INFLUENCES OF HEAT TREATMENT TEMPERATURES ON TENSILE BEHAVIOR OF UD-C/C COMPOSITES

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Abstract

The tensile behavior of unidirectionally reinforced (UD-) C/C composites was evaluated as a function of the heat treatment temperatures (HTTs). The carbon fiber strength and the fiber/matrix interfacial bonding strength were also evaluated as a function of the HTT using a single-fiber tensile test and a fiber bundle push-out test. Based on the experimental results, the strength dominant factors of the UD-C/C were discussed. The tensile strength of the UD-C/Cs slightly increased with increase in HTT in the range between 1473 K and 2673 K. However, the tensile strength of carbon fiber decreased significantly with increase in HTT. On the other hand, the fiber/matrix bonding strength decreased remarkably with HTT. Hence, the strength enhancement of the UD-C/Cs heat-treated above 2673 K was considered to be caused by the enhanced crack deflection along the weak fiber/matrix interfaces.

1 Introduction

The tensile strength of carbon fiber-reinforced carbon matrix composites (C/Cs) is generally much lower than that calculated from the rule-of-mixture. The utilization of the fiber strength in C/Cs is usually less than 50% [1-4]. It is well known that the mechanical behavior of C/Cs is highly sensitive to the processing conditions such as surface treatments of the reinforcing fibers, heating rates during the carbonization processing, numbers of densification cycles, and the final heat treatment temperatures (HTTs) [1,3-7]. Especially, HTT affects the mechanical properties of C/C composites significantly because the properties of the individual constituents (reinforcing fiber and matrix) and interfacial bonding between the fiber and matrix are drastically changed by HTT conditions. Therefore, in order to clarify the strength dominant factors, it is necessary to evaluate the properties of the individual constituents of the C/Cs.

The effects of HTTs on the mechanical behavior of C/Cs have been reported by many researchers [3-7]. However, in the previous researches, the fiber/matrix interfacial properties have been indirectly estimated, for example, by observing the pull-out length of the reinforcing fibers and the microstructure at the fiber/matrix interface. Recently, Hatta et al. have developed a technique namely a fiber bundle push-out method for evaluating the fiber/matrix interfacial properties [1,8]. By using the fiber-bundle push-out method, they reported that fiber/matrix interfacial shear strength decreases with an increase in the HTT using this method. In addition, the tensile strength of C/Cs was reported to increase with decrease in the fiber/matrix bonding strength. However, influences of the HTTs have been discussed mainly for the C/Cs reinforced with the pitch-based carbon fibers and the experimental results for the polyacrylonitrile (PAN)-based fiber C/Cs are not sufficient.

In the present study, influences of the HTTs on the tensile behavior of a UD-C/C reinforced with a PAN-based carbon fiber were examined. In addition, in order to clarify the strength dominant factors, the strengths of the carbon fiber and the fiber/matrix bonding were evaluated as a function of the HTT.

2 Experimental Procedures

2.1 Materials

The UD-C/Cs used in this study were prepared by a hot isostatic pressing (HIP) method using a coal-tar pitch as matrix precursor. Carbon-fiber tow (TORAYCA® T-300B, 12000 filaments, Toray Co., Japan) was first wound on a C/C plate with the dimensions of 100 mm x 100 mm x 10 mm^t. The plate was then placed in a HIP furnace and a coal-tar pitch was impregnated followed by the carbonization at ≈1073 K under a high pressure in Ar atmosphere. This impregnation-carbonization process was repeated for three times for densification. The carbonized materials were finally heat-treated at different temperatures of 1473 K, 1773 K, 2273 K, 2673 K and 3273 K for 1h in Ar atmosphere. The fiber volume fraction of the UD-C/C was ≈60%.

2.2 Tensile Tests of UD-C/Cs

The tensile strength and stress-strain relations of the UD-C/Cs were obtained at room temperature in ambient air. The dimensions of the specimens were 80 mm x 5 mm x 0.5 mm^t. Aluminum tabs were bonded to the grip parts of the specimens (the gauge length was ≈25 mm). The tensile tests were conducted using a screw-driven testing machine (Model 44R1125, Instron, USA) under a constant cross-head speed of 0.2 mm/min. The tensile load was applied parallel to the fiber direction. The tensile strains were measured using strain gages adhered to both surfaces of a specimen; the average strains was used as a representative value. After tensile testing, fracture surfaces were observed using a scanning electron microscope (SEM, S-4700, Hitachi, Japan).

2.3 Single-fiber Tensile Tests

The HTT of the as-received T-300B fiber is supposed to be about 1773 K. The UD-C/Cs examined in this study were heat-treated in the range between 1473 and 3273 K. Thus, the tensile properties of the carbon fibers in the UD-C/Cs are expected to be different from that given in the supplier's data. Moreover, fiber properties are reported to change depending on the gage length [9].

In this study, single-fiber tensile tests were conducted for T-300B fibers heat-treated at different HTTs according to JIS R 7606 (JIS: Japan Industrial Standards). The monofilament was mounted on a paper supporting tab with a fixed gage length of 25 mm, as shown in Fig.1. The single-fiber tensile tests were conducted using a screw-driven testing machine (Model 4502, Instron, USA) under a constant cross-head speed of 0.25 mm/min. The ultimate tensile strength σ_f was calculated by:

$$\sigma_f = F_{max} / \pi \cdot (d/2)^2 \qquad (1)$$

where F_{max} is the ultimate tensile load and d is the fiber diameter. In this study, the value of d was assumed to be 7 μm for all the fibers.

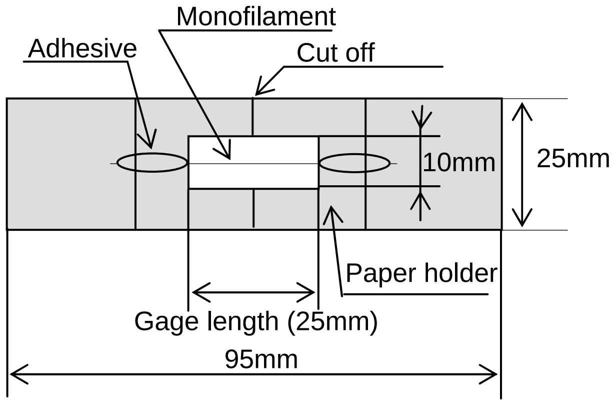

Fig.1 Schematic drawing of the single-fiber tensile tests according to JIS R 7606.

2.4 Fiber Bundle Push-out Tests

The fiber/matrix interfacial bonding strength of the UD-C/Cs was measured by a fiber bundle push-out method [1,8]. Figure 2 shows the schematic drawing of the test setup. In this experiment, interfacial shear fracture was induced by a load applied with a tungsten-carbide pushrod with a flat end to the fiber bundle, where the fibers are aligned parallel to the applied load. The diameter of the pushrod and the thickness of the specimens were 50 μm and ≈150 μm, respectively. These tests were conducted using a screw-driven testing machine (AG-5000A, Shimadzu Co., Japan) under a cross-head speed of 0.01 mm/min.

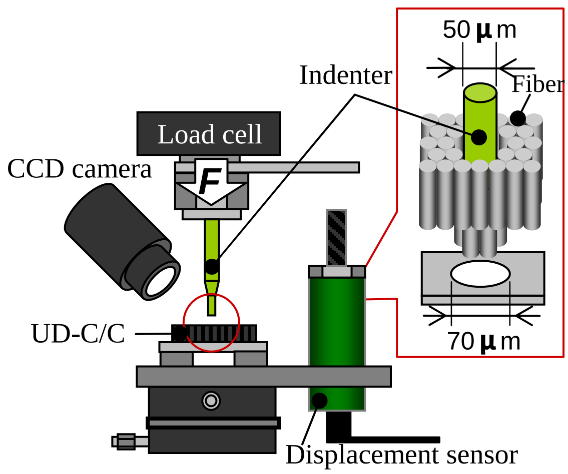

Fig.2 Schematic drawing of the fiber-bundle push-out tests.

3 Results and Discussion

3.1 Tensile Tests of UD-C/Cs

Figure 3 shows typical tensile stress - strain curves of the UD-C/Cs with different HTTs. The tensile stiffness of the UD-C/Cs heat-treated at 1473 K and 1773 K slightly increases with the tensile strain up to the final fracture. This slight increase in the stiffness is attributed to the nonlinear elastic behavior of the carbon fibers [10]. In contrast, stiffness degradation was clearly observed prior to the final fracture for the UD-C/Cs heat-treated exceeding 2273 K. The stiffness degradation may be caused by the accumulation of damage in the specimens. The initial elastic modulus of the UD-C/Cs increases significantly with an increase in the HTT. This enhancement is attributed to further graphitization of the carbon fibers due to heat treatments.

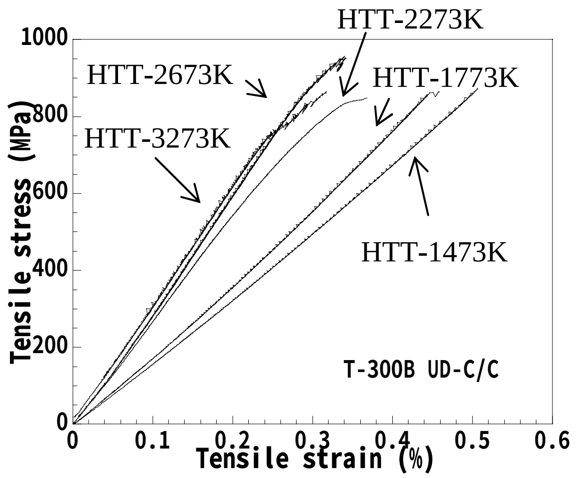

Fig.3 Typical tensile stress-strain curves of the UD-C/Cs with different HTTs at room temperature.

The ultimate tensile strength and ultimate strain of the UD-C/Cs are summarized in Fig.4. The ultimate tensile strengths slightly increased up to 2673 K and it decreased at 3273 K. The ultimate tensile strain decreases monotonically with an increase in the HTT.

3.2 Fracture Surface Observations

Figure 5 shows the typical fracture surfaces of the UD-C/Cs heat-treated at various HTTs. As shown in this figure, the pull-out length of the reinforcing fibers increases with the increase in the HTT in the range between 1473 K and 2673 K. However, the pull-out length was observed to decrease at 3273 K.

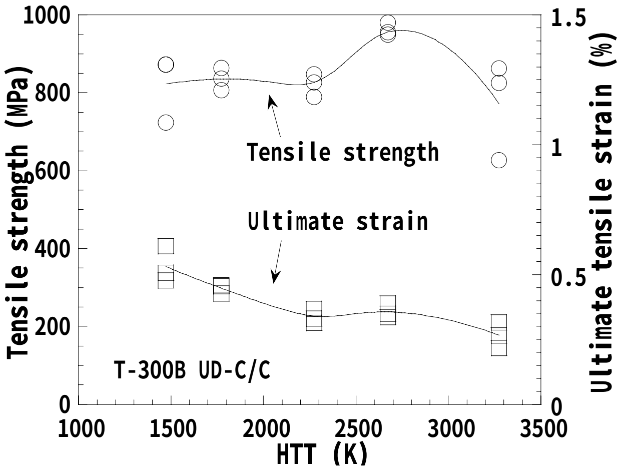

Fig.4 Ultimate tensile strength and tensile strain of the UD-C/Cs as a function of the HTT.

Figure 6 shows the fracture surfaces observed at high magnification. As shown in these figures, the interface between the fiber and matrix seems to be bonded well for the UD-C/C heat-treated at 1473 K and 1773 K. In contrast, intensive debondings are observed along the fiber/matrix interfaces above 2273 K, suggesting weak fiber/matrix bonding strength. These observation results indicate that the fiber/matrix interfacial properties play an important role on the tensile fracture behavior of the UD-C/Cs.

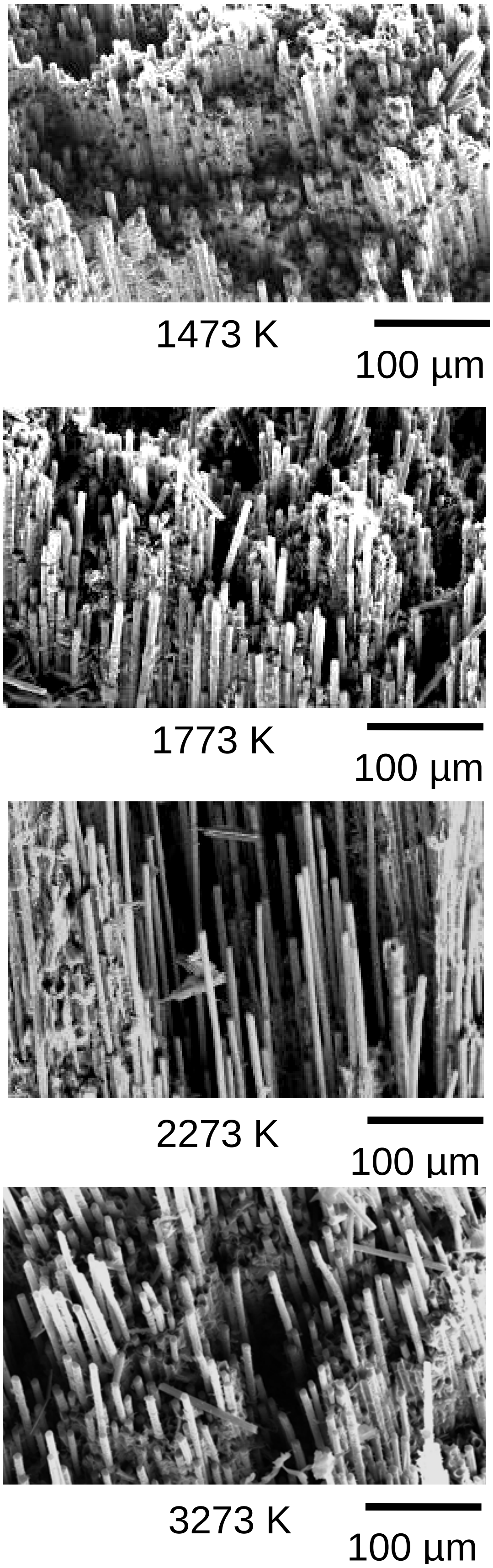

Fig.5 SEM photographs of fracture surfaces of the UD-C/Cs heat-treated at various temperatures.

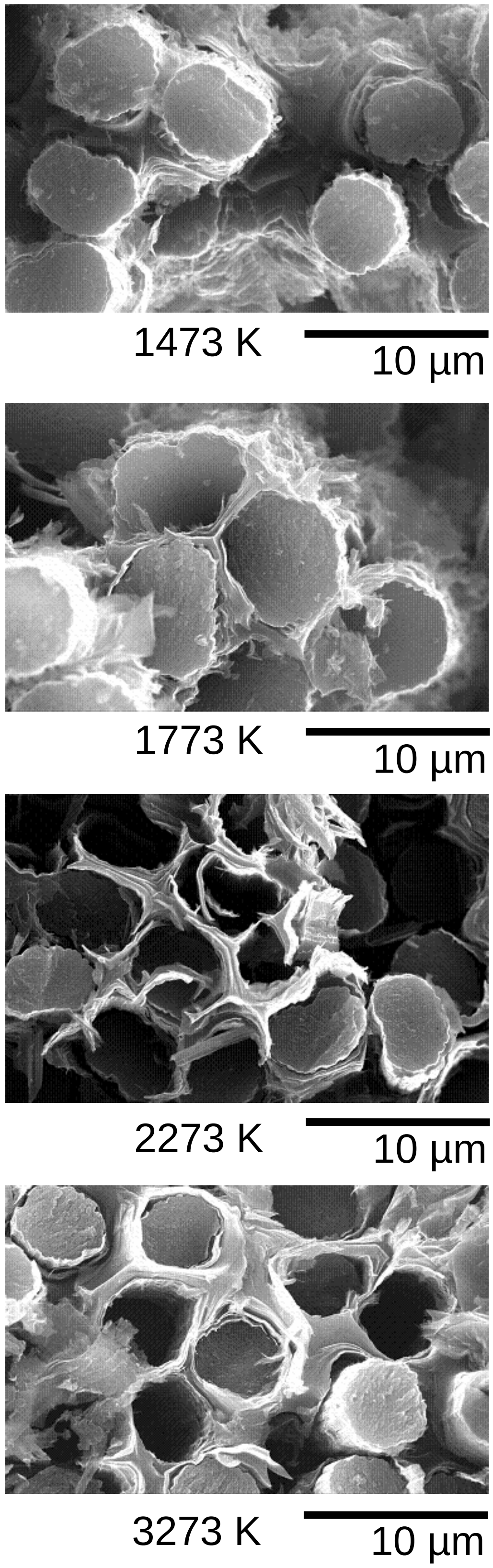

Fig.6 Fiber/matrix interfaces of the UD-C/Cs heat-treated at various temperatures after the tensile tests.

3.3 Strength of Carbon Fiber

The typical load - displacement curves obtained from the single-fiber tensile tests are shown in Fig.7. It can be seen in this figure that the stiffness of the T-300B carbon fiber monotonically increases with an increase in the HTT. In addition, the ultimate tensile load and the fracture strain decrease drastically with increasing the HTT. The averaged tensile strengths of the carbon fibers are shown in Fig.8 as a function of the HTT.

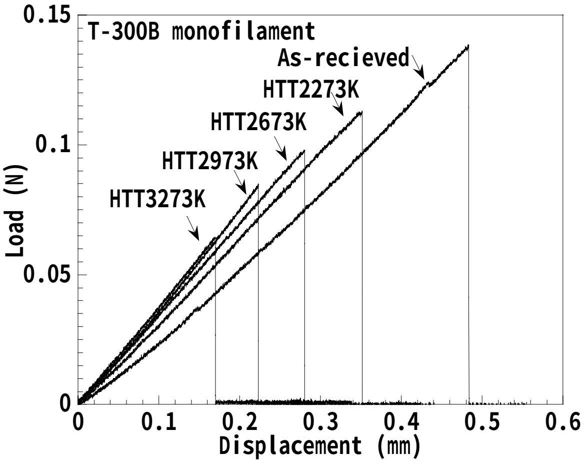

Fig.7 Typical load - displacement curves of T-300B monofilaments with different HTTs.

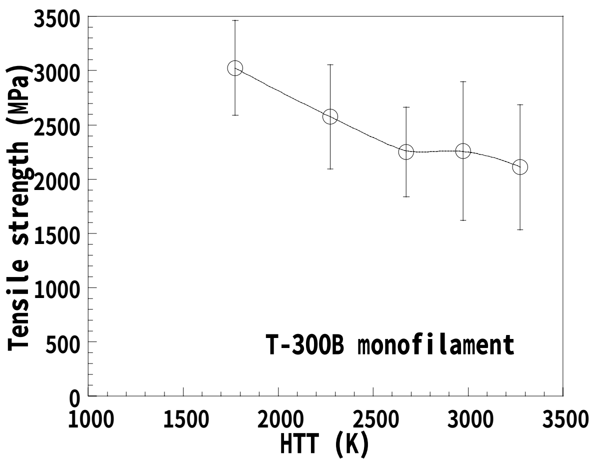

Fig.8 Tensile strength of T-300B monofilaments as a function of the HTT.

3.4 Fiber/Matrix Interfacial Bonding

Figure 9 shows the typical interfacial shear stress - displacement curves obtained from the fiber-bundle push-out test. The interfacial shear stress τ_i in this figure was calculated from:

$$\tau_i = F / P_e \cdot L \qquad (2)$$

where F, P_e and L are the applied load, the perimeter of the pushed-out bundle determined by SEM after testing and the thickness of the specimen, respectively.

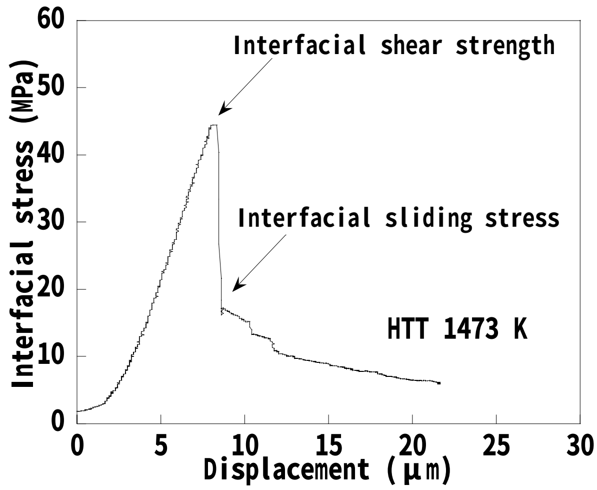

Fig.9 Typical fiber/matrix interfacial shear stress - displacement curve obtained by the fiber-bundle push-out test of the UD-C/C heat-treated at 1473 K

Initially, the value of τ_i rapidly increases up to its maximum value. After the maximum stress, the value of τ_i abruptly drops and gradually decreases with a further increase in the displacements. SEM observations revealed that the fiber/matrix interfacial fracture completed just after the maximum stress and the loaded bundle began to slide thereafter. Hence, the maximum stress and the stress after the abrupt stress drop are defined as the fiber/matrix interfacial shear strength and sliding stress, respectively.

Figure 10 shows the fiber/matrix interfacial shear strength and sliding stress as a function of the HTT. The fiber/matrix interfacial shear strength decreases significantly from 1473 K to 2273 K followed by a nearly constant value up to 3273 K. It should be noted that a large scattering was observed for the UD-C/C heat-treated at 1437 K. This result suggests that both the strong and weak fiber/matrix interfaces exist together in the UD-C/Cs heat-treated at 1473 K.

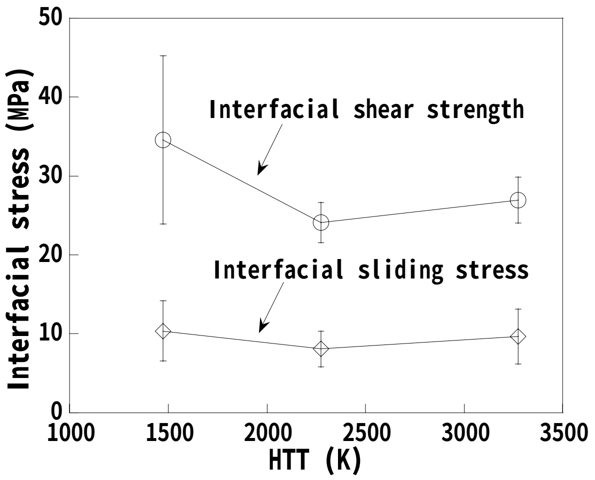

Fig.10 Fiber/matrix interfacial shear strength and sliding stress obtained by the fiber bundle push-out tests.

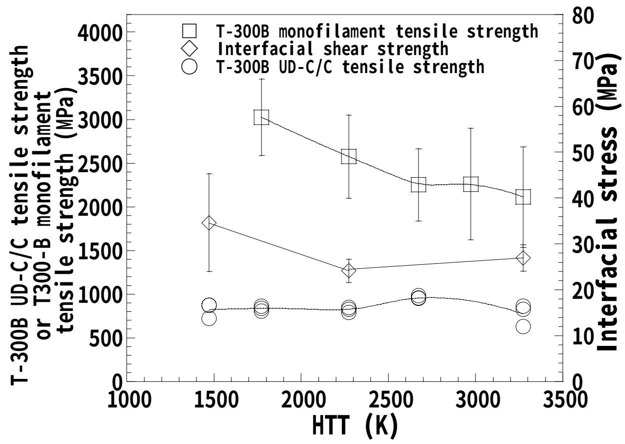

Fig.11 Tensile strength of UD-C/Cs and T-300B carbon fibers, and fiber/matrix interfacial shear strength as a function of the HTT.

3.5 Tensile Fracture Mechanism of UD-C/Cs

The tensile strengths of UD-C/Cs and T-300B monofilaments, and the fiber/matrix interfacial shear strength are summarized in Fig.11 as a function of the HTT. The tensile strength of the T-300B monofilaments decreases by ≈30% when the HTT is increased from 1773 K to 3273 K. However, the tensile strength of UD-C/Cs maintained nearly constant value (≈800 MPa). This result indicates that fiber strength is much effectively utilized in the UD-C/Cs as the HTT is increased.

Figure 12 shows the tensile fracture process estimated for the present UD-C/Cs. In the HTTs between 1473K and 1773K, the fiber strength is relatively strong as shown in Fig.8. Hence, matrix cracking is expected to occur first during the tensile tests. Then, the matrix cracks penetrate into the adjacent carbon fibers because the fiber/matrix bonding is too strong. As a result, many fibers would be broken at once and load redistribution to the unbroken fibers becomes large, yielding brittle final fracture. In contrast, in the HTTs exceeding 2273K, fiber breakage is considered to initiate even at low stress due to the weak fiber strength. However, these cracks would be deflected parallel to the loading direction because the fiber/matrix interface is sufficiently weak. As a result, the fiber breakage accumulates gradually with an increase in the applied load, yielding the progressive stiffness reduction in the stress-strain curves.

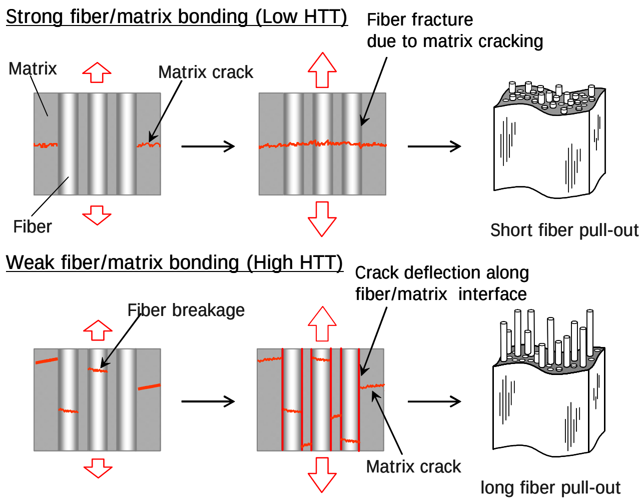

Fig.12 Tensile fracture processes estimated for the low and high HTT UD-C/Cs.

4. Conclusions

The tensile behavior of UD-C/Cs reinforced with T-300B carbon fibers was evaluated as a function of the HTT. The carbon fiber strength and the fiber/matrix interfacial shear strength were also evaluated by a single-fiber tensile test and a fiber bundle push-out test. The tensile strength of the UD-C/Cs slightly increased with an increase in the HTT in the range between 1473 K and 2673 K. However, the carbon fiber strength decreased significantly with an increase in the HTT. While, the fiber/matrix interfacial shear strength remarkably decreased with increasing the HTT. Hence, it was considered that the strength enhancement of the UD-C/Cs up to the

HTT of 2673 K was caused by the enhanced crack deflection along the weak fiber/matrix interfaces.

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