 2.5 vol% NFA-97.5 vol% SiC samples and in Figs. 3 (i-l) for the 5.0 vol% NFA-95.0 vol% SiC samples deserve further discussion. For both compositions, some lamellar phases are present in the sintered matrix. As a matter of fact, the lamellar phase exhibits no difference among all the NFA-SiC samples. However, the structures around the lamellar phase show some differences at different sintering temperatures. As shown in Figs. 3(e-f) and (i-j), at lower sintering temperatures of 1950°C and 2000°C, the 2.5 vol% NFA-97.5 vol% SiC and 5.0 vol% NFA-95.0 vol% SiC samples have porous structures around the lamellar phases. At higher sintering temperatures of 2050°C and 2100°C, the 2.5 vol% NFA-97.5 vol% SiC and 5.0 vol% NFA-95.0 vol% SiC

samples have much denser structures, and the lamellar phases are tightly embedded in the matrix, as indicated in Figs. 3(g-h) and (k-l).

It has been reported that when sintering the NFA-SiC composites, reactions between NFA and SiC happen [23-25] as follows based on XRD analysis:

The lamellar phases shown in Fig. 3 should be related to the reaction products from the above equations. The elemental mapping images for the lamellar phases in the 5.0 vol% NFA-95.0 vol% SiC sample sintered at 2000°C are shown in Fig. 4. The lamellar area is identified as the carbon phase, and no elements of Fe and Si are observed in this area. It is believed that after the reactions (Eqs. 1-5) the products of (Fe,Cr)Si and (Fe,Cr)Si₂ diffuse into the matrix from connected pores, while the carbon products remain and aggregate into the lamellar phases, which can be confirmed by XRD as shown in Fig. 2. The isolated Fe-rich domains are in the form of round shapes with the size around 1 μm. They are believed to be the Fe-rich phases of (Fe,Cr)Si and (Fe,Cr)Si₂ from Eqs. (1-5), which can also be observed by the XRD patterns in Fig. 2. Due to the dense local microstructures without any easy diffusion path, the Fe-rich phases are more likely to be isolated. The appearance of the carbon lamellar phase further demonstrates that the reaction products of the carbon phases defer the densification process and lead to porous microstructures around them.

3.4 Hardness

Fig. 5 shows the Vickers hardness of the sintered pure SiC samples and NFA-SiC composite samples at four different sintering temperatures. The pure SiC samples have the

highest hardness among all the samples at 1950°C-2100°C. The Vickers hardness has the increasing tendency with the values of 21.9 ± 0.8 GPa at 1950°C, 24.0 ± 0.7 GPa at 2000°C, 24.1 ± 0.5 GPa at 2050°C, and 25.3 ± 0.6 GPa at 2100°C.

The 2.5 vol% NFA-97.5 vol% SiC samples have slightly lower hardness than the pure SiC samples, but still show a steadily increasing tendency from 1950°C to 2100°C. Their Vickers hardness values are 17.4 ± 0.9 GPa at 1950°C, 20.7 ± 0.7 GPa at 2000°C, 22.4 ± 0.5 GPa at 2050°C, and 23.3 ± 0.5 GPa at 2100°C. With more NFA addition, the 5.0 vol% NFA-95.0 vol% SiC sample has lower Vickers hardness, especially at the sintering temperatures of 1950°C and 2000°C, with the corresponding values of 15.5 ± 0.8 and 15.7 ± 1.0 GPa, respectively. When the sintering temperature further increases to 2050°C, the Vickers hardness of the 5.0 vol% NFA-95.0 vol% SiC sample rapidly increases to 22.3 ± 0.7 GPa, which is about the same as that of the 2.5 vol% NFA-97.5 vol% SiC sample. There is no continuous increase of the hardness when the sintering temperature increases to 2100°C.

Fig. 6 shows two nanoindents on the 5.0 vol% NFA-95.0 vol% SiC sample sintered at 2100°C. The size of the indents is about 400 nm. The major elemental compositions for the matrix phase (Fig. 6a) and the bright phase (Fig. 6b) are listed in Table. 1. The white phase contains high amounts of Fe and Cr. However, the matrix contains negligible amounts of such elements. Thus, the bright phase is probably the iron-rich phase of iron silicide/iron carbide that comes from the reaction between NFA and SiC. Furthermore, the nanohardness measurements show that the bright phase has a smaller hardness value of 37.75 GPa, compared to 42.35 GPa for the matrix. Thus, the reaction products between SiC and NFA decrease the overall hardness of the NFA-SiC composite samples.

4. Discussion

4.1 Densification

The melting point of NFA is about 1500°C, while the melting point of SiC is about 2700°C. The sintering of the NFA-SiC composites at the temperatures between 1950°C and 2100°C can be considered as liquid phase sintering. The liquid melts of NFA move into the grain boundaries of the SiC matrix and facilitate the densification of the NFA-SiC composites. It is reported that α -SiC ceramics [30] can achieve full density by hot pressing with the addition of Y_2O_3 - Al_2O_3 as the liquid phase. However, the reactions between NFA and SiC at these high sintering temperatures cause the decomposition of SiC, and introduce new impurity phases (carbon and silicide identified by XRD in Fig. 2). This subsequently impacts the densification of the composites. As a result, a small amount of NFA (e.g., 2.5 vol%) benefits the densification of the composites due to the liquid phase formation. However, a high amount of NFA (e.g., 5.0 vol%) compromises the densification of the composites because of the negative effect of the reactions between NFA and SiC.

As expected, the relative density of the pure SiC sample increases with the sintering temperature. Without any detrimental reactions between SiC and NFA, the pure SiC samples have much higher relative densities than the 5.0 vol% NFA-95.0 vol% SiC samples. However, the relative density of the 2.5 vol% NFA-97.5 vol% SiC sample declines when the sintering temperature is higher than 2000°C. This indicates that the 2.5 vol% NFA-97.5 vol% SiC samples should be sintered at lower temperatures of 1950°C-2000°C to achieve full density. More silicide products and carbon phases are produced with the sintering temperature increase, which adversely decreases the relative density of the 2.5 vol% NFA-97.5 vol% SiC samples. The relative density of the 5.0 vol% NFA-95.0 vol% SiC sample does not change significantly at different sintering temperatures, likely because the positive effect of the liquid

phase sintering is balanced by the negative effect of the reaction products of silicide phases and carbon phases in the entire temperature range.

4.2 Mechanical properties

The increasing trend of the Vickers hardness for the pure SiC samples is consistent with the relative density change at different sintering temperatures. The Vickers hardness of the pure SiC sample reaches the maximum value of 25.3 ± 0.6 GPa when the relative density reaches the maximum value of $99.0 \pm 0.1\%$. This means that higher density enhances the hardness. The similar increasing trend of the Vickers hardness with the relative density has been demonstrated for the SPS sintered SiC [13]. The hardness of the sintered pure SiC at 2100°C has the maximum value of 25.3 ± 0.6 GPa, which is slightly higher than 25 ± 0.2 GPa of the pure SiC sintered by SPS at 1800°C [13], but lower than 30 ± 1 GPa of the pure SiC sintered under ultra-high pressure at 2200°C - 2400°C [31]. The NFA addition samples have the maximum hardness value of 23.3 ± 0.5 GPa at 2100°C , which is higher than 22.8 ± 0.8 GPa of the hot pressed SiC [32] with Y_2O_3 and Al_2O_3 as additives at 1880°C , but lower than 30.2 ± 2.4 GPa of the pressureless sintered SiC at 2200°C with B_4C as an additive [33].

The lower Vickers hardness for the NFA-SiC composite samples can be understood as follows. The hardness of iron (4 GPa [34, 35]) is much lower than that of SiC (25-30 GPa [13, 31, 33]). Thus, the NFA-SiC composites with even a small amount of NFA addition would lower the hardness. For the lower Vickers hardness of the 5.0 vol% NFA-95.0 vol% SiC samples vs. the 2.5 vol% NFA-97.5 vol% SiC samples at 1950°C and 2000°C , another reason should be considered. From the microstructures in Fig. 3 and the elemental maps in Fig. 4, the NFA-SiC composites have porous structures around the carbon phases; it is believed that these porous structures contribute to the lower hardness of the NFA-SiC composite samples. The higher NFA addition in the 5.0 vol% NFA-95.0 vol% SiC sample causes more porous

structures, lower densities, and subsequently lower hardness than that of the 2.5 vol% NFA-97.5 vol% SiC sample.

However, the Vickers hardness of the 5.0 vol% NFA-95.0 vol% SiC samples is close to that of the 2.5 vol% NFA-97.5 vol% SiC samples at 2050°C and 2100°C. This is because at higher temperatures, the porous structures are greatly reduced and the matrix becomes much denser compared to the samples sintered at lower temperatures, as indicated in Fig. 3. Although the densities for the 5.0 vol% NFA-95.0 vol% SiC samples are not as high as for the 2.5 vol% NFA-97.5 vol% SiC samples, the new phases compromise the Vickers hardness of the 5.0 vol% NFA-95.0 vol% SiC samples.

To assess the strength of the sintered SiC and NFA-SiC samples, the relation between flexural strength and Vickers hardness for SiC ceramics is obtained by a linear fitting of the literature data [36-43] and can be expressed as follows:

$$\sigma_{FS} = 0.0303H_v - 96.4 \quad (6)$$

where σ_{FS} is the flexural strength, which has the unit of MPa; H_v is Vickers hardness, which has the unit of MPa as well. The specific information is in the supplemental information. Based on Eq. (6), the flexural strength of our sintered SiC and NFA-SiC composites is predicted as shown in Fig. 7. The change in the flexural strength is similar to that of the Vickers hardness as shown in Fig. 5. Higher sintering temperatures produce higher flexural strength for the SiC samples than for the NFA-SiC composites due to its higher Vickers hardness. Its highest flexural strength approaches 670 ± 17 MPa at 2100°C, which is comparable to 652 ± 33 MPa for hot pressed SiC ceramics using Al_2O_3 as an additive [43], and 691 ± 12 MPa for hot pressed SiC ceramics using Al/B/C as additives [42]. For the NFA-SiC composite samples, the lower content of 2.5 vol% NFA leads to higher flexural strength. The 2.5 vol% NFA-97.5 vol% SiC and 5.0 vol% NFA-95.0 vol% SiC samples reach their highest

flexural strength of 608 ± 16 MPa and 580 ± 29 MPa at 2100°C , which are slightly lower than that of the pure SiC sample.

5. Conclusions

Pure SiC and NFA-SiC composites with 2.5 vol% and 5.0 vol% NFA were sintered at 1950°C - 2100°C by spark plasma sintering (SPS). The density of the pure SiC samples shows an increasing trend with the sintering temperature and reaches nearly full density at 2100°C . NFA addition of 2.5 vol% benefits the full densification of the NFA-SiC samples at the sintering temperature of 1950 - 2000°C , but slightly reduces the densification of the NFA-SiC samples at the sintering temperature of 2050 - 2100°C . The 5.0 vol% NFA-95.0 vol% SiC samples have the lowest density among all the samples. The pure SiC samples have no secondary phases, whereas the NFA-SiC composites contain reaction products of graphite carbon and iron-rich phases, which compromise the densification process. The Vickers hardness of the pure SiC and NFA-SiC composites increases with the sintering temperature in the range of 1950°C - 2100°C . The estimated flexural strength for the pure SiC is higher than that for the NFA-SiC composites. The reactions between SiC and NFA decrease the overall hardness of the NFA-SiC composites.

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List of Figure Captions:

Fig. 1. Relative density of the pure SiC and NFA-SiC samples at different sintering temperatures.

Fig. 2. XRD patterns of the pure SiC and NFA-SiC composite samples at different sintering temperatures.

Fig. 3. SEM images of the fracture surfaces for the pure SiC and NFA-SiC composite samples at different sintering temperatures.

Fig. 4. Elemental mapping around the carbon-rich phase in the 5.0 vol% NFA-95.0 vol% SiC sample sintered at 2000°C.

Fig. 5. Vickers hardness of the pure SiC and NFA-SiC composites at different sintering temperatures.

Fig. 6. SEM images of nanoindents for the 5.0 vol% NFA-95.0 vol% SiC sample sintered at 2100°C.

Fig. 7. Flexural strength of the pure SiC and NFA-SiC samples at different sintering temperatures.

List of Table Caption:

Table 1. Elemental compositions of the matrix phase and the bright phase (Fig. 6) in the 5.0 vol% NFA-95.0 vol% SiC sample sintered at 2100°C.

Table 1. Elemental compositions of the matrix phase and the bright phase (Fig. 6) in the 5.0 vol% NFA-95.0 vol% SiC sample sintered at 2100°C.

Elements	C/wt%	Si/wt%	Fe/wt%	Cr/wt%
Matrix phase	32.8±0.4	66.8±0.4	0.4±0.2	0
Bright phase	20.9±2.7	50.2±1.9	26.7±0.9	2.2±1.1