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Research article

Evaluation of strength anisotropy and fracture behavior of UD NITE-SiC/SiC composites with various fiber orientations

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Abstract: A SiC/SiC composite by Nano-infiltration and Transient Eutectic-phase (NITE) process is the attractive candidate materials for advanced energy systems, aero-space system. The NITE process is improved to the industrialization grade process from the laboratory grade process. In order to ensure reliability of products by NITE-SiC/SiC composites, understanding of the strength anisotropy is important. This paper presented the basic strength anisotropy knowledges of unidirectional (UD) type NITE-SiC/SiC composites with various fiber orientations by correlation evaluation of microstructure and mechanical properties. Also, the strength anisotropy evaluation of UD NITE-SiC/SiC composites utilizing the strength anisotropy prediction theories were discussed. The axial/off-axial mechanical properties of UD NITE-SiC/SiC composites by the industrialization grade process were evaluated by axial/off-axial tensile test. The mechanical properties of NITE-SiC/SiC composites fabricated tended to decrease with increasing of fiber orientation angle. The experiment results by axial/off-axial tensile test were consistent with the strength anisotropy prediction theories by the maximum normal stress theory and the Tsai-Hill criterion. Also, the failure mode of SiC/SiC composites fabricated with each fiber orientation angle was consistent with fracture surface observation results. The strength anisotropy of UD NITE-SiC/SiC composites was suggested to be able to predict by Tsai-Hill criterion. The basic strength anisotropy of UD SiC/SiC composites was understood.

Keywords: advanced composites; SiC/SiC composite; NITE method; mechanical properties; strength anisotropy; fracture behavior

1. Introduction

A silicon carbide fiber-reinforced silicon carbide matrix (SiC/SiC) composite is the attractive candidate materials for advanced energy systems, aero-space system and etc. due to their potential excellent mechanical properties at high-temperature, chemical stability, radiation resistance and low induced radioactivity [1,2,3]. In generally, SiC/SiC composites has strength anisotropy depending on the reinforcement fiber architecture and their orientation. Therefore, in order to perform adequate fiber-architecture deign for each structural components, it is essential to understand the strength anisotropy due to fiber architecture.

On the other hands, there are many methods for SiC/SiC fabrication including chemical vapor infiltration (CVI), polymer impregnation and pyrolysis (PIP), reaction sintering (RS), liquid-phase sintering (LSP) and theirs hybrid process [4,5,6]. It is well known that the performance of SiC/SiC composites is different by fabrication process types. Therefore, properties evaluation of each SiC/SiC composites are necessary. The nano-infiltration and transient eutectic-phase (NITE) process is one of the most attractive processes for SiC/SiC fabrication because of its advantages in the formation of high density SiC matrix, size and shape flexibility and cost efficiency, which is the modified liquid phase sintering process [7,8,9]. The NITE process is improved to the industrialization grade process from the laboratory grade process by Organization of Advanced Sustainability Initiative for Energy System/Material (OASIS), Muroran Institute of Technology, Japan [2,10,11,12]. The feature of the industrialization grade NITE process is to utilize dry type inter-mediate materials such as green sheets and prepreg sheets. Since mechanical properties of SiC/SiC composites depend on fiber architecture, in order to ensure reliability of products by SiC/SiC composites, understanding of the strength anisotropy is important. However, strength anisotropy knowledges of NITE-SiC/SiC composites fabricated by the industrialization grade process are insufficient since this process is a new process.

This study aims to understand the strength anisotropy of NITE-SiC/SiC composites fabricated by the industrialization grade process with various fiber architecture. This paper is provided the basic strength anisotropy knowledges of Unidirectional (UD) type NITE-SiC/SiC composites with various fiber orientations by evaluation of microstructure and mechanical properties. The strength anisotropy prediction theories were also discussed to evaluate the anisotropic strength of UD NITE-SiC/SiC composites.

2. Materials and Method

The SiC mixed slurry for SiC green sheet fabrication consisted of β-SiC nano-powder (IEST, Japan, mean grain size of 32 nm) and sintering additives with Al_2O_3 (Kojundo Chemical Laboratory Co. Ltd., Japan, mean grain size of 0.3 μm, 99.99%) and Y_2O_3 (Kojundo Chemical Laboratory Co. Ltd., Japan, mean grain size of 0.4 μm, 99.99%). SiC green sheet were produced by OASIS, Muroran Institute of Technology, Japan. UD prepreg sheets were prepared by a similar fabrication process with those of green sheets, where PyC-coated Cef-NITE fibers (IEST, Japan) were used as a reinforcing fiber. The Cef-NITE fiber is one of the highly crystallized SiC fibers. The PyC coating was formed by chemical vapor deposition (CVD) process, and the thickness of the coating was appropriately 0.5 μm. The reinforcements were dipped to mixed slurry in slurry bath before to fabricate the prepreg sheets. The prepreg sheets fabricated were stacked for preparation of UD preforms. The number of prepreg sheets stacked is 30 sheets. The fiber orientation angle variations of preforms are kinds of four types (UD 0°, 30°, 45°, 60°). The preforms prepared were hot-pressed at 1870 °C for 1.5 h in Ar under a pressure of 20 MPa. The bulk density and open porosity of the composites fabricated were measured by the Archimedes' principle. Strength anisotropy evaluation was performed by axial/off-axial tensile test with the crosshead speed of 0.5 mm/min at room-temperature. The specimens were straight bar type, which measured 40L × 4W × 2.0T mm with a gauge length of 15 mm. Aluminum tabs were bonded at the gripping sections. Tensile strains were measured by a couple of strain gauges bonded on the both surface of a specimen. Fracture surface observation after axial/off-axial tensile test was performed by digital microscope and a scanning electron microscope (SEM).

3. Results and Discussion

Density, fiber volume fraction and mechanical properties of SiC/SiC composites fabricated with various fiber orientation angles are summarized in Table 1.

Table 1. Density, fiber volume fraction and mechanical properties of SiC/SiC composites fabricated with various fiber orientation angles in this study.

ID	UD0	UD30	UD45	UD60
Angle [°]	0	30	45	60
Fiber volume fraction [%]	46	45	46	46
Bulk density [g/cm^3]	2.9	2.9	2.9	2.8
Elastic modulus [GPa]	289 ± 8	231 ± 15	194 ± 7	170 ± 7
Proportional limit strength [MPa]	210 ± 1	88 ± 9	44 ± 7	27 ± 2
Ultimate tensile strength [MPa]	210 ± 1	90 ± 10	66 ± 10	31 ± 3
Strain at fracture [%]	0.073 ± 0.003	0.041 ± 0.003	0.036 ± 0.005	0.019 ± 0.001

The composites with fiber orientation angles of UD 0°, 30°, 45°, 60° is called UD0, UD30, UD45, UD60, respectively. PLS and UTS indicates the proportional limit strength (PLS) and ultimate tensile strength (UTS), respectively. The fiber volume fraction of SiC/SiC composites fabricated was about 45%. SiC/SiC composites fabricated have >2.8 g/cm^3 of the balk density regardless of fiber orientation angle. Figure 1 shows elastic modulus and proportional limit strength of SiC/SiC composites fabricated with various fiber orientation angles. Elastic modulus and PLS of UD0 was the most highest. Both of elastic modulus and PLS tend to decrease with increasing of fiber orientation angle. Figure 2 shows digital microscope images of SiC/SiC composites fabricated with various fiber orientation angles after tensile tests. Some fiber pull-outs were observed on UD0 (Figure 2(a)). UD30, UD45 and UD60 indicated fracture along the fiber reinforce direction (Figure 2(b)–(d)). Figure 3 shows SEM images of fracture surface of SiC/SiC composites fabricated with various fiber orientation angles after tensile tests. In the UD0, some fiber pull-outs in the fiber-bundle unit were observed (Figure 3(a)). On the other hands, inter-laminar detachment fracture along the fiber directions was observed in the UD30, UD45 and UD60 (Figure 3 (b)–(d)).

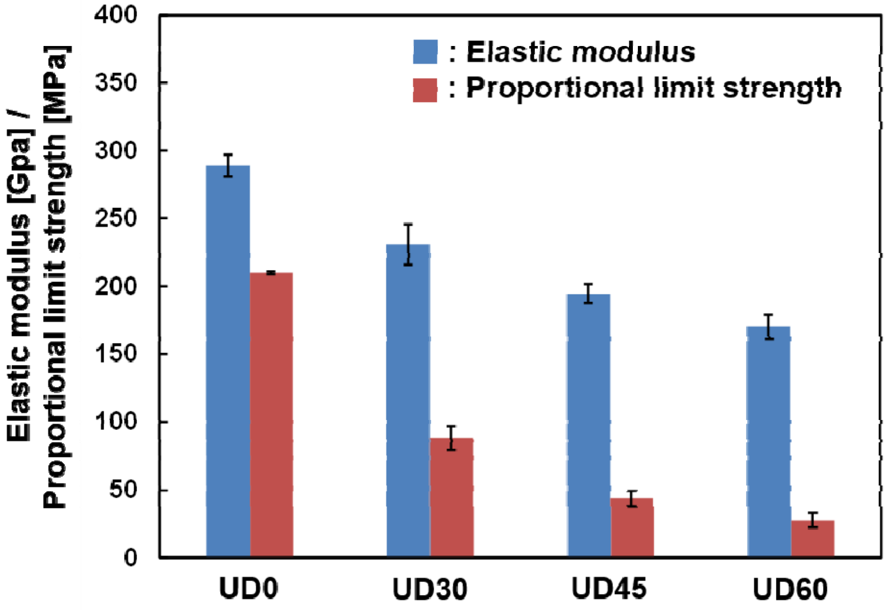

Figure 1. Elastic modulus and proportional limit strength of SiC/SiC composites fabricated with various fiber orientation angle.

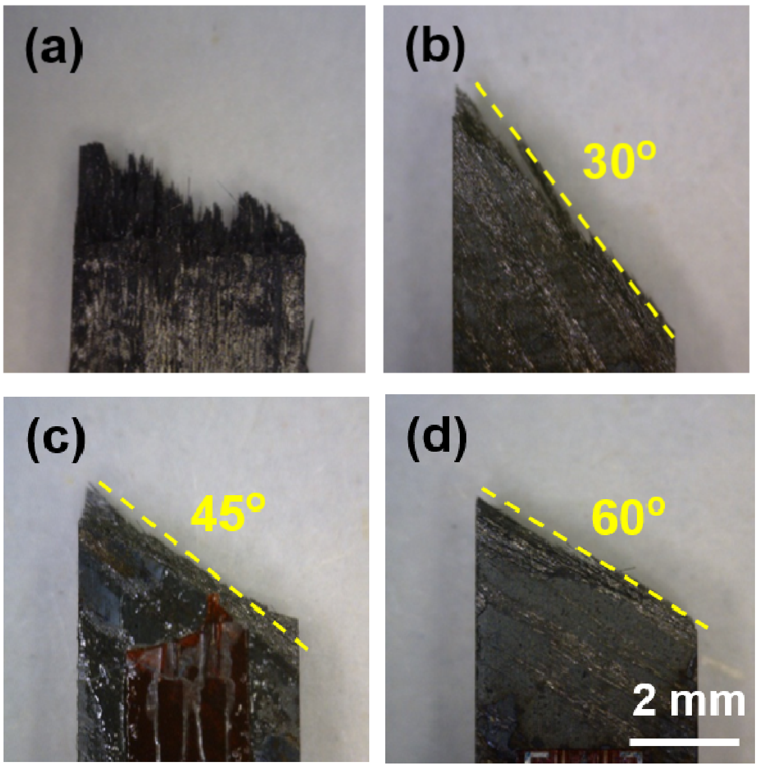

Figure 2. Digital microscope images of SiC/SiC composites fabricated with various fiber orientation angle after tensile test: (a) UD0, (b) UD30, (c) UD45, (d) UD60.

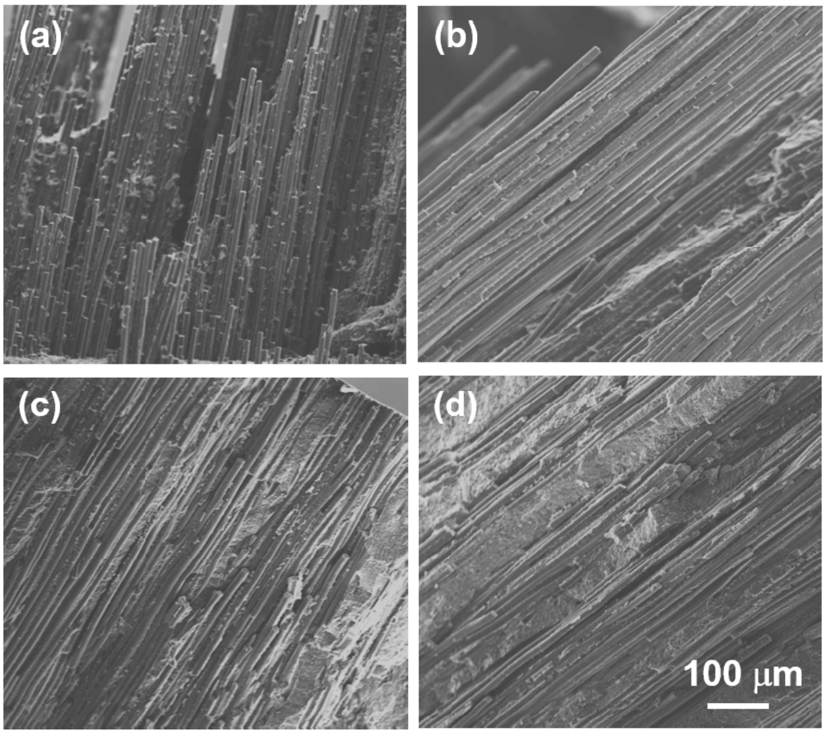

Figure 3. SEM images of SiC/SiC composites fabricated with various fiber orientation angle after tensile test: (a) UD0, (b) UD30, (c) UD45, (d) UD60.

To evaluate correlation of the experimental results and the prediction strength by strength anisotropy prediction theory, the maximum normal stress theory and the Tsai-Hill criterion were applied. The basic equations of the maximum normal stress theory can be described as:

Maximum normal stress theory;

$$\sigma_\theta < \frac{F_{T,PLS}}{\cos^2\theta} \tag{1}$$

$$\sigma_\theta < \frac{F_{TL,PLS}}{\sin\theta\cos\theta} \tag{2}$$

$$\sigma_\theta < \frac{F_{L,PLS}}{\sin^2\theta} \tag{3}$$

where F_T is tensile strength in the fiber orientation angle 0^o, F_{TL} is in-plan shear strength, and F_L is inter-laminar detachment strength. Note that the axial T and L correspond to the directions parallel and perpendicular to the fiber longitudinal direction, respectively. The maximum normal stress has assumed to cause failure when any of stress of each equation (1), (2) or (3) reached failure limits. $F_{TL, PLS}$ and $F_{L, PLS}$ of general NITE-SiC/SiC composites by several experimental methods have been reported by T. Nozawa et al. [13]. T. Nozawa et al. reported that $F_{TL, PLS}$ by the Iosipescu method and

$F_{L,\ PLS}$ by the trans-thickness tension method of UD NITE-SiC/SiC composites are 52 ± 7 MPa and 19 ± 2 MPa, respectively. The reinforcements and fiber/matrix interphase of reference materials are highly crystallized and near-stoichiometric SiC fibers and the pyrolytic carbon, respectively. The reference and the fabricated materials were based on the NITE process and a fiber-architecture of both materials was same in the UD type. The tensile strength of reference material in the fiber direction UD 0^o was 160 ± 24 MPa. The tensile strength of fabricated material is a little higher than reference material. Here, as a simple parameter study, F_{TL} and F_L of fabricated material were assumed to be determined by considering data scatter of F_{TL} and F_L of reference material. The strength anisotropy predictions by prediction theories were discussed by case 1 and case 2. The case 1 and the case 2 was defined as calculation results utilizing minimum scatter of reference material and maximum scatter of that, respectively. The case 1 was calculated as $F_{T,\ PLS}$ = 210 MPa, $F_{TL,\ PLS}$ = 45 MPa and $F_{L,\ PLS}$ = 17 MPa. The case 2 was also calculated as $F_{T,\ PLS}$ = 210 MPa, $F_{TL,\ PLS}$ = 59 MPa and $F_{L,\ PLS}$ = 21 MPa. Figure 4 shows an anisotropy map developed by maximum normal stress theory. The dotted line and solid line indicates the case 1 and the case 2, respectively. The experimental results in each fiber orientation angle were consistent with both of the case 1 and the case 2. UD30, UD45 and UD60 are estimated inter-laminar detachment failure from this anisotropy map. The failure mode of UD30, UD45 and UD60 was consistent with fracture surface observation results. The strength in the high fiber orientation angle indicates the relative low strength. In the case of fiber orientation angle 60^o, the strength was about 40% strength comparing with the strength in the fiber orientation angle 0^o. This reason is that failure in the high fiber orientation angle is dominated by inter-laminar detachment failure. Thus, if SiC/SiC composite products are fabricated, fiber architecture design for suppression of the failure by tensile stress in the inter-laminar detachment angle is very important.

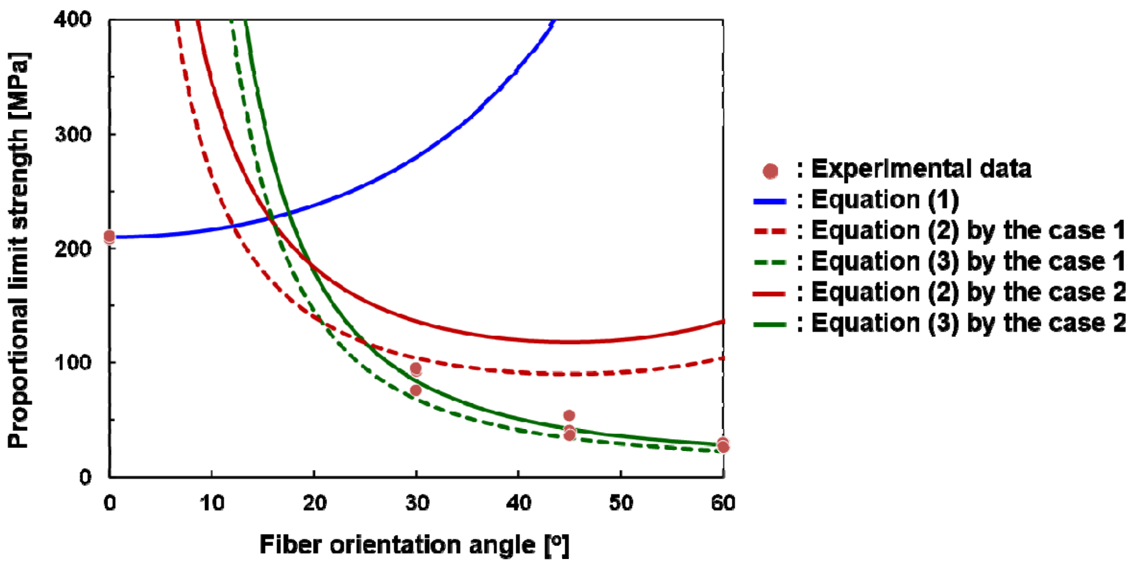

Figure 4. Anisotropy map by the maximum normal stress theory.

Tsai-Hill criterion;

The basic equation of the Tsai-Hill criterion can be described as:

$$\sigma_\theta = \left[\frac{\cos^4 \theta}{F_{T,PLS}{}^2} + \left(\frac{1}{F_{TL,PLS}{}^2} - \frac{1}{F_{L,PLS}{}^2} \right) \sin^2 \theta \cos^2 \theta + \frac{\sin^4 \theta}{F_{L,PLS}} \right]^{-\frac{1}{2}} \quad (4)$$

The Tsai-Hill criterion is one of the criteria considering the mixed failure modes [14]. In generally, since structural materials are often used under the complex stress, it is important to consider application of the criteria by the mixed failure modes. It is well known that prediction values in the low fiber orientation angle side are different between the normal stress theory and the criteria by mixed failure modes such as Tasi-Hill criterion. It is also reported that the prediction values by the Tasi-Hill criterion were consistent with experiment results in the off-axial tensile test than that by the normal stress theory [15,16]. In order to more accurately understand strength anisotropy, it is necessary to evaluate strength anisotropy by the normal stress theory as well as the criteria by mixed failure modes. As a first step of the anisotropy evaluation for the industrialization grade NITE-SiC/SiC composites, the Tasi-Hill criterion were investigated in this study. Although the Tasi-Hill criterion does not consider the compression mode, this might be rationalized in this tensile mode only case. The anisotropy map developed by the Tsai-Hill criterion is shown as Figure 5. The dotted line and solid line indicates the case 1 and the case 2, respectively. In the case of case 1, although the experimental results of UD45 and UD60 were consistent with the prediction values, that of UD30 were a little different. On the other hand, the prediction values by case 2 were almost consistent with the experimental results. F_{TL} and F_L of fabricated materials are thought to close to case 2 than case 1 because the mechanical properties of fabricated materials are higher than that of reference materials. This result is suggested that the strength anisotropy of UD NITE-SiC/SiC composites is able to predict by Tsai-Hill criterion.

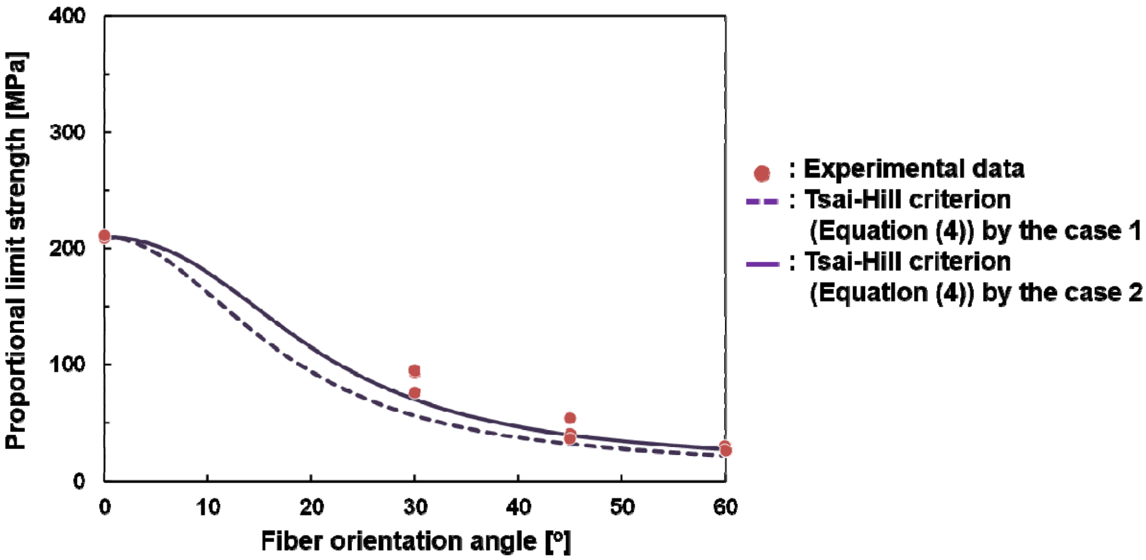

Figure 5. Anisotropy map by the Tsai-Hill criterion.

4. Conclusion

The axial/off-axial mechanical properties of UD NITE-SiC/SiC composites by the industrialization grade process were evaluated by axial/off-axial tensile test. Elastic modulus and proportional limit strength of NITE-SiC/SiC composites tended to decrease with increasing of fiber orientation angle. The experiment results by axial/off-axial tensile test were consistent with the strength anisotropy prediction theories by the maximum normal stress theory and the Tsai-Hill criterion. The failure modes of SiC/SiC composites fabricated with each fiber orientation angle were consistent with fracture surface observation results. Also, the strength anisotropy of UD NITE-SiC/SiC composites was suggested to be able to be predicted by Tsai-Hill criterion. The basic strength anisotropy of UD SiC/SiC composites was understood from correlation evaluation of mechanical properties, fracture surface observation and the strength anisotropy prediction theories.

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Conflict of Interest

The authors declare that there is no conflict of interest regarding the publication of this manuscript.

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