FLEXURAL BEHAVIOR OF C/C COMPOSITES AT HIGH TEMPERATURE

Yoshihiro Sawada[1], Norio Iwashita[1], Kazuhiro Fujita[1], and Akira Shinjo[2]

[1]*Osaka National Research Institute - AIST, MITI*
1-8-31 Midorigaoka, Ikeda, Osaka 563-8577, Japan
[2]*Ohwada Carbon Industrial Co. Ltd.*
2-1-16 Hinodecho, Toyonaka, Osaka 561-0821, Japan

SUMMARY: Unidirectional carbon fiber reinforced carbon composites (UD-C/C composites) were prepared from a high strength type PAN-based carbon fiber and a phenol resin. The mechanical behaviors of C/C composites were measured by four points bending and short beam three points bending tests up to 2600°C. The modulus and proof stress were constant until 1500°C and they were not affected by loading speed, but they measured over 2000°C were decreased at slower loading speed. Decreasing modulus and proof stress, plastic deformation was increased. Bending strength and shear strength were increased with the test temperature. Those strengths were influenced by very little presence the disoriented filaments, which was brought about by fabrication process of the composite.

KEY WORDS: C/C composites, high temperature, modulus, bending strength, proof stress, plastic deformation, interlaminar shear strength

INTRODUCTION

Carbon fibers have high specific modulus and strength and retain these properties to high temperatures in non-oxidizing environments. Carbon and graphite materials don’t melt at ambient pressure and stabilize until sublimation over 3600°C. In addition, they show tremendous thermal shock resistance and the strength increases with temperature. Therefore, carbon fiber reinforced carbons (C/C composites) have the most outstanding potential of mechanical properties from room temperature to high temperature. They are the only material having a potential for structural service over 2000°C, where high specific strength is required in combination with good thermal shock tolerance.

On the structural service of C/C composites at the high temperature environment to utilize fully the potential of the materials, it is necessary to have a complete and accurate description of their mechanical behavior. Their mechanical properties at ambient temperature have been reported in many publications. However, there are few reports on the mechanical behavior at high temperature conditions, particularly over 2000°C, because of the experimental difficulties. The main interest of this work is to deepen knowledge of the mechanical behavior of C/C composites at the high temperature. Present paper describes a method to characterize the mechanical behavior of C/C composites at high temperature and the experimental data collected by four point bending and short beam shear tests on unidirectional C/C composites from 25°C to 2600°C.

EXPERIMENTAL

Specimen

The UD-C/C composites used were prepared from a high strength type PAN-based carbon fiber and a phenol resin. The carbon fiber bundle was immersed in the precursor resin and winded on a steel frame. The unidirectional fiber arrayed sheets were cut out from the frame and were stack up in a metal die. Then, it was hot pressed at 150°C under 29.4MPa for 30 minutes. After then, the obtained CFRP was heat treated up to 180°C in an air circulating oven. The after cured CFRP plates were heat treated in a vacuum furnace up to 1600°C. Moreover, the carbonized plates were densified by impregnating another phenol resin using a vacuum system, curing in an autoclave and carbonizing in vacuo up to 1600°C for 5 cycles. Some of them were additionally heat treated to 3000°C in an Acheson type furnace, burying the densified C/C composites in a high purity grade graphite granule. The final heat treatment temperatures were 1600°C or 3000°C.

After the heat treatment, specimens were cut out aligned to the fiber direction parallel to the longitudinal axis of the specimens, and were machined to 5 × 1.25 × 65 (fiber direction) mm^3 and 5 × 5 × 40 (fiber direction) mm^3 for four points bending and short beam shear tests, respectively. The target of fiber volume fraction was 60% after heat treatment at 1600°C. The machined C/C specimens' bulk densities were 1.67 – 1.80 g/cm^3.

Four Points Bending Test

The flexural behaviors of the C/C composites were measured by four point bending method up to 2600°C. The supporting and loading rollers' spans were 51mm and 17mm, respectively, and diameters of those rollers were all 6mm. All of jig's parts used in this research were made by graphite materials that was heat treated at 3000°C. Their texture is isotropic and fine grain, and their characteristic is strong, hard and high density.

Almost of the test was carried out with a constant cross head displacement rate of 0.2 mm/min. At room temperature, the tests were carried out in air, an argon or vacuum. The tests at higher temperature were performed in an argon atmosphere, heating up the specimens by avoiding thermal shock and holding 15 minutes at the test temperature to ensure a homogeneous temperature. The correction factor on mechanical compliance of the measuring system was obtained at each testing temperature by pressing supporting and loading rollers uprightly.

Anisotropy of the specimens was examined. The one direction is the loading to the same direction with pressing direction for preparing CFRP, that is, the common direction. Another is the loading to the perpendicular direction to the pressed direction of CFRP. The effects of the loading speed were also examined at some temperatures with the rate between 0.02 to 5 mm/min, using C/C specimen of heat treated up to 3000°C to the common direction.

Short Beam Shear Test

Short beam three points bending test was also carried out. The span, diameter of supporting rollers and diameter of loading roller were 25mm, 4mm and 10mm, respectively. All tests were carried out with a constant cross-head displacement rate of 0.05 mm/min.

RESULTS AND DISCUSSION

Modulus

The modulus evaluated by the four points bending test are plotted against test temperature, as shown Fig. 1. There can be seen the C/C composites treated to 3000°C shows higher modulus than that of 1600°C. They remain constant until 1500°C, and decrease beyond that temperature. The modulus at 2000°C was 70% of that measured at room temperature. There is no anisotropy, that is, no difference by loading directions.

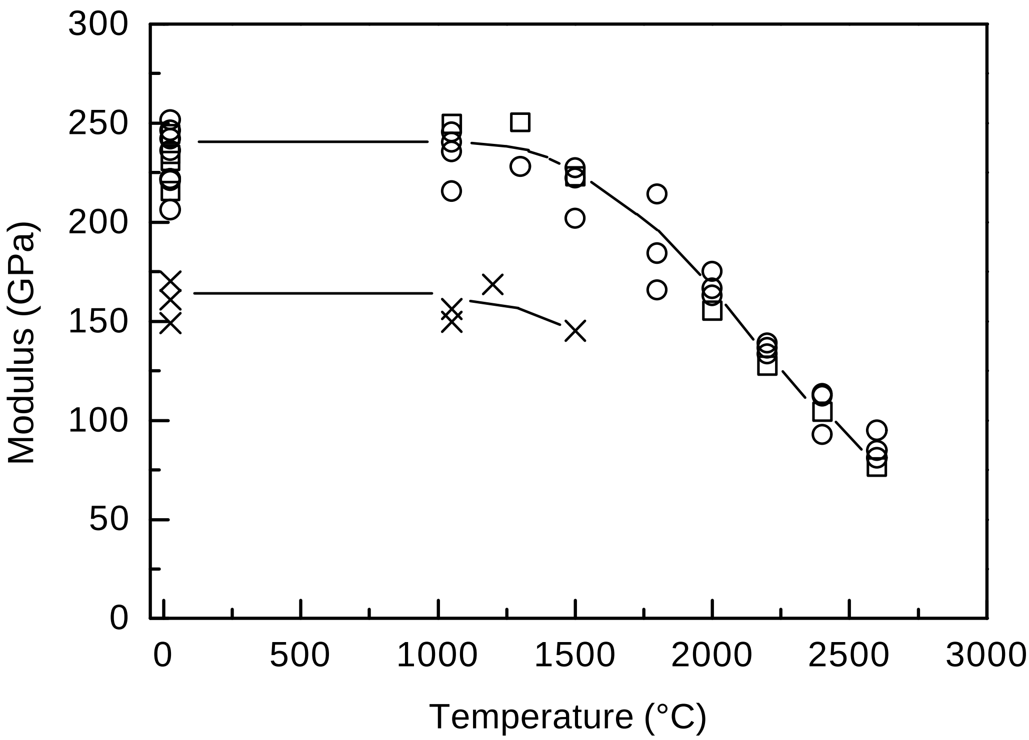


Fig. 1: Modulus evaluate by four point bending test as a function of test temperature. The cross symbols are HTT 1600°C - common direction, the circle symbols are HTT 3000°C - common direction, and the square symbols are HTT 3000°C - perpendicular direction.

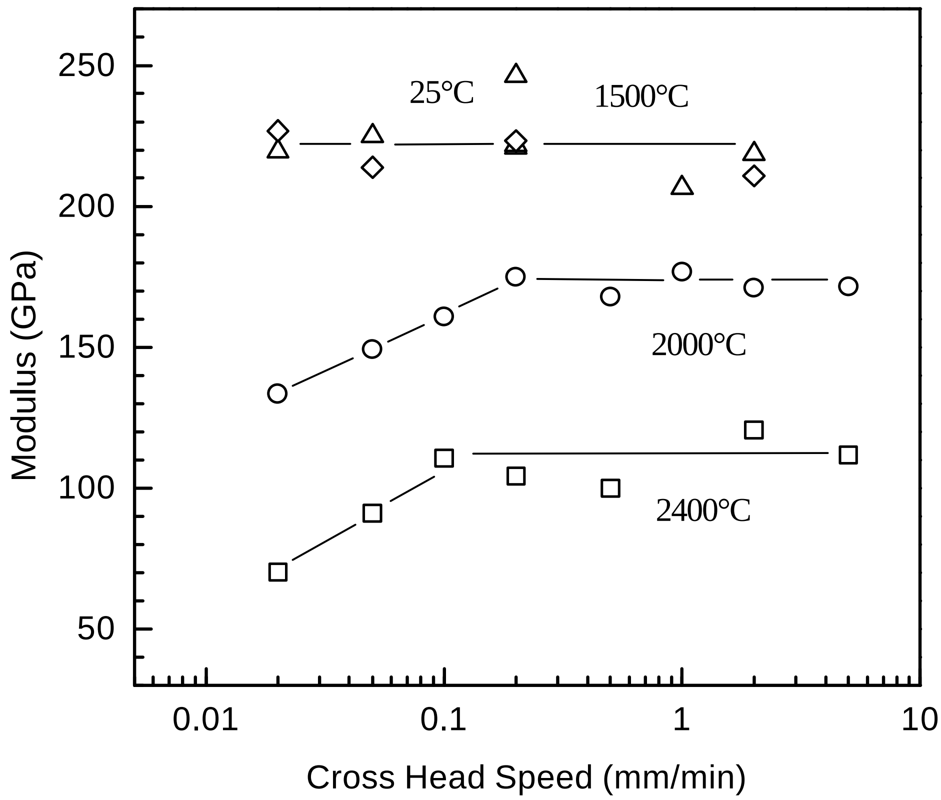


Fig. 2: Influence of cross head speed on modulus at 25, 1500, 2000 and 2400°C.

Effects of loading speed were also examined at 25, 1500, 2000 and 2400°C. As shown in Fig. 2, the moduli were constant at the cross head speed between 0.02 and 5 mm/min at 25°C and 1500°C. On the other hand, at 2000°C and 2400°C, the moduli measured at cross head speed over 0.2 mm/min were constant, but they were decreased with decreasing the cross head speed at less than 0.1 mm/min.

Proof Stress

Proof stress was defined as the stress appearing 0.01mm permanent vertical deflection between the supporting rollers and loading rollers. That deflection is correspond to 0.025% strain in the outer fiber among the inner span. The obtained proof stresses were plotted against the test temperature, as shown in Fig. 3. These stresses also remain practically constant until 1500°C, and decrease beyond that temperature. There is difference also by the final heat treatment temperature, but isn't seen anisotropy. Thus, the proof stress shows same tendency as modulus.

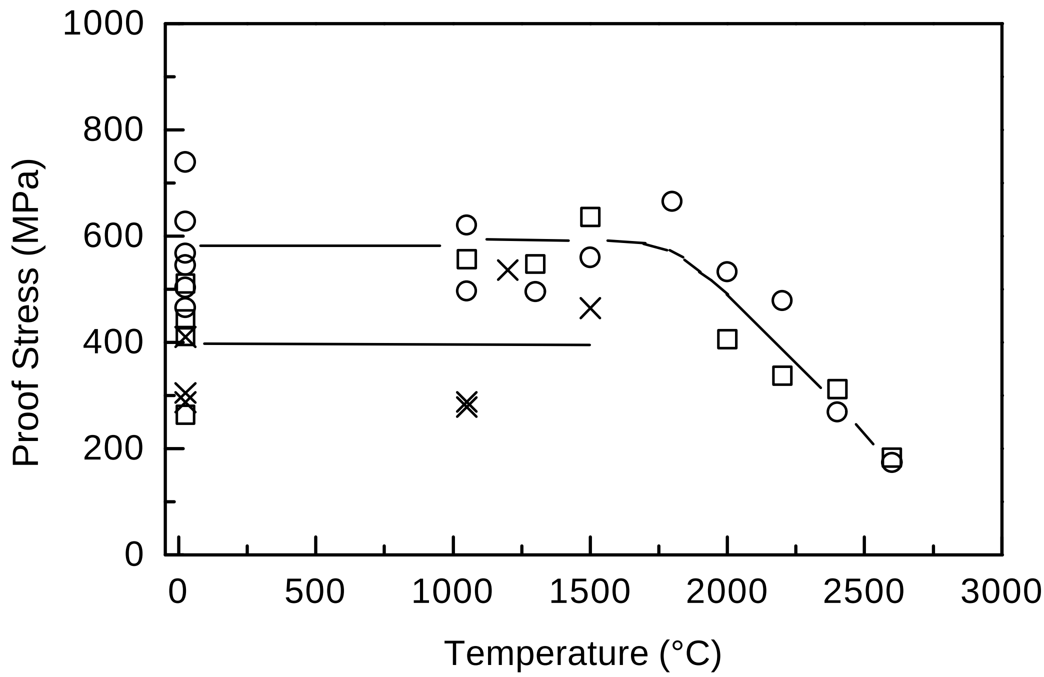


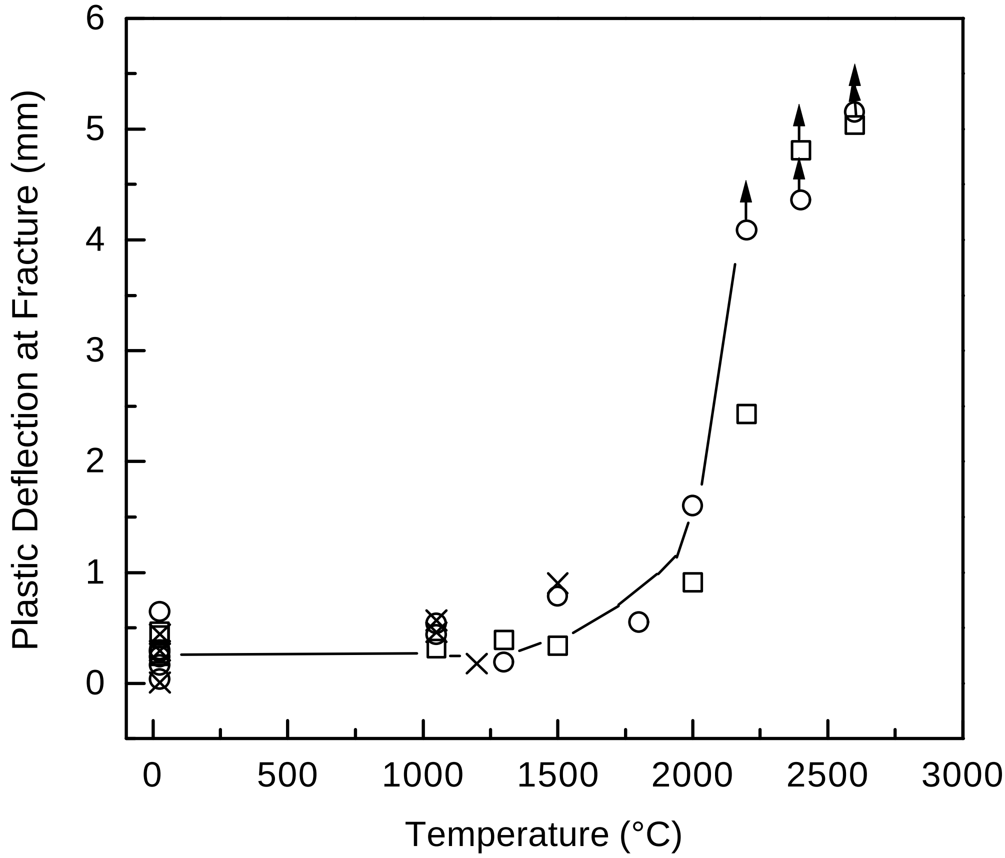
Fig. 3: Proof stress at four point bending test as a function of temperature. The cross symbols are HTT 1600°C - common direction, the circle symbols are HTT 3000°C - common direction, and the square symbols are HTT 3000°C - perpendicular direction.

Plastic Deformation

The amount of plastic deflection after proof stress to the fracture was increased rapidly with decreasing of modulus and proof stress, as shown in Fig. 4. The loading – unloading – loading tests on a few specimens were also carried out at 25°C and 2400°C. At 25°C, any hysteresis phenomenon could not be found out. At 2400°C, however, hysteresis and work hardening phenomena were detected. It is regarded that stress relaxation phenomenon was occurred in those C/C composites at the high temperature, because there could be seen hysterisis and also effects of loading speed on modulus and proof stress.

Bending Strength

The bending strengths increase with the test temperature as shown in Fig. 5. The strength of the C/C composite treated until 1600°C showed lower than that treated to 3000°C. At higher than 2400°C, specimens were deflected plastically over 6mm deflection, and the declines on



load-deflection diagram were not detected in many specimens.

Fig. 4: Plastic deflection by four point bending test as a function of test temperature. The cross symbols are HTT 1600°C - common direction, the circle symbols are HTT 3000°C - common direction, and the square symbols are HTT 3000°C - perpendicular direction.

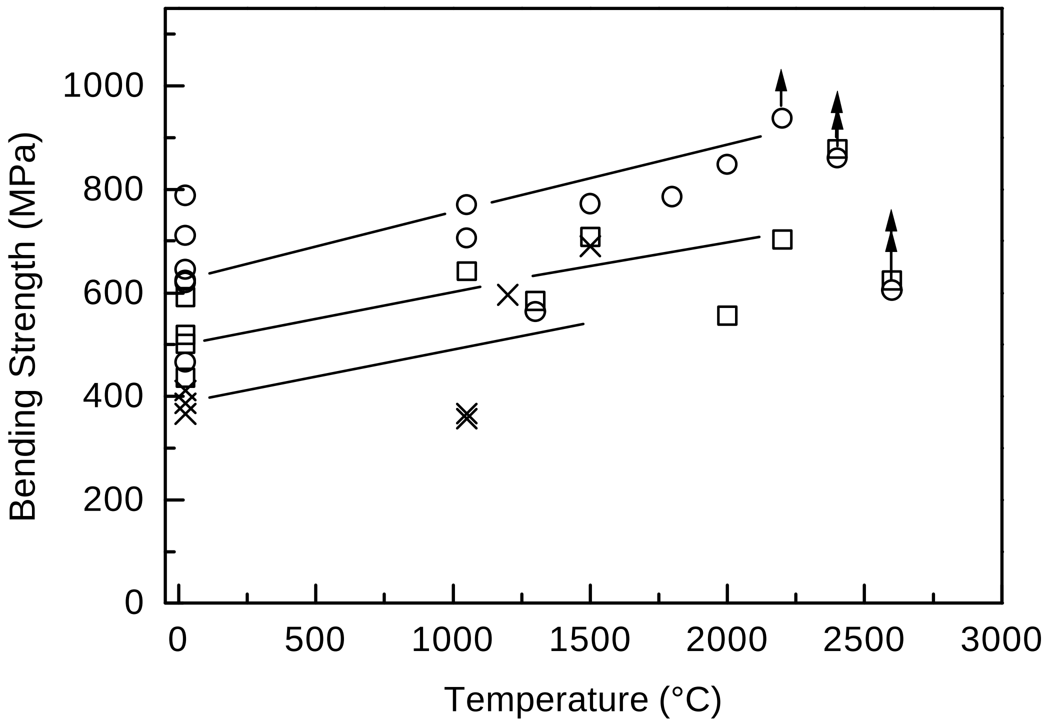


Fig. 5: Bending strength by four point bending test as a function of test temperature. The cross symbols are HTT 1600°C - common direction, the circle symbols are HTT 3000°C - common direction, and the square symbols are HTT 3000°C - perpendicular direction.

Those evaluated by loading to the perpendicular direction was weaker than that of loading to the common direction. This anisotropy must be effect of disoriented filament's probability in C/C composites, which was brought about by fabrication process of the CFRP. This is because the observed shrinkage ratios at carbonization up to 1600°C are 20% and 5% to the thickness and width directions, respectively.

Shear Strength

The evaluated shear strengths are plotted as a function of test temperature, as shown in Fig. 6. There can be seen that the shear strength is increasing with the increase of test temperature, as seen on bending strength. The evaluated shear strength by loading to perpendicular direction was higher than that of loading to the common direction. This anisotropy is the opposite tendency as the above mentioned bending strength. This is the same tendency with the results for CFRPs, but larger than that of CFRPs reported [1]. This anisotropy in the shear strength must be also effect of the disoriented filaments. The interfacial bonding strength of C/C composites is weaker than that of CFRP, so the influence by a little presence of disoriented filament on the strength of C/C composites would be larger.

When specimen had been loading to the common direction, the fracture mode was interlaminar shear, that is, the fracture surface was in the horizontal plane direction. However, that was splitting under the loading roller when it had been loading to the perpendicular direction, that is, the fracture occurred in the vertical plane. Then, these fracture surfaces were the direction of interlaminar plane in both loading directions. Therefore, these C/C specimens fractured by transverse tensile stress, and the actual out-of-laminar shear strengths should be higher than the evaluated values.

The C/C composite treated until 1600°C showed lower shear strength than the C/C composite treated to 3000°C. This is the same tendency with shear strengths which was measured by a mono-filament pulled out from a micro C/C composite [2].

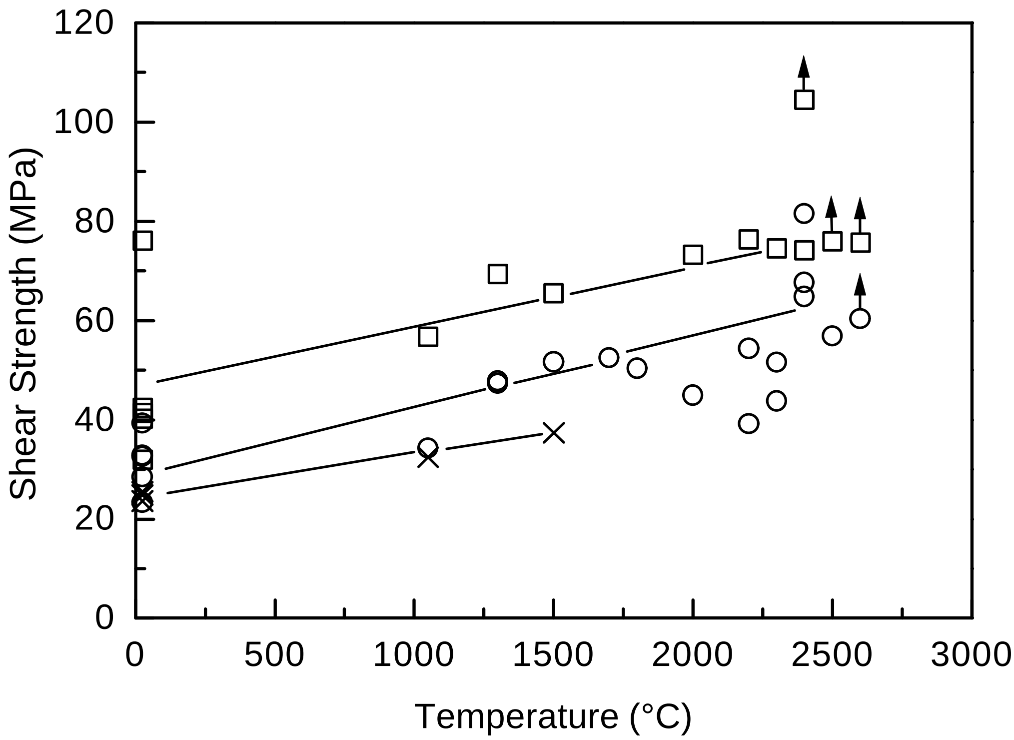


Fig. 6: Evaluated shear strength as a function of test temperature. The cross symbols are HTT 1600°C - common direction, the circle symbols are HTT 3000°C - common direction, and the square symbols are HTT 3000°C - perpendicular direction.

At higher test temperature than 2400°C, the decline of load was not seen until 6mm deflection. Therefore, the plots in Fig. 6 at higher temperature than 2400°C were the apparent maximum shear stress calculated from the load at the deflection of 6 mm.

CONCLUSIONS

Four points and short beam three points bending tests on an unidirectional carbon fiber reinforced carbon composites were carried out up to 2600°C. The modulus and the proof stress were constant until 1500°C, and decreased beyond that temperature. The bending strength and the shear strength increased with temperature. They are bent plastically when they are loaded at a high temperature over 2000°C. In addition, stress relaxation phenomenon was also seen at the high temperature. Slight difference of the presence of disoriented filaments has considerable influence on bending strength and shear strength, but no effect on modulus and proof stress.

REFERENCES

1. T. Honzyo, Y. Sawada and Y. Nakanishi, «Transverse anisotropy in the strength and fracture surface of unidirectional carbon fibre-epoxy composites», J. Mater. Sci. Letters, Vol.9, 1990, pp.514-515.
2. K. Fujita, H. Sakai, N. Iwashita, Y. Sawada and Y. Takigawa, «Interfacial shear strength measurement of C/C using a micro composite», Tanso, No.175, 1996, pp.272-278.