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THERMAL SHOCK DAMAGE MECHANISM OF FIBER BONDED CERAMICS

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ABSTRACT

Thermal shock behavior of unidirectionally reinforced Si-Ti-C-O fiber bonded ceramics was experimentally examined. Water quench tests were carried out with various temperature differences (ΔT) up to 800K. Macroscopic cracks were introduced with ΔT of 600K, although no degradation was observed in Young's modulus. With larger ΔT, large cracks were introduced accompanied by the degradation of Young's modulus. SEM observation revealed that microscopic damage near the surface occurred even when ΔT was less than 600K. The microscopic damage might play an important role to release the thermal stresses induced by the thermal shock.

KEYWORD

Thermal Shock, Water Quench Test, Fiber Bonded Ceramics, Micro-damage

INTRODUCTION

Various types of ceramic matrix composites (CMC) have been developed and studied for high temperature applications. Among them, Si-Ti-C-O fiber bonded ceramics (FBC) is one of the promising materials, which possess superior high temperature mechanical properties and long-term durability at elevated temperature [1].

In actual operation conditions, the materials will be exposed to severe temperature changes, which will induce large thermal stresses. Because of this, knowledge of thermal shock resistance is indispensable for structural design. However, thermal shock behavior of the CMCs is generally complicated compared with monolithic ceramics [2-4], and is not fully understood yet. In this study, water quench tests were carried out on the unidirectionally reinforced (UD) FBC to investigate micro- and macroscopic damages induced by the thermal shock. Young's modulus before and after thermal shock tests were also measured to clarify the effect of the thermal shock induced damage on the mechanical properties of the UD FBC.

EXPERIMENTAL PROCEDURE

Materials

Material used in this study was unidirectionally (UD) reinforced Si-Ti-C-O fiber bonded ceramics (FBC) manufactured by UBE Industries [5]. Continuous Si-Ti-C-O fiber was used as the starting material for the manufacturing of the FBC. The fiber was first heat treated in air to form an oxide layer on the fiber surface. The pre-oxidized Si-Ti-C-O fibers were hot-pressed to pack interstices by the oxide material on the fiber surface. A thin carbon layer was formed between the fiber and the oxide layer after the hot-pressing [1]. Typical optical micrograph of as-received UD FBC is shown in Fig.1. Fiber volume faction was approximately 90%, which was much higher than those of other CMCs.

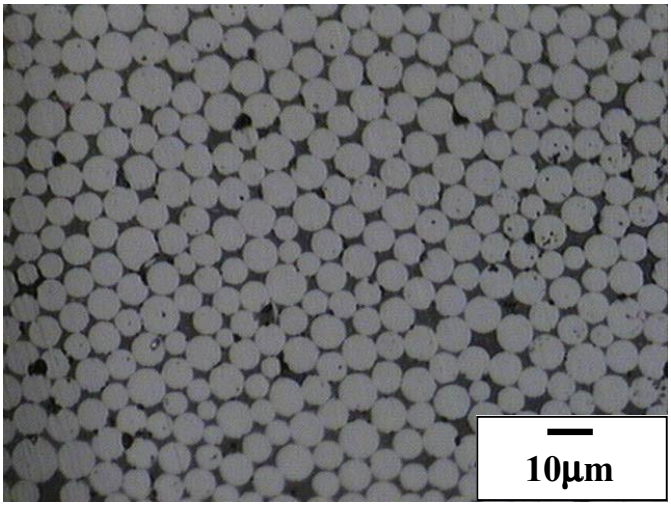

Fig.1 Cross-sectional view of as-received UD FBC.

Thermal Shock Test

The UD FBC plate was machined into rectangular bars, and the wedge made of the UD FBC was bonded to the tip of the specimen. The wedge was necessary to avoid formation of thick air layers on the specimen surface when the specimen entered into the water. As already reported [6,7], heat transfer at specimen surface was increased by attaching a wedge due to change in the boiling condition on the surface. This resulted in more severe thermal shock condition compared with a specimen without a wedge. In the thermal shock test, the specimen was heated in an inert atmosphere and kept for 15 min. at a given temperature (473 K ~ 800 K). Then, the specimen was made to fall freely and stopped in the water bath. Both falling height and falling depth from the water surface were set to 600 mm following the Japanese standard for the thermal shock test of ceramics (JIS-R1615). In order to investigate macro- and microscopic damage induced in the UD FBCs, the dye penetrant test and the SEM observation were carried out on the surface of the thermally shocked specimens. Cross-sections were also observed to estimate depth of the damaged area.

Finite element analyses (FEA) were carried out to estimate thermal stresses induced in the FBC. Two dimensional transient thermal stress analysis was carried out by modeling cross-section of the specimen. Fracture mechanical analysis also carried out to clarify the macroscopic fracture criteria. The boundary conditions were determined by experiments using rod-shaped silver specimens with and without the wedge. A thermocouple was set in the silver specimens to measure the temperature change during the thermal shock test. Because the temperature difference between the center and the surface of the silver specimen was less than 8%, the heat flow at the surface could be estimated assuming uniform temperature distribution in the silver specimen. The heat flow on each silver specimen surface was assumed to correspond to the top surface or the side surface of the FBC specimen.

RESULTS AND DISCUSSION

Thermal Shock Damage Observation

Figure 2 shows a typical thermal shocked specimen (ΔT=800K). Two types of cracks along the fiber direction were introduced in the UD FBC. Type A crack was first observed when ΔT was 600K. With increasing ΔT, the length of Type A crack was increased and Type B crack appeared on the side surface of the specimen. With these results, the critical temperature difference (ΔT_C) of the UD FBC could be determined as 600K.

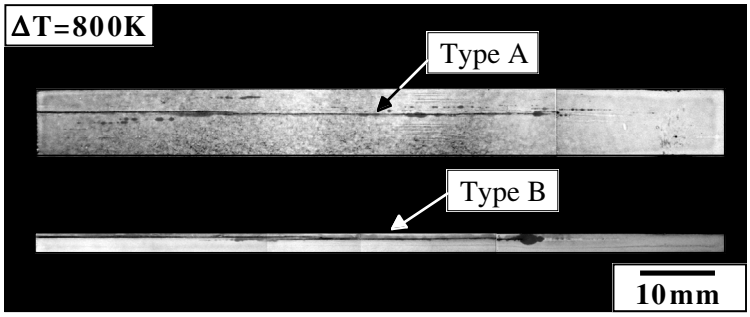

Fig.2 Cracks introduced by thermal shock

Change in the longitudinal Young's modulus was measured by the four-point flexure tests as shown in ***Fig.3***. Normalized electrical resistance is also shown in the figure. When ΔT was lower than 600K, the Young's modulus after the thermal shock test was the same with that of the as-received specimen. However, when ΔT was above 600K, the Young's modulus dramatically decreased with increasing ΔT. This was due mainly to formation of Type B cracks in the UD FBC. The electrical resistance also changed drastically above 600K. At ΔT of 800K, the electrical resistance showed ten times larger than that of as-received specimen. These results suggested that the electrical resistance be closely related to the damage induced in the UD FBC. If we pay attention to ΔT lower than 600K, the electrical resistance gradually increased with increasing ΔT, even though no macroscopic damages were observed in the UD FBC.

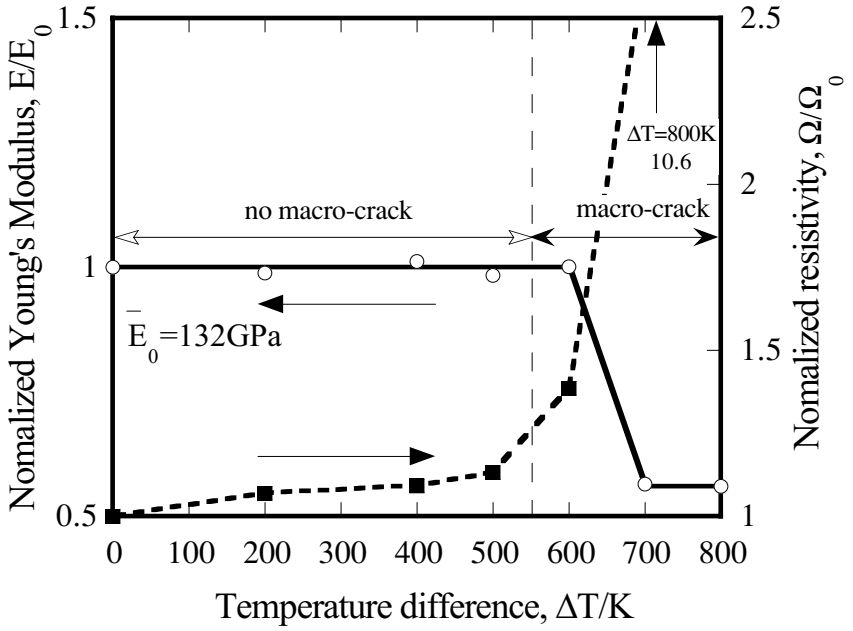

Fig.3 Change in Young's modulus and electrical resistance

Figure 4 shows the typical cross-sectional views of the as-received and the thermally shocked specimens. Even in the specimens tested at the lower ΔT than 600K, fiber spacing near the surface increased. This must be a microscopic damaged area induced by the thermal shock. ***Figure 5*** shows a microscopic damage area at a higher magnification. Micro-cracks were generated at the fiber/matrix interface.

The depth of microscopic damage area (t_d) was measured at 20 arbitrary points in each specimen, and plotted against ΔT as shown in Fig.12. The t_d increased monotonously with increasing ΔT, and saturated at ΔT_C. These observations suggested that the breakaway of the fibers from the specimen surface and/or delamination at the fiber/matrix interfaces near the surface occur due to the thermal shock even below the ΔT_C. It was also expected that slight increase in the electrical resistance corresponded to formation of the microscopic damage in the UD FBC.

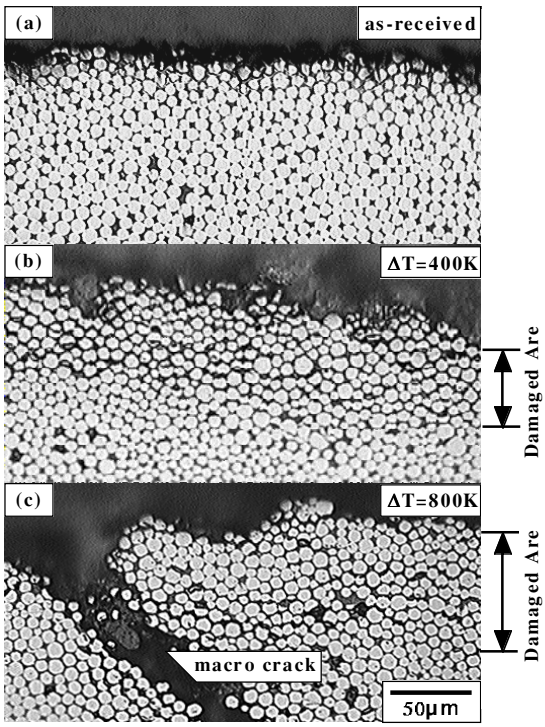

Fig.4 Typical optical micrographs of cross-sectional view of the UD FBC.
(a) before test, (b) ΔT=400K, (c)ΔT=800K

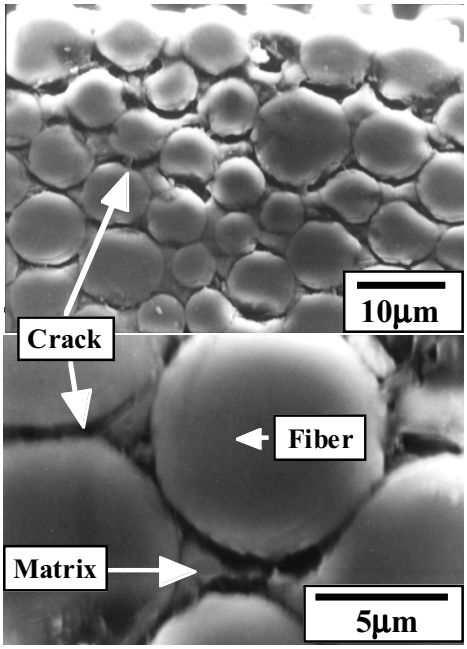

Fig.5 Fiber-matrix interface view of the thermal shocked UD FBC (ΔT=400K).

Thermal Stress Induced by thermal shock

For estimating thermal stress induced in the specimen, finite element analysis was carried out. Linear elastic two dimensional model was used assuming the plane strain condition. The cross section was modeled using 8 nodes isoparametric elements. The transient heat flow can be estimated from the experiments of the silver specimen and considered in the boundary condition.

The calculated maximum thermal stress induced in the UD FBC is shown in Fig.6. Even at ΔT of 200K, the maximum thermal stress was higher than the transverse tensile strength of the UD FBC. These results suggested that macroscopic fracture can not be predicted by the maximum stress criterion. As another approach to predict the macroscopic fracture, fracture mechanical analysis was also carried out by the virtual crack closure method [8]. Energy release rates were calculated for various crack length and various temperature difference. Figure 7 shows the maximum energy release rates in each condition. Results showed that if the initial crack size is less than 100 μm, crack extension will occur at 600K or higher temperature difference. These calculated results well agree with the experimental results. Form

these analyses, it can by concluded that the fracture toughness criterion can be predict the macroscopic fracture. In addition, it is also expected that the microscopic damage observed near the surface of the UD FBC specimen play an important role to relax thermal stress. This must be an unique stress release mechanism to the UD FBCs.

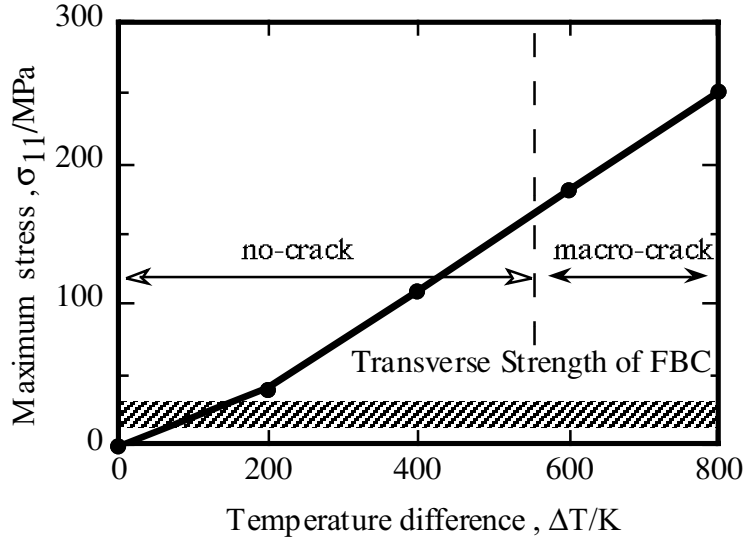

Fig.6 Maximum Thermal Stress Induced in the Specimen

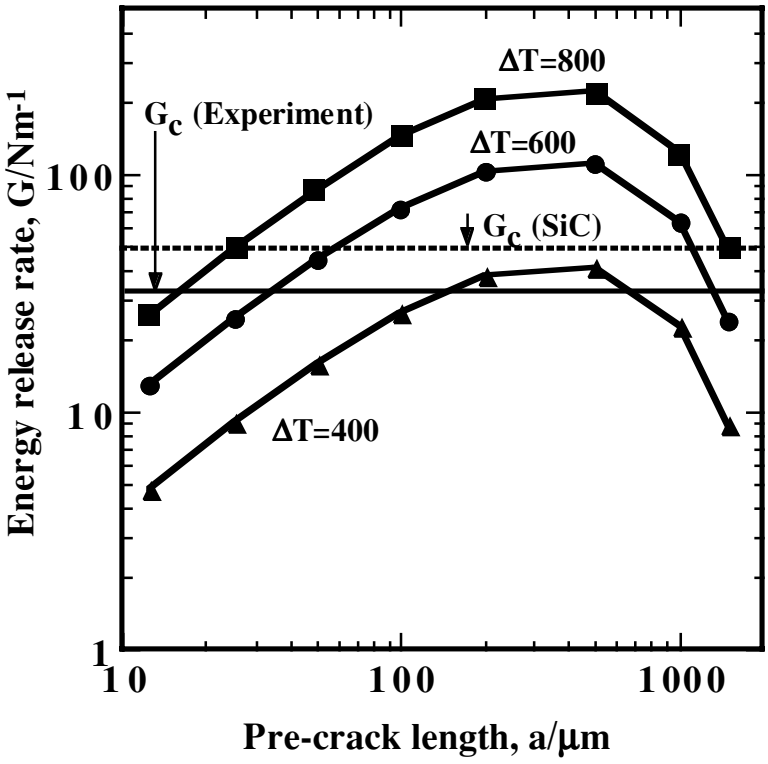

Fig.7 Maximum Energy Release Rate during Thermal shock Test

SUMMARY

The thermal shock behavior of the unidirectionally reinforced Si-Ti-C-O fiber bonded ceramics was experimentally examined, and following results were obtained.

1. The critical temperature difference of the unidirectionally reinforced Si-Ti-C-O fiber bonded ceramics was estimated as 600K, which was much higher than those of monolithic ceramics.
2. Even below the critical temperature difference, microscopic damage such as delamination of fiber/matrix interface and/or breakaway of fibers from the surface occurred in the unidirectionally reinforced Si-Ti-C-O fiber bonded ceramics.
3. Macroscopic fracture was controlled by the fracture toughness criterion rather than the maximum stress criterion.
4. The microscopic damage was expected to release the thermal stresses induced in the material. This must be an unique stress release mechanism.

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