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Mechanical properties of silicon impregnated C/C composite material at elevated temperature

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Abstract—Fracture tests were conducted for silicon impregnated C/C composite material at elevated temperature. Two kinds of carbon fiber orientations were used; one was (0/90) cross-ply and the other was (+45/−45) cross-ply. The fracture mode of this material is discussed compared with that of the conventional C/C composite material. The roughness of the fracture surface of silicon impregnated C/C composite material is much smaller than that of C/C composite material. The delamination between layers is not so remarkable in the fracture of silicon impregnated C/C composite material while it is predominant on the fracture surface of C/C composite material. The anisotropy of the tensile strength of silicon impregnated C/C composite material is small compared with that of conventional C/C composite material.

Keywords: Silicon impregnated C/C composite; C/C composite; fracture; fracture surface; delamination.

1. INTRODUCTION

C/C composite material has potential as an aerospace structural material because of its specific strength and heat resistance. However, this material will be easily oxidized in the ambient atmosphere at elevated temperature, which prevents the wide practical use of the material. Recently, silicon impregnated C/C composite material [1] has been developed in order to add oxidation-resistance to the conventional C/C composite material. However, the mechanical properties of this material have scarcely been investigated. Hatta *et al.* [2] investigated the oxidation and mechanical behavior of SiC (silicon carbide) coating on the surface of C/C composite material formed by silicon melt impregnation method [3] and has shown that the interlaminar shear strength is improved but the bending strength is degraded by the

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silicon impregnation. The SiC coating layer in the study by Hatta *et al.* [2] can be regarded as almost the same as silicon impregnated C/C composite material [1] from the standpoint of fabrication. In the present study, the fracture mode of the silicon impregnated C/C composite material at elevated temperature is investigated and the difference between the mechanical properties and those of the C/C composite material is discussed.

As specimens of the C/C composite material and the silicon impregnated C/C composite material, two types of the carbon fiber direction were used; one was (0/90) cross-ply and the other was (+45/−45) cross-ply. Tensile tests were performed until fracture in an electric furnace filled with argon gas at various temperatures. Tensile strength of each specimen was measured and macroscopic overview of each fractured specimen was also investigated. Furthermore, the three-dimensional profiles of the fracture surfaces were observed in order to clarify the difference between the fracture modes of silicon impregnated C/C composite material and the conventional C/C composite.

2. MATERIALS

In this study, a new material, namely, silicon impregnated C/C composite material was tested as well as the conventional C/C composite.

As the conventional C/C composite, cross-ply material manufactured by Across Co. Ltd., Japan through the preformed yarn method [4] was used. In this fabrication method, a preformed yarn consisting of a bundle of carbon fibers in a matrix precursor of coke and pitch binder powders is encased in a flexible thermoplastic sleeve for better penetration of the binder into the carbon fiber bundle; this is then woven into sheets and chopped to fill molds which produce C/C composite material after hot pressing. In this material, there remain periodically situated vacancies at boundaries among preformed yarns as shown in Fig. 1a. The carbon fiber ratio and the density of the material used in this study is 40% in volume and 1.7 g/cm^3 respectively. The thickness of each layer is approximately 0.5 mm.

Recently, silicon impregnated C/C composite material was developed by NGK Insulators, Ltd., Japan [1]. This material is made of the same C/C composite as the above. Silicon is infiltrated into the vacancies among preformed yarns of the C/C composite to form SiC around each preformed yarn and boundary between layers as shown in Fig. 1b. Therefore, this material can be regarded as composed of three phases, namely, C, Si and SiC. The back-scattered electron image of this material is shown in Fig. 1c in which white, gray and black regions represent Si, SiC and C regions respectively. The volume fractions of C, SiC and Si are 75%, 23% and 2%, respectively, and the density is 2.0 g/cm^3. It has been reported that the oxidation resistance, the tribological properties and the anisotropy of the thermal expansion coefficient of this material are much improved compared with the original C/C composite material [1, 2].

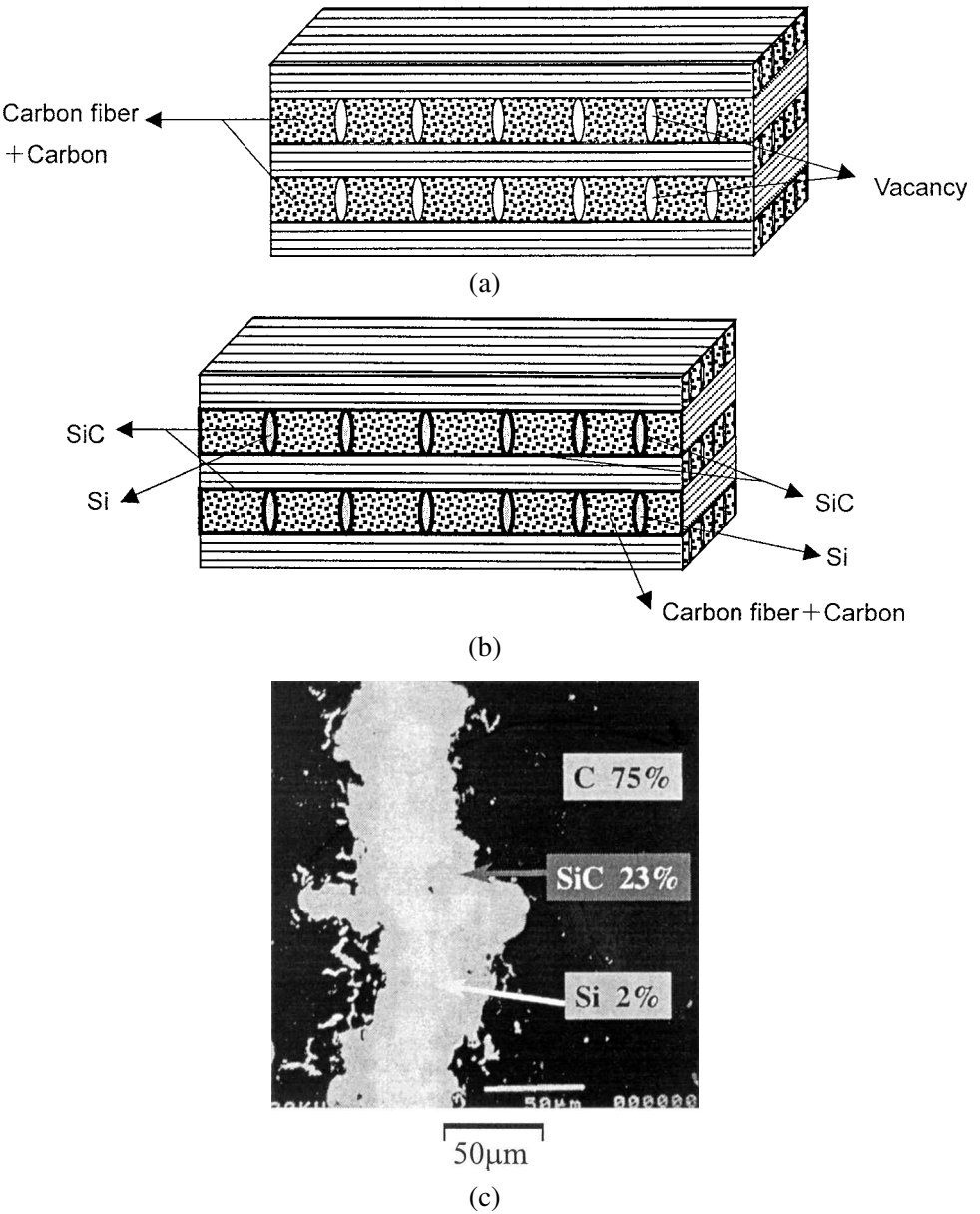

Figure 1. C/C composite material and silicon impregnated C/C composite material. (a) Schematic illustration of C/C composite material made by the preformed yarn method. (b) Schematic illustration of silicon impregnated C/C composite material made of the same C/C composite as the above. (c) BEI of the silicon impregnated C/C composite material.

3. EXPERIMENTATION

3.1. Specimen

The shape and dimensions of the specimen are shown in Fig. 2. The thickness of the specimen was 4 mm and the number of layers was approximately 8. Two types

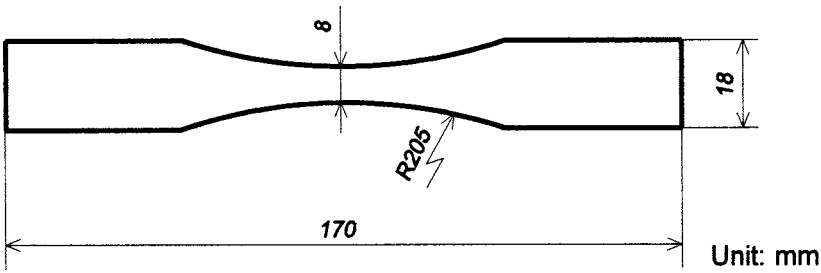

Figure 2. Shape and dimensions of specimen (thickness = 4 mm).

of carbon fiber direction were used; one was (0/90) cross-ply and the other was (+45/−45) cross-ply. It is well known that the strength of C/C composite material becomes high as the temperature increases. Therefore, the necking is given to each specimen so that it does not break at the grip but breaks at its center even at elevated temperature.

3.2. Tensile test

Tensile tests at various temperatures were conducted using the universal testing machine of MTS Systems Corporation, USA. Specimens were heated to the desired temperatures (1200°C, 1600°C, 2000°C) in the electric furnace filled with argon gas and extended with the displacement rate 0.04 mm/min until fracture. Temperature was measured using a thermocouple (below 1600°C) or an infrared thermometer (above 1600°C). Fracture tests in the ambient atmosphere at room temperature were also conducted.

3.3. Observation of the fractured specimen

At first, the overview of each fractured specimen was observed. Next, fractured specimens were embedded in polyester resin and sliced at an interval of 0.8 mm parallel to the longitudinal direction and perpendicular to each layer. Subsequently, three-dimensional profiles of the fractured specimens were observed quantitatively.

4. RESULTS AND DISCUSSION

Examples of overview of the fractured C/C composite material and silicon impregnated C/C composite material are shown in Figs 3 and 4, respectively. Figures 5 and 6 are the slices of fractured specimens of each material embedded in polyester resin cut along the longitudinal direction perpendicular to each layer, showing the feature of the fracture surface typically. At every temperature, regarding the C/C composite material, many long pull-outs are observed on its fracture surface and it is expected that the fracture is governed by the delamination at the interface between layers, while on the fracture surface of the silicon impregnated C/C composite material, there are fewer and shorter pull-outs than the corresponding C/C composite

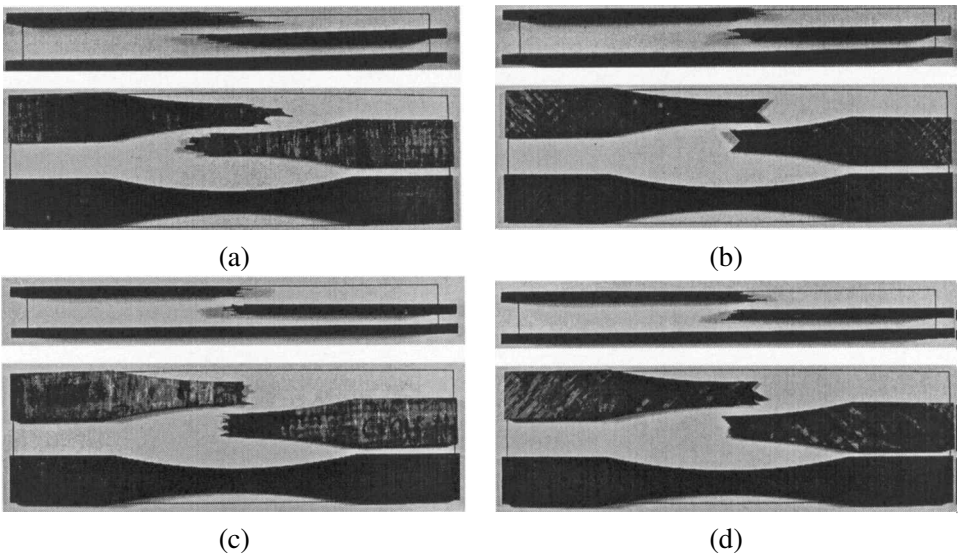

Figure 3. Examples of the overview of fractured C/C composite material. (a) (0/90) cross-ply at room temperature; (b) (+45/−45) cross-ply at room temperature; (c) (0/90) cross-ply at 1600°C; (d) (+45/−45) cross-ply at 1600°C.

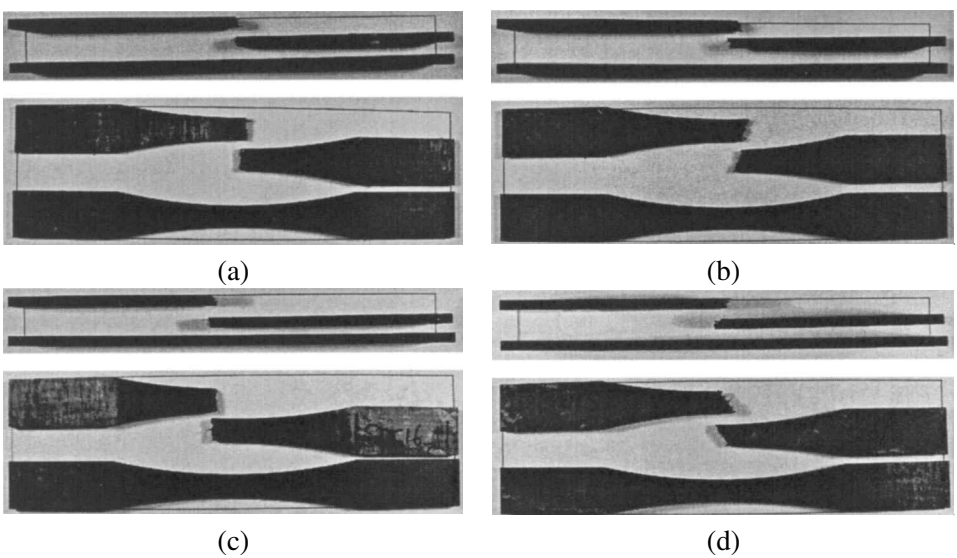

Figure 4. Examples of the overview of fractured silicon impregnated C/C composite material. (a) (0/90) cross-ply at room temperature; (b) (+45/−45) cross-ply at room temperature; (c) (0/90) cross-ply at 1600°C; (d) (+45/−45) cross-ply at 1600°C.

material. Furthermore, on the fracture surfaces of the (+45/−45) cross-ply C/C composite specimens, it is observed that the fracture occurred at vacancies between carbon fiber yarns, parallel to the carbon fiber direction of each layer; therefore,

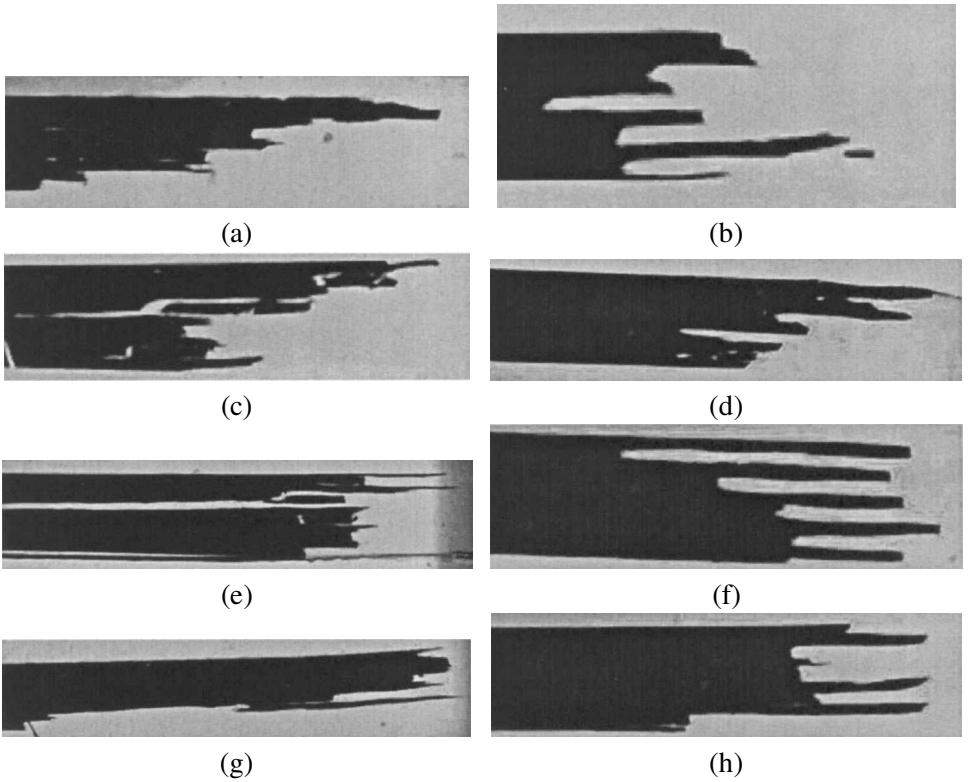

Figure 5. Examples of slices of fractured specimen of C/C composite material cut along the longitudinal direction perpendicular to each layer. (a) (0/90) cross-ply at room temperature; (b) (+45/−45) cross-ply at room temperature; (c) (0/90) cross-ply at 1200°C; (d) (+45/−45) cross-ply at 1200°C; (e) (0/90) cross-ply at 1600°C; (f) (+45/−45) cross-ply at 1600°C; (g) (0/90) cross-ply at 2000°C; (h) (+45/−45) cross-ply at 2000°C.

the fracture directions in the respective layers differ one from another, while in the case of the (+45/−45) cross-ply silicon impregnated C/C composite specimens, the fracture directions are almost the same in all layers. In other words, in some layers of the silicon impregnated C/C composite materials, fracture occurred in a different direction from the carbon fiber direction.

Figures 7 and 8 show the three-dimensional profiles of the fracture surfaces obtained by observing the slices of the fractured specimens embedded in polyester resin. It is obvious from the figures that fracture surfaces of silicon impregnated C/C composite material are less rough than those of C/C composite material for both fiber directions. Fracture surface area is calculated from these figures and is shown in Fig. 9 as the function of the temperature for both composite materials. In this figure, fracture surface area is normalized by the area of the narrowest cross-section of the original specimen (= 32 mm^2). As a result, it has been shown that the fracture surface area of the silicon impregnated C/C composite material is much smaller than that of the C/C composite material. This is due

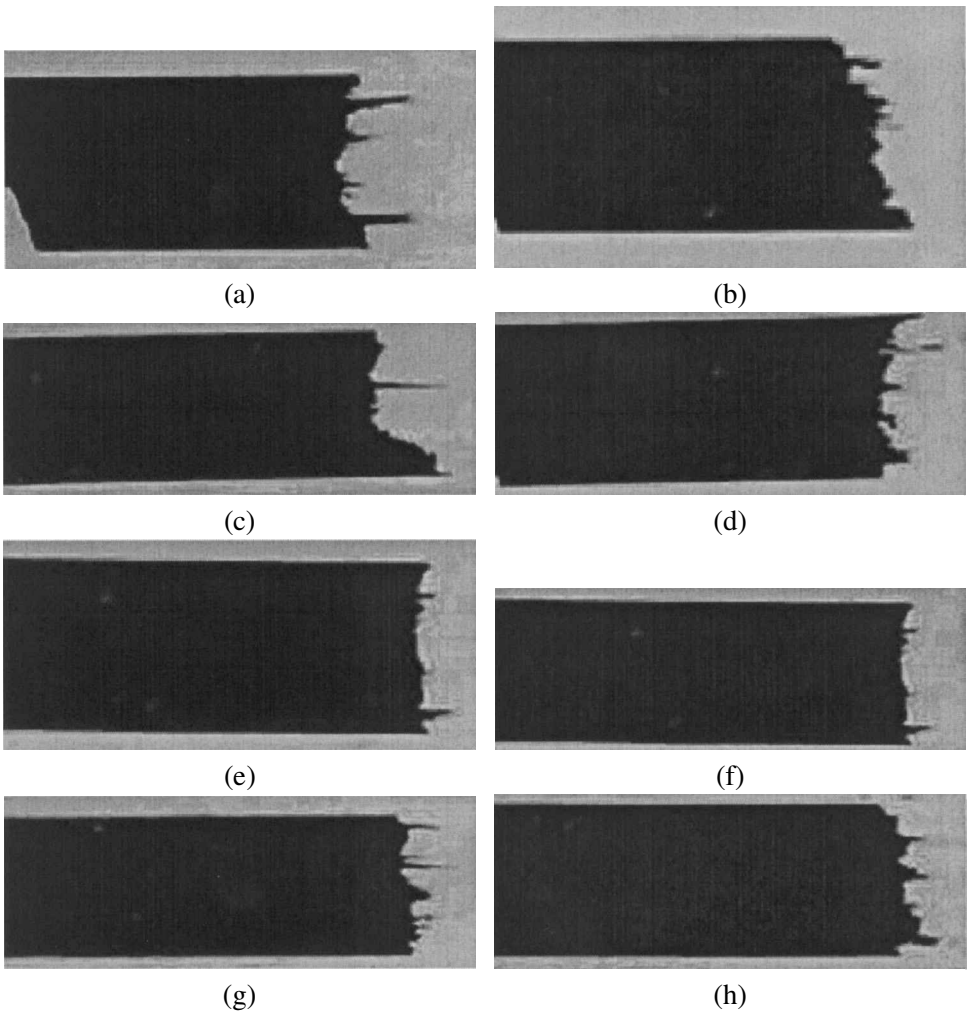

Figure 6. Examples of slices of fractured specimen of silicon impregnated C/C composite material cut along the longitudinal direction perpendicular to each layer. (a) (0/90) cross-ply at room temperature; (b) (+45/−45) cross-ply at room temperature; (c) (0/90) cross-ply at 1200°C; (d) (+45/−45) cross-ply at 1200°C; (e) (0/90) cross-ply at 1600°C; (f) (+45/−45) cross-ply at 1600°C; (g) (0/90) cross-ply at 2000°C; (h) (+45/−45) cross-ply at 2000°C.

to the fact that the interlaminar fracture surface area is large in the conventional C/C composite material because the interlaminar strength of the conventional C/C composite material is small. However, the silicon impregnated C/C composite material is strengthened at the interface between layers and the total fracture surface area becomes smaller than that of the conventional C/C composite material. Furthermore, the temperature dependence of the fracture surface area of the silicon impregnated C/C composite material is small while that of the C/C composite material is large.

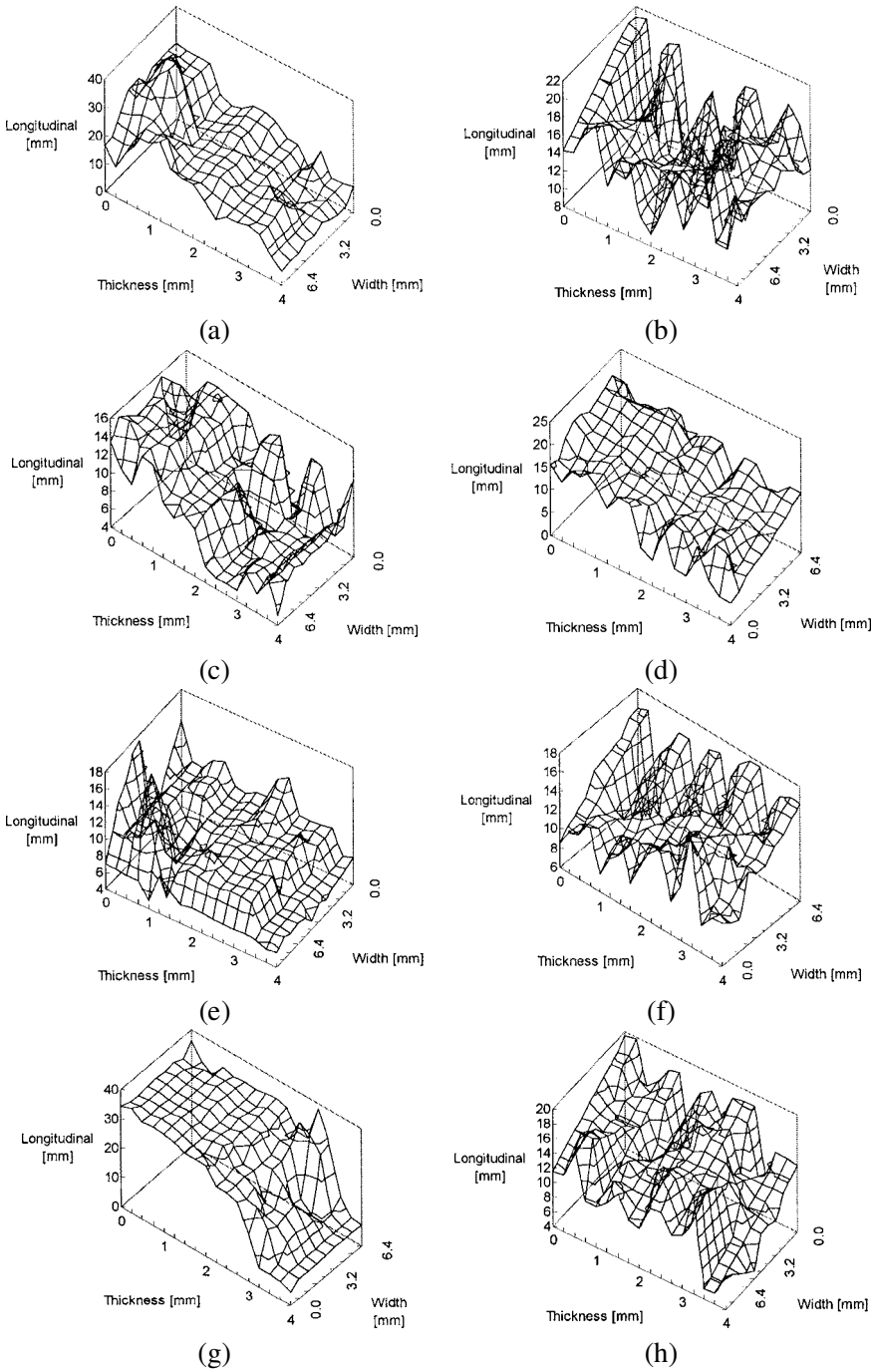

Figure 7. Three-dimensional fracture surfaces of C/C composite material. (a) (0/90) cross-ply at room temperature; (b) (+45/−45) cross-ply at room temperature; (c) (0/90) cross-ply at 1200°C; (d) (+45/−45) cross-ply at 1200°C; (e) (0/90) cross-ply at 1600°C; (f) (+45/−45) cross-ply at 1600°C; (g) (0/90) cross-ply at 2000°C; (h) (+45/−45) cross-ply at 2000°C.

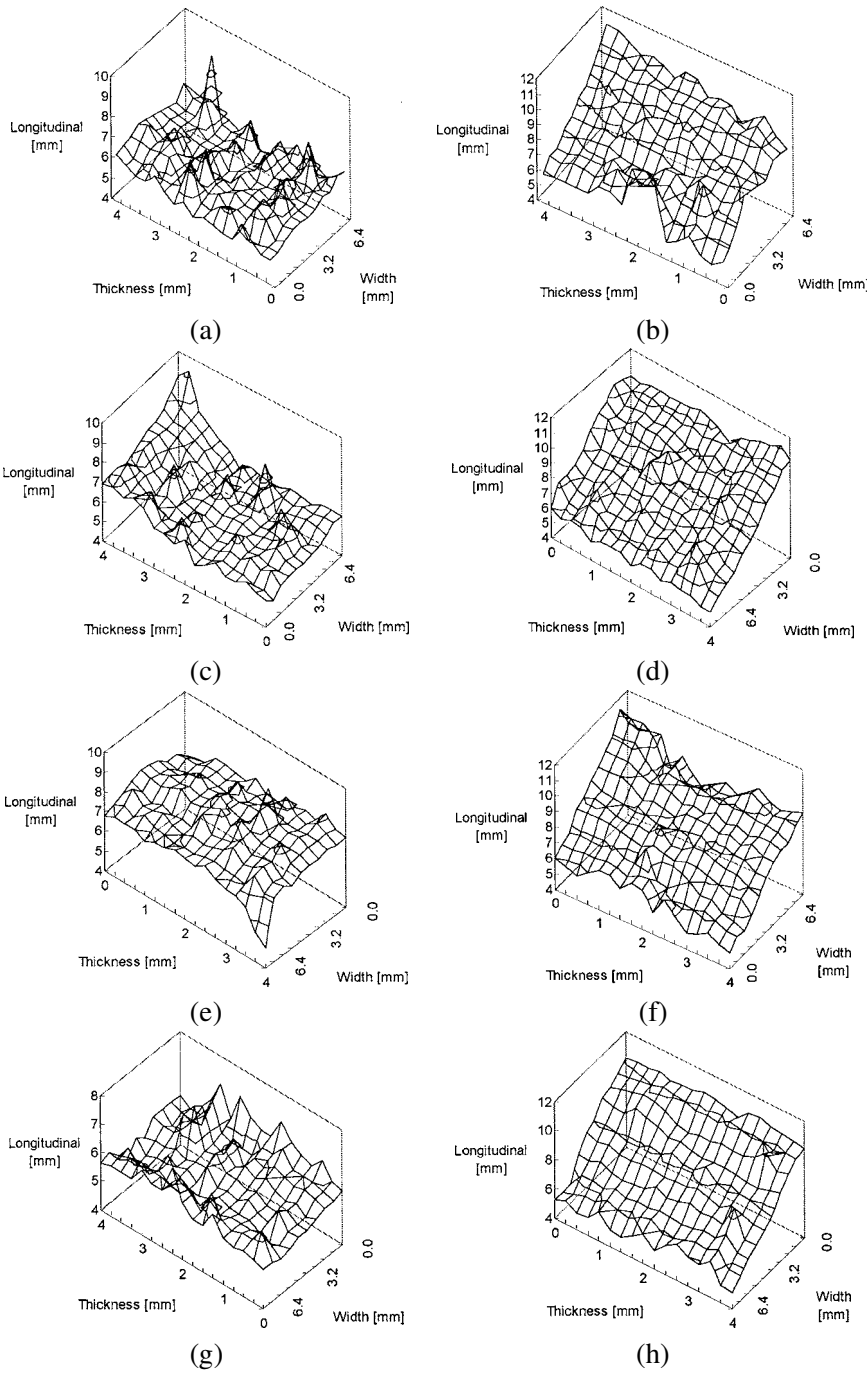

Figure 8. Three-dimensional fracture surfaces of silicon impregnated C/C composite material. (a) (0/90) cross-ply at room temperature; (b) (+45/−45) cross-ply at room temperature; (c) (0/90) cross-ply at 1200°C; (d) (+45/−45) cross-ply at 1200°C; (e) (0/90) cross-ply at 1600°C; (f) (+45/−45) cross-ply at 1600°C; (g) (0/90) cross-ply at 2000°C; (h) (+45/−45) cross-ply at 2000°C.

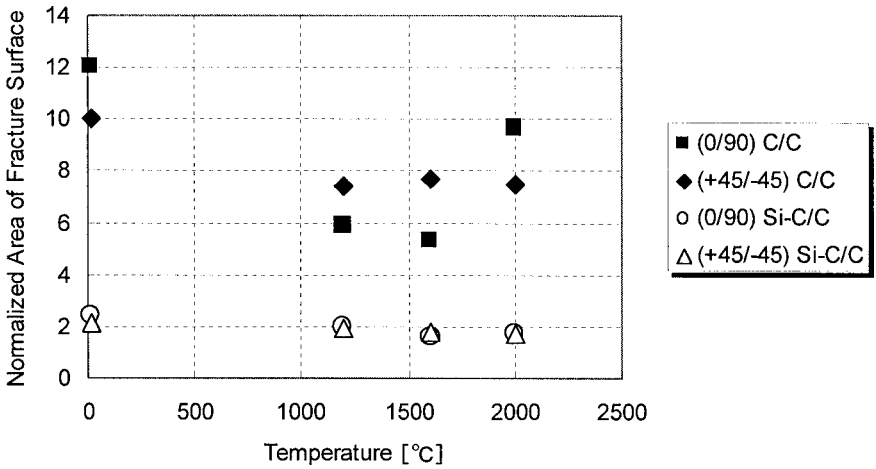

Figure 9. Temperature dependence of the area of fracture surface of each specimen.

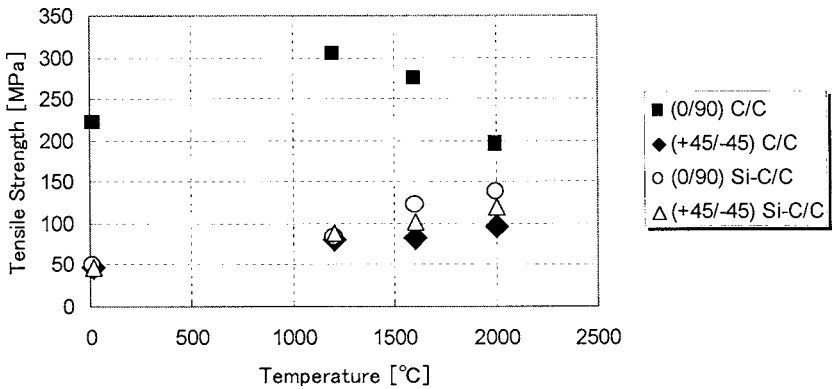

Figure 10. Temperature dependence of the tensile strength of each specimen.

Figure 10 shows the relationship between the tensile strength and temperature for C/C composite and silicon impregnated C/C composite material. The strength of (0/90) cross-ply C/C composite material is much larger than that of any other type of specimen. C/C composite of (0/90) cross-ply can bear the tensile load by the fibers of 0° direction; however, each layer of (+45/−45) cross-ply C/C composite can bear little tensile load by itself because of the vacancies at the boundaries among preformed yarns but transmit tensile load at the boundaries between layers. Therefore, the strength of (+45/−45) cross-ply is much smaller than that of (0/90) cross-ply for C/C composite. Regarding silicon impregnated C/C composite, as the vacancies are filled with silicon, the effect of the fiber direction on the tensile strength is small compared with C/C composite. In general, the tensile strength at elevated temperature is larger than that at room temperature for silicon impregnated C/C composite material as well as the conventional C/C composite material. It is noteworthy that the tensile strength of the silicon impregnated

C/C composite material of (0/90) cross-ply is much smaller than that of the corresponding conventional C/C composite material for every temperature. This is probably due to the fact that each carbon fiber becomes slenderer by silicon impregnation because a part of it changes into SiC.

5. CONCLUSIONS

In this study, fracture mode of silicon impregnated C/C composite material was investigated compared with that of the conventional C/C composite. It has been shown that the delamination at the interface between layers is predominant in the fracture of C/C composite material while in the fracture of silicon impregnated C/C composite material, the delamination between layers is not so important. In general, the fracture surface area of silicon impregnated C/C composite material is much smaller than that of C/C composite. The temperature dependence of the fracture surface is not so obvious in silicon impregnated C/C composite material but the strength is larger at elevated temperature than at the room temperature in both composite materials. The anisotropy of the strength of C/C composite is large; however, that of silicon impregnated C/C composite material is small.

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