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Effects of normal stress on the off-axis mechanical behavior of a plain-woven C/C composite

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Abstract—In-plane shear behavior of a plain-woven C/C composite was examined at room temperature. Three representative test methods were performed: Iosipescu, ±45° tensile and ±45° compressive tests. Test methods were evaluated by comparing apparent shear behavior and shear damage progress. Highest and lowest shear strengths were obtained in Iosipescu and ±45° tensile tests, respectively. In particular, the ±45° tensile method yielded most remarkable stiffness reduction because tensile normal stresses have a profound influence on shear damage progress. Therefore, it was concluded that the ±45° tensile method is not appropriate for evaluating in-plane shear strength. In contrast, ±45° compressive tests yielded almost identical damage evolution to that in Iosipescu tests. Although apparent shear behavior depended remarkably on test methods, a common damage process with propagation of transverse cracks, and debonding among fiber bundles was observed. Finally, to demonstrate normal stress effects on shear damage progress, mechanical behavior under various biaxial loads was calculated using a continuum damage mechanics (CDM) approach. Calculated behavior agreed reasonably well with experimental results when shear/normal stresses coupling effects were taken into account.

Keywords: C/C composite; off-axis mechanical behavior; shear damage; normal/shear stresses coupling effect; modeling

1. INTRODUCTION

Carbon fiber reinforced carbon matrix (C/C) composites maintain high strength and stiffness even at elevated temperatures exceeding 2300 K [1, 2]. For this reason, C/C composites have been applied to thermal protection systems (TPSs) of reentry space vehicles, aircraft brake disks, nuclear reactors, etc. To design these components, many studies have been conducted focusing on their mechanical properties. A general conclusion is that although C/C composites exhibit high

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strength and brittle behavior under loadings parallel to a fiber axis, a remarkable non-linearity followed by extremely low strength appears in shear [3–5]. For example, Heredia *et al.* [6] reported that their cross-ply C/C composite showed an ultimate strength of ≈300 MPa in tension parallel to a fiber direction, but only ≈30 MPa for shear. They concluded that because of extremely low shear strength, shear damage parallel to loading direction generated around stress concentrations in notched or holed C/C specimens. The damage is believed to cause stress redistribution and notch insensitivity [5]. Therefore, poor shear strength and non-linearity become important issues in designing actual C/C structures.

In spite of the importance of in-plane shear properties, a few discussions of validity of shear test methods have been reported for C/C composites. Among the test methods, Iosipescu, off-axis tensile, and off-axis compressive tests with fibers oriented at certain angles seem promising from viewpoints of costs and ease of operation [7–11]. The Iosipescu tests perform evaluations in a nearly pure shear stress field between the V-notches. In contrast, off-axis tests principally provide tensile or compressive stress parallel and normal to fiber directions along with shear. Such differences between stress fields may cause variations in test results. Denk *et al.* have applied Iosipescu, 10° and ±45° off-axis tensile methods to a laminated cross-ply C/C composite [3]. In their research, Iosipescu test showed highest shear strength and apparent strength decreased remarkably with increasing the off-axis angles. Therefore, normal stress effects in off-axis tests require discussion to evaluate in-plane shear properties of C/C composites.

Three representative test methods were performed in the present study: Iosipescu, ±45° tensile and ±45° compressive tests. Shear behavior derived from each test method was evaluated by comparing shear strength and stiffness reductions during loadings. Microscope observations were also conducted to clarify shear mechanisms. Special attention was given to identification of normal stress effects on apparent shear behavior. Finally, to demonstrate normal stress effects, mechanical behavior under various off-axis loadings was calculated using continuum damage mechanics (CDM) reported by Siron *et al.* [12]. The methodology incorporates shear non-linearity, permanent strains, and shear/normal stresses coupling effects on shear damage progress. As the first step, tension/tension and compression/compression biaxial stress states are considered in the present paper.

2. EXPERIMENTAL PROCEDURE

2.1. Materials

Material examined in this study was a C/C composite reinforced with a plain-woven cloth and supplied by Ohwada Carbon Co., Japan. Reinforcing fiber, fiber volume fraction and final heat treatment temperature were Besfight HTA (Toho Tennax Co., Japan), ≈50% and 1873 K, respectively. Phenolic resin was used as carbon matrix precursor with several impregnation and pyrolysis cycles. Both fill and wrap yarns contain 12K-filaments with the periodicity of ≈2 mm. The plain-woven C/C composite processes many cracks running parallel to fiber axes as indicated by arrows in Fig. 1. We denote these cracks by transverse cracks (TCs). The TCs induced during processing because of pyrolysis of matrix resin and thermal expansion anisotropy of carbon fibers.

2.2. Mechanical tests

Figure 2 illustrates shapes and dimensions of specimens tested in this study. Three shear tests were performed: Iosipescu, ±45° off-axis tensile, and ±45° compressive tests. The 10° and 22.5° off-axis tensile and compressive tests were performed to demonstrate the CDM model used to calculate mechanical behavior under various biaxial loadings. Mechanical tests except for Iosipescu method were performed using a servo-hydraulic testing rig (Model 8501; Instron Corp., USA). Aluminum tabs with 1-mm thickness were bonded at specimen ends with the load applied using hydraulic wedge grips. Iosipescu tests were performed using a screw-driven testing machine (Model 4482; Instron Corp., USA) and a modified Wyoming type fixture. All mechanical tests were performed at room temperature under a constant displacement rate of 0.1 mm/min.

Shear strains were monitored with strain gages bonded on each specimen face. Shear stress τ and strain γ were calculated according to the following equations.

$$\text{For Iosipescu tests:} \quad \tau = P/A, \quad \gamma = \varepsilon_{+45} - \varepsilon_{-45}, \tag{1}$$

$$\text{For } \pm 45^\circ \text{ off-axis tests:} \quad \tau = 0.5 \times P/A, \quad \gamma = |\varepsilon_\mathrm{L}| + |\varepsilon_\mathrm{T}|. \tag{2}$$

In those equations, P denotes the applied load, A the cross-sectional area of gauge sections, $\varepsilon_{\pm 45}$ the strains measured in the ±45° directions, and ε_L and ε_T the strains in longitudinal and transverse directions, respectively.

3. IN-PLANE SHEAR BEHAVIOR AND DAMAGE MECHANISMS

3.1. Tensile and compressive test results

Figure 3 shows typical stress–strain ($\sigma-\varepsilon$) curves of the plain-woven C/C composite under tensile and compressive loadings parallel to a fiber direction. Both the $\sigma-\varepsilon$ curves were almost linear up to final failure. The Young's modulus of ≈74 GPa, and Poisson's ratio of ≈0.02 were obtained for both tensile and compressive loadings. Observations by replica films revealed that no debonding between fiber bundles and no additional transverse cracking in fiber bundles perpendicular to loading axis occurred before final failure.

3.2. In-plane shear test results

Figure 4 shows typical stress–strain curves in shear ($\tau-\gamma$ curves) of the plain-woven C/C composite derived from loading/unloading testing. Initial $\tau-\gamma$ relations were nearly identical for all the test methods. Elastic behavior was observed

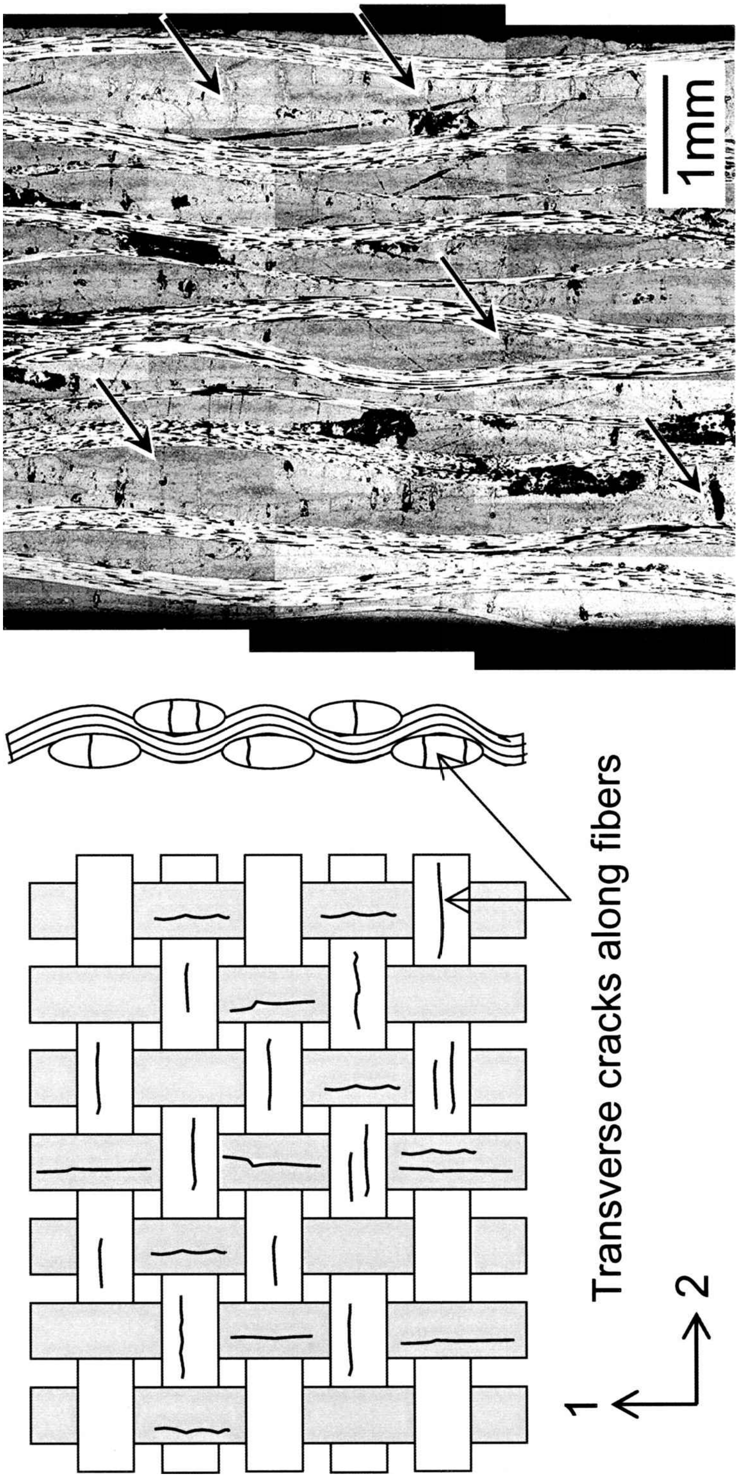

Figure 1. Cross-sectional view of the plain-woven C/C composite.

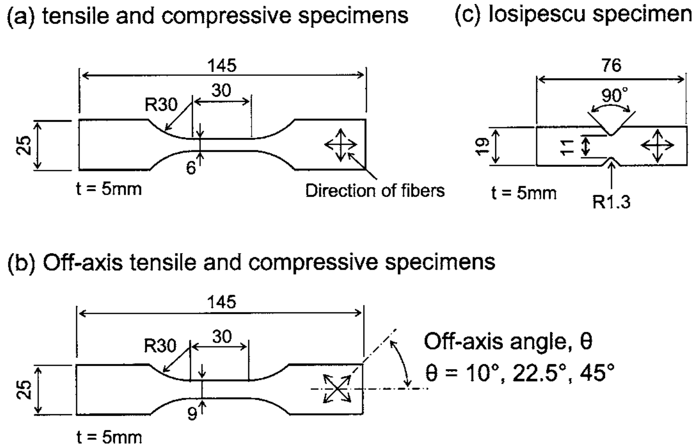

Figure 2. Shapes and dimensions of specimens examined in the present study.

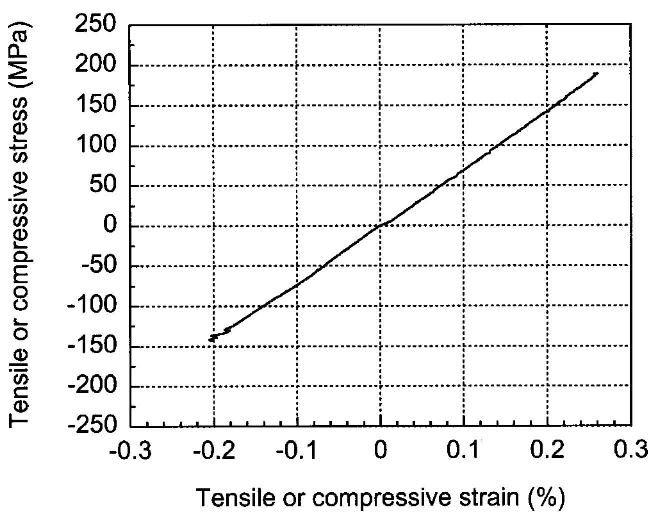

Figure 3. Stress–strain curves of the plain-woven C/C composite under loadings parallel to a fiber direction.

up to the shear stress of 10 MPa and the initial shear modulus G_0 of 7.1 ± 0.5 GPa was obtained. After initial elasticity, non-linearity appeared in all test methods. As can be seen in Fig. 4, in-plane shear strength depended remarkably on test methods. Highest and lowest shear strengths were obtained in Iosipescu and ±45° tensile tests, respectively. Similarly to Denk's results [3], the ±45° tensile method yielded the most remarkable stiffness reduction surpassing all other test methods.

To assess the phenomenological nature of shear damage, secant shear modulus and permanent strain were evaluated. Figures 5a and 5b show changes of shear moduli G_{12} and permanent strain ε_p, respectively. In this paper, shear modulus change was described using the shear damage parameter d_6. The d_6 indicates

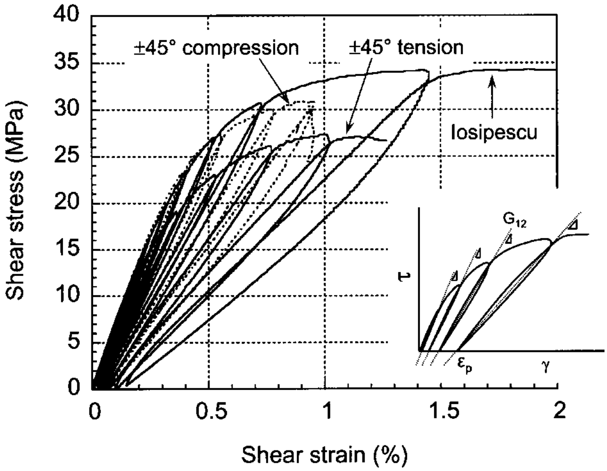

Figure 4. Shear stress–strain curves of the plain-woven C/C composite obtained by various test methods.

progress of shear damage and is given by equation (3):

$$d_6 = 1 - \frac{G_{12}}{G_{12}^0}, \tag{3}$$

where G_{12}^0 is the initial shear modulus and G_{12} is the shear modulus. The G_{12} was calculated from the secant of each hysteresis loop as shown schematically in Fig. 4. Beyond shear stress of 10 MPa, both d_6 and ε_p increased concomitant with increasing applied stress. It was again confirmed that ±45° tensile tests yielded the greatest increase in both d_6 and ε_p.

3.3. Microscope observations and normal stresses effects on shear damage

To clarify above dependencies on test methods, microscopic observations were performed for each test method. Observations were performed by applying replica film technique on polished specimen top and side surfaces during loading/unloading testing.

Figure 6 shows typical microstructural changes appearing on side surfaces of ±45° compressive specimens. A schematic representation of fiber bundle configuration for the observed surface is given in Fig. 6a. At $\tau \approx 10$ MPa, i.e. after onset of non-linearity, debonding between fiber bundles and plies was clearly identified. As indicated by arrows in Fig. 6c, debonding initiated from tips of TCs where stress concentration occurred. Upon further loading, debonding propagated along bundle interfaces and finally specimens failed by debonding connections. Figure 6d shows that the debonding connections occurred mainly through TCs. Similar damage behavior was observed in other test methods, as shown in Fig. 7. Therefore, we conclude that the dominant mechanism yielding shear non-linearity is debonding between fiber bundles and plies. Gupta *et al.* and Keith *et al.* performed in-plane

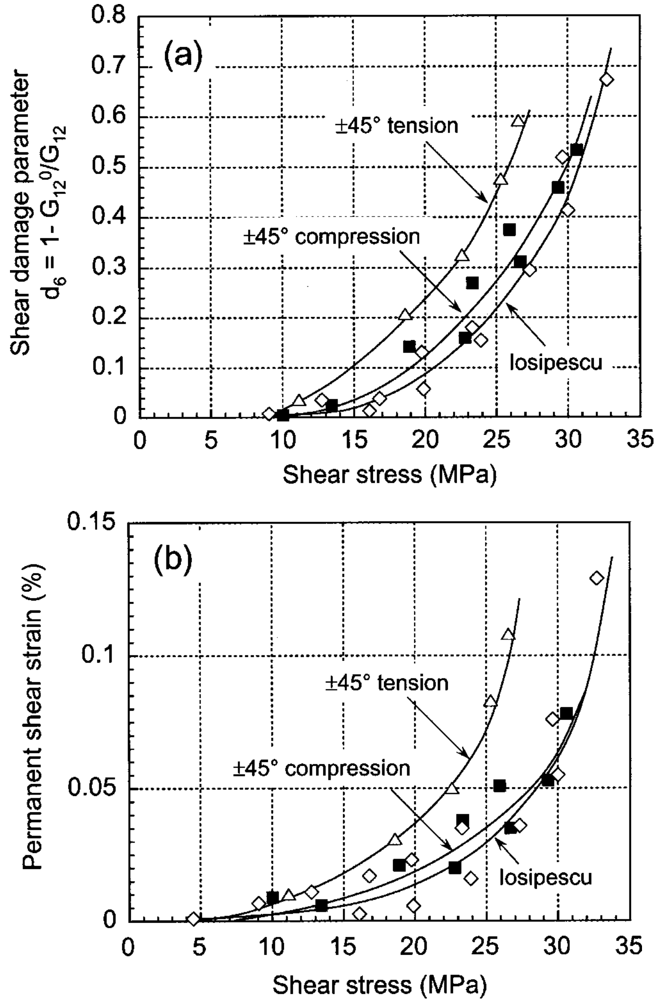

Figure 5. Changes in shear damage parameter (a) and shear permanent strain (b) obtained from loading/unloading testing.

shear tests of 8-harness satin weave C/C and SiC/SiC laminates. They also observed fiber bundle debonding [13, 14]. In particular, Gupta *et al.* performed finite element analysis to investigate stress states at fiber bundle interfaces. According to their results, debonding tends to initiate at crimp interfaces where fill and wrap bundles within a ply cross-over because tensile through-thickness stress appears at these locations. Thus, debonding between fiber bundles and plies manifests itself as typical shear damage for 2D woven C/C and SiC/SiC composites.

Figure 8 show typical microstructural changes appearing on top surfaces of Iosipescu specimens. Before tests, many as-processed TCs running parallel to fiber directions were seen, as indicated by arrows. When shear load was applied, the TCs started to propagate and fiber bundle debonding finally occurred. It

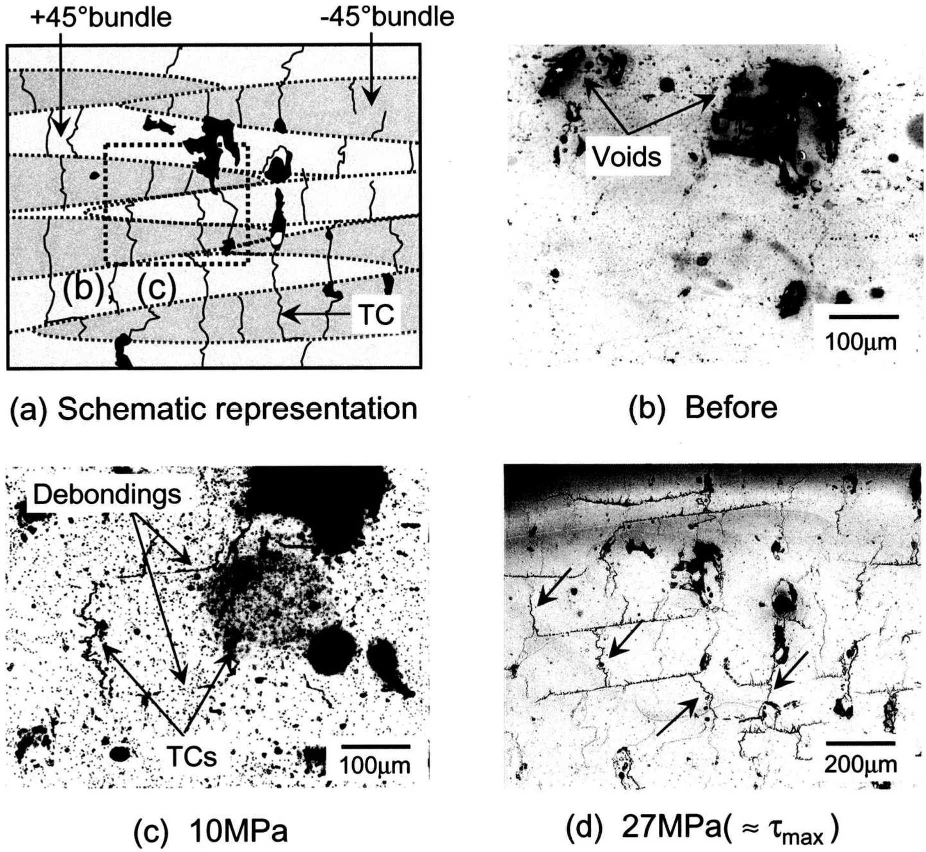

Figure 6. Typical microstructural changes appearing on side surfaces of gauge sections in ±45° compressive tests.

should be noted here that, although propagation of as-processed TCs was frequently observed, formation of new TCs was rarely identified. To verify this behavior, we measured TC spacing: the distance between adjacent TCs for ±45° tensile and ±45° compressive specimens. These measurements were performed using replica film images taken on sides of gauge sections at various loading stages. Figure 9 summarizes observation results. In both test methods, TC spacing clearly maintained its initial value until final failure.

Based on above observation results, we discuss normal stress effects on apparent shear behavior. Figures 10a and 10b illustrate estimated damage mechanisms for ±45° tensile and compressive tests. In the case of the ±45° tensile tests, tensile stress with identical magnitude to shear stress appears principally in both the parallel and transverse to fiber directions. In contrast, compressive normal stress appears in ±45° compressive tests. One may expect that many new TCs initiate along fiber directions in ±45° tensile tests, because tensile transverse strength of fiber bundles is quite small (several MPa). However, as indicated in Fig. 9, formation

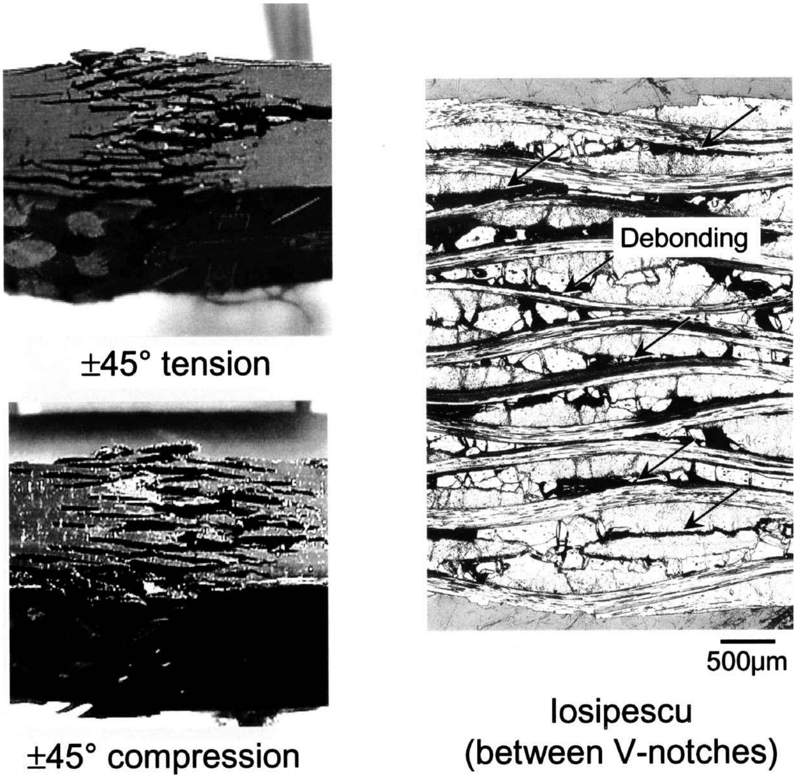

Figure 7. Fracture behavior of the plain-woven C/C composite after the three in-plane shear tests.

of new TCs rarely occurred because transverse cracking was almost saturated due to tensile thermal stress induced during processing. Instead, tensile normal stress propagated and opened the as-processed TCs. Actually, as shown in Fig. 11 by arrows, TC openings appear larger for ±45° tensile specimens than those in ±45° compressive specimens even at the same shear stress. Considering that debonding initiates from tips of TCs at bundle interfaces, tensile normal stress in ±45° tensile methods enhanced onset and propagation of debonding as shown schematically in Fig. 10. Thus, the ±45° tensile tests exhibited the most remarkable development of the d_6 and ε_p among the three test methods. By contrast, in ±45° compressive tests, almost identical development of the d_6 and ε_p was observed to that in Iosipescu tests. We therefore conclude that compressive normal stresses contribute slightly to shear damage progress because transverse cracks do not open by compressive normal stress. The following two conclusions were obtained according to the discussions above: first, a ±45° tensile test is not appropriate for evaluating in-plane shear strength of present plain-woven C/C composite; second, although apparent shear behavior depends markedly on the test method used, principal damage mechanisms in the three shear tests are almost identical.

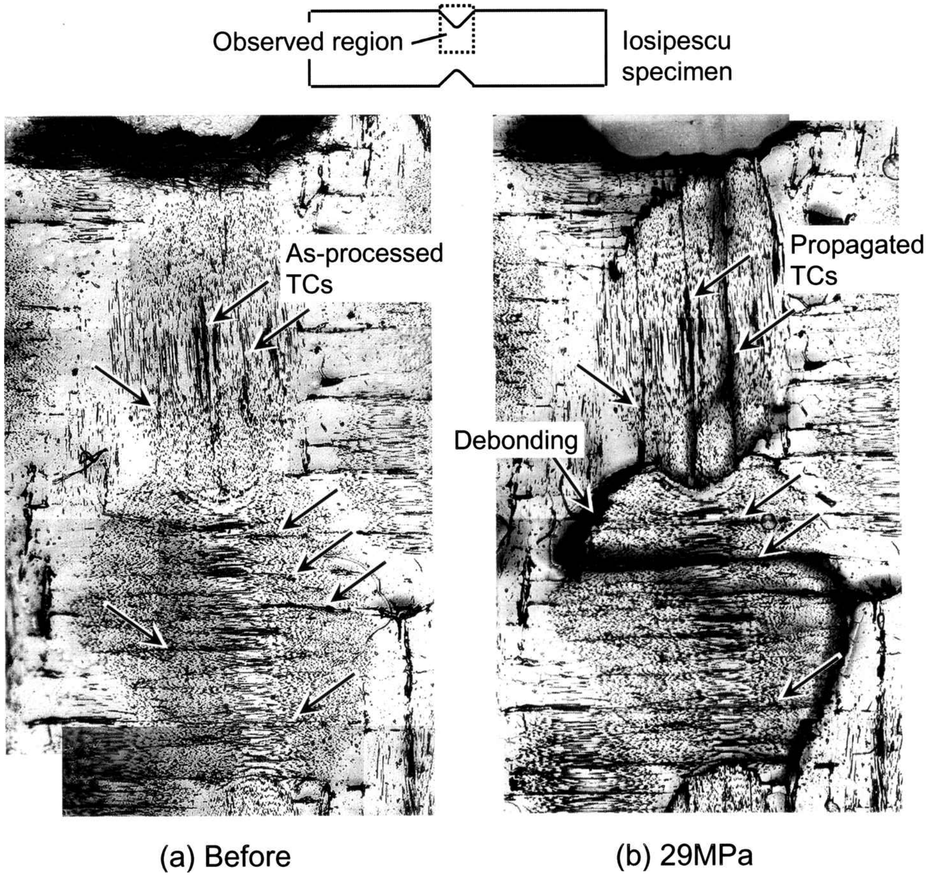

Figure 8. Typical microstructural changes appearing on top surfaces of Iosipescu specimens.

4. CALCULATION OF OFF-AXIS MECHANICAL BEHAVIOR

Finally, stress–strain behavior under various off-axis loadings was calculated to demonstrate normal stress effects on apparent mechanical behavior. Calculations were performed for two cases: considering and omitting normal stress effects.

Recently, Ladeveze *et al.* and other researchers have sought to develop a simplified CDM approach. Successful results were obtained in calculating non-linearity of various composite materials and structures [12, 15–18]. According to their model, progressive losses of moduli are described by damage parameters that depend on both shear and normal stresses. We hereafter denote normal stress effects on shear modulus degradation by shear/normal stress coupling effects. The Siron model was adopted here because it discusses averaged effects of many cracks on bulk elastic moduli. Such a simplified CDM is attractive for composites with complex fiber architecture. Important assumptions and calculation results will be explained in the following because the calculation procedure used in this study is almost identical to Siron's. Assumptions used are:

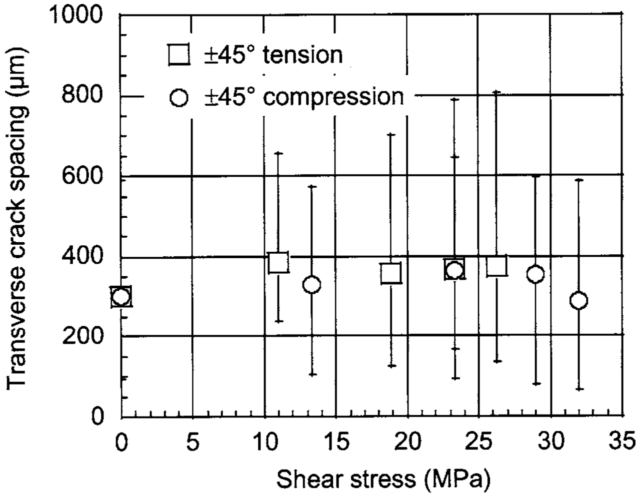

Figure 9. Relationships between TC spacing and applied shear stress during ±45° tensile and ±45° compressive tests.

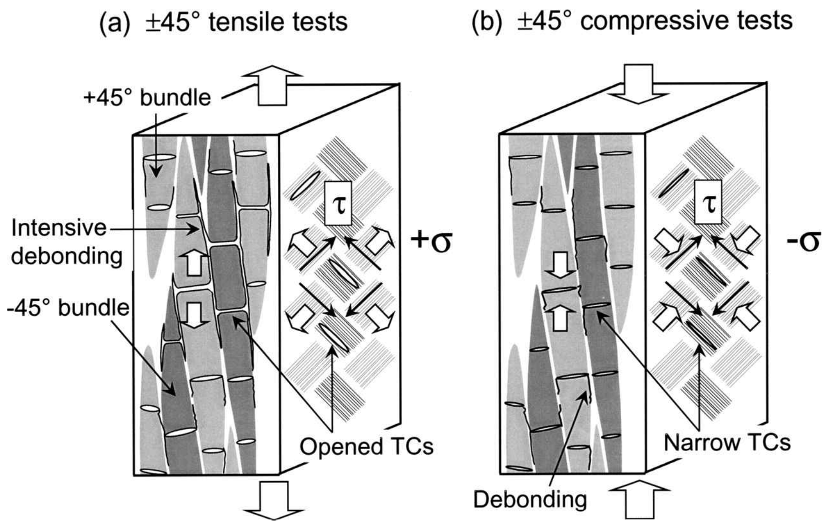

Figure 10. Estimated shear damage behavior during ±45° tensile and ±45° compressive tests.

(1) Only shear modulus degradation and development of shear permanent strain were considered because the present plain-woven C/C composite showed elastic and brittle behavior under loading parallel to the fiber direction as shown in Fig. 3.

(2) Shear modulus degradation and development of permanent strains occurs depending on both shear and normal stresses. The coupling effects were determined by comparing Iosipescu results (pure shear) with that in ±45° tensile and ±45° compressive test results shown in Fig. 5 [12].

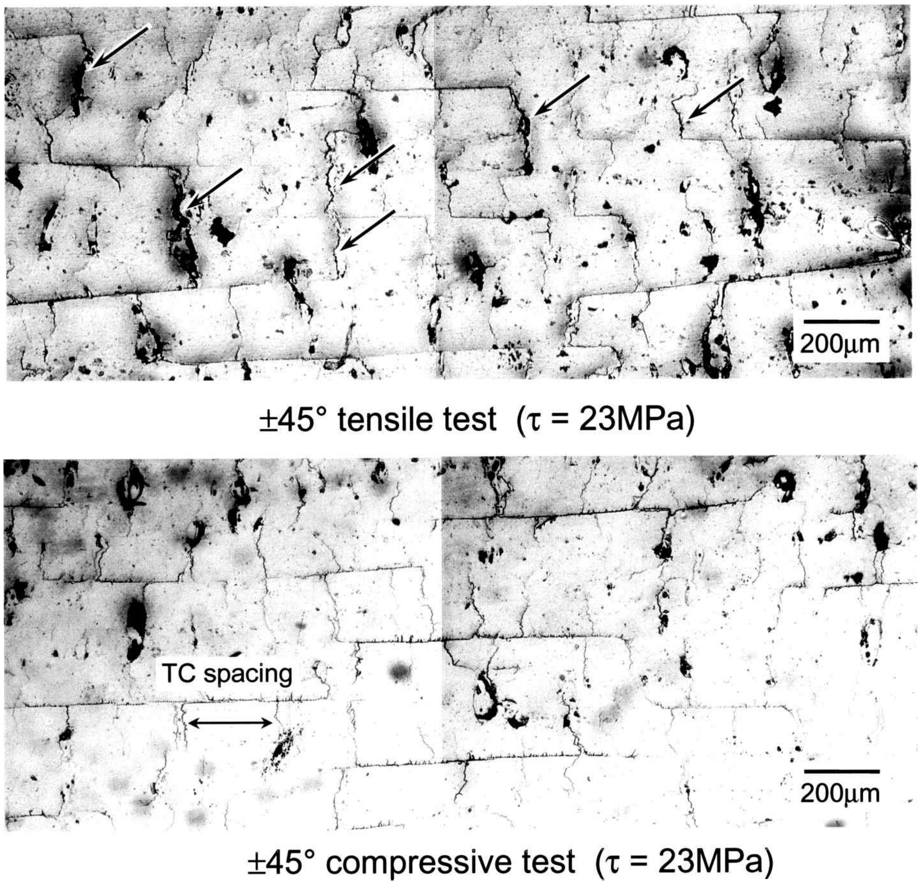

Figure 11. Opening and spacing of transverse cracks observed at the shear stress of 23 MPa in ±45° tensile and ±45° compressive tests.

(3) Adequate failure criteria must be defined because Siron's model only considers stiffness reduction. In the present study, final failure was supposed to occur when d_6 reached experimentally obtained maximum value of 0.7 or calculated longitudinal stress reached failure stress in corresponding experiments.

Figures 12a and 12b show comparisons of experimental and calculated stress–strain behavior in 10°, 22.5° and 45° off-axis tensile and compressive tests. Solid lines represent experimental results and thin and thick dotted lines correspond respectively to calculated results with and without consideration of coupling effects. For cases omitting coupling effects, shear moduli were degraded with respect only to shear stress. That is, shear modulus was degraded based on Iosipescu results. As shown in Fig. 12, good agreement was obtained when the shear/tensile coupling effect was taken into account, especially for off-axis tensile tests. However, a large discrepancy was obtained when the coupling effect was omitted. This result clearly demonstrates effects of tensile normal stress on shear damage progress.

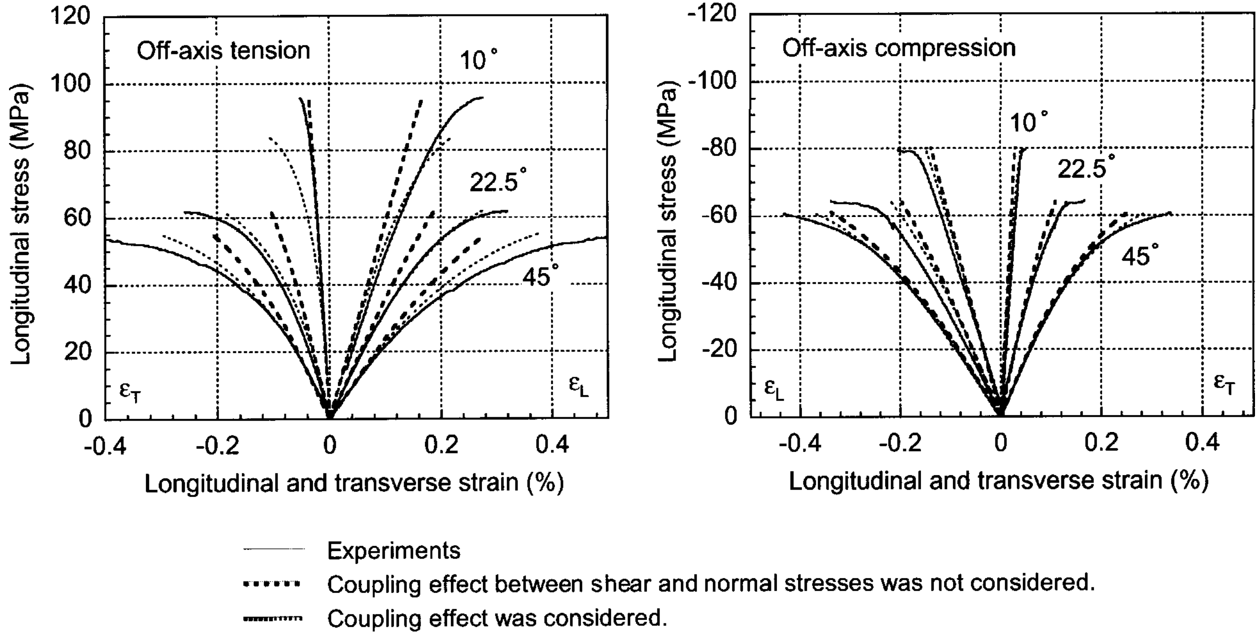

Figure 12. Comparison of the measured and calculated stress–strain curves in 10°, 22.5°and 45°off-axis tensile and compressive tests.

5. CONCLUSIONS

Three typical in-plane shear tests were performed for a plain-woven C/C composite and test methods were evaluated based on experimental results and observed damage mechanisms. Comparison of shear stress–strain relations revealed that tensile normal stress enhances shear damage evolution whereas compressive slightly contributes to it. Thus, we conclude that the $\pm 45^\circ$ tensile test method is inappropriate for evaluating in-plane shear strength of the present plain-woven C/C composite. Although apparent shear behavior depended markedly on the test method used, a common damage process with propagation of transverse cracks and debonding among fiber bundles was observed in the three test methods. Finally, using a CDM approach, normal stress effects on stress–strain behavior under various off-axis loading was demonstrated. Good agreement between calculated and experimental results was obtained when shear/normal stress coupling effects were taken into account.

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