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Tension–Compression Fatigue of a Hybrid Polymer-Matrix/Ceramic-Matrix Composite at Elevated Temperature

Marina Ruggles-Wrenn * and Joshua Schmidt

Department of Aeronautics & Astronautics, Air Force Institute of Technology, Wright-Patterson Air Force Base, Ohio, OH 45433-7765, USA

* Correspondence: marina.ruggles-wrenn@us.af.mil

Abstract: Fully reversed tension–compression fatigue of a hybrid material comprising polymer matrix composite (PMC) co-cured with a ceramic matrix composite (CMC) was investigated. The PMC portion had a polyimide matrix reinforced with 15 plies of carbon fibers woven in an eight-harness satin weave (8HSW). The CMC portion had three plies of a quartz-fiber 8HSW fabric in a zirconia-based ceramic matrix. The hybrid PMC/CMC was developed for use in aerospace thermal protection systems (TPS). Hence, the experimental setup aimed to simulate the TPS service environment—the CMC side was kept at 329 °C, whereas the PMC side was open to laboratory air. Compression stress–strain response was studied, and compressive properties were measured at room and elevated temperature. Tension–compression fatigue tests were conducted at elevated temperature at 1.0 Hz. The evolution of tensile and compressive strains with fatigue cycles, as well as changes in the stress–strain hysteresis behavior and stiffness were examined. The tension–compression fatigue of a PMC with the same constituents and fiber architecture as the PMC portion of the PMC/CMC was studied for comparison. Tension–compression fatigue was found to be more damaging than tension–tension fatigue for both materials. The PMC outperformed the PMC/CMC in tension–compression fatigue. Post-test examination showed widespread delamination and striking non-uniform deformation modes of the PMC/CMC.

Keywords: polymer matrix composites (PMCs); ceramic matrix composites (CMCs); multiple matrix composites; fatigue; high-temperature properties; mechanical properties



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1. Introduction

Advanced composite materials that combine high strength and stiffness with low weight have been gradually replacing conventional isotropic metallic alloys in various aerospace structural applications [1,2]. Among many advantages offered by fiber-reinforced composites is their exceptional design adaptability due to having a variety of constituent materials and fiber architectures [3]. Many airframe structural components must operate in elevated temperature environments. Examples include hot trailing edges of B-2 and C-17 wings, engine-related components (such as engine vanes, engine ducts, and exhaust flaps), and hypersonic vehicle airframes. These aerospace applications demand materials that have excellent thermal resistance and damage tolerance, corrosion resistance, as well as improved mechanical properties and performance.

An important class of lightweight aerospace composites is material systems made of high-temperature thermosetting polyimide resins with carbon fiber reinforcement [1]. In addition to high use temperatures, these materials possess excellent damage tolerance. The PMR-15 thermosetting polyimide developed by NASA [4] is accepted as a state-of-the-art matrix material for advanced high-temperature carbon fiber-reinforced composites. While the PMR-15 resin exhibits many attractive properties, it also contains carcinogenic elements. Hence, numerous efforts have been devoted to developing alternative polyimide resins. Among the alternative polyimides is the NRPE resin, developed by Performance Polymer

Solutions Inc. (P²SI/PROOF Research, Moraine, OH, USA) purposely to overcome the processing limitations of the PMR-15 resin stemming from intrinsic matrix chemistry [5]. The NRPE resin also promises to address another shortcoming of the PMR-15 resin, namely its borderline hot/wet performance. The NRPE resin is a high-temperature, structural thermosetting polyimide with a low-melt viscosity that exhibits hot/wet mechanical properties suitable for 288–316 °C service temperatures.

Advanced thermal protection systems (TPS) are key to the successful operation of many aerospace structures and components. Unitized multifunctional airframe materials that combine structural and insulating functions can offer dramatic weight reduction for air vehicles. Aiming to develop novel TPS materials and processing methods, many researchers have concentrated on ceramic matrix composites (CMCs) [6–8]. Recently, Performance Polymer Solutions Inc. (P²SI/PROOF Research, Moraine, OH, USA) pioneered a hybrid polymer-matrix/ceramic-matrix composite system processed by co-curing the NRPE polyimide resin with the SMP-730 preceramic polymer resin. The multiple-matrix composite is a PMC co-cured with a CMC layer, which serves as the TPS protecting the PMC.

The hybrid PMC/CMC composite system shows promise for use in high-temperature aerospace structural components. However, its performance under complex loading scenarios at elevated temperature remains to be explored. Mechanical properties and the mechanical behavior of the unitized PMC/CMC material must be evaluated via comprehensive testing in relevant environments. Recently, Wilkinson and Ruggles-Wrenn [9] evaluated basic tensile properties of the unitized PMC/CMC at 23 °C and at elevated temperature. Additionally, this study investigated the tension–tension fatigue behavior of the hybrid PMC/CMC composite at an elevated temperature [9]. Because 2D composites with 0/90 fiber orientation exhibit outstanding tensile strength and tension–tension fatigue resistance, it is often (erroneously!) assumed that composite structures will be subjected to tensile loads only. However, in realistic applications, composite materials experience a much broader range of loading conditions. In contrast to engineering alloys, composite materials are challenged under compressive loading. Yet compression properties and behavior as well as tension–compression fatigue response are important design criteria for carbon fiber-reinforced composites. While some recent efforts have focused on understanding the compression behavior of composites [10–15], accepted characterization methods are rare. Even less common in the literature are reports on the characterization of tension–compression fatigue behavior of 2D woven composites. Aerospace structural components are frequently subject to tension–tension fatigue with positive mean stress. However, cyclic loading with other mean stresses is just as likely. Hence, we cannot limit investigations of the fatigue performance to tension–tension cycling; composite performance under other types of cycles must be investigated as well. We aim to explore the fatigue performance of the hybrid material system consisting of a PMC co-cured with a CMC under tension–compression fatigue with zero mean stress at an elevated temperature.

2. Materials and Experimental Arrangements

The material system studied in this work was a unitized composite consisting of a CMC co-cured with a PMC (Figure 1). The goal of combining the two different types of composites was to produce a hybrid material system with a CMC layer that acted as a thermal barrier protecting the PMC. The CMC and the PMC parts were both reinforced with a 2D fabric woven in an 8HSW, but the matrix materials and fiber reinforcements were dissimilar. The PMC part comprised 12 plies of de-sized Cytec T650-35 carbon fiber fabric in a P2SI[®] NRPE polyimide matrix. The P2SI[®] NRPE resin, developed by Performance Polymer Solutions Inc. (P²SI/PROOF Research, Moraine, OH, USA) is a high-temperature structural thermosetting polyimide. It has low melt viscosity and is formulated to retain its structural integrity following prolonged exposures to temperatures up to 343 °C. The CMC part comprised 3 plies of 1059 HT sized JPS Astroquartz[®] III 4581 fabric in a C5 ceramic matrix. The C5 ceramic matrix, developed by Performance Polymer

Solutions Inc., is manufactured by mixing KDT HTT-1800 polysilazane-based pre-ceramic resin and yttria-stabilized zirconia with silica additives. The process of co-curing used to produce the hybrid PMC/CMC composite is proprietary. The 2D PMC/CMC comprises two distinctive materials. Therefore, the physical properties of the 2D PMC/CMC panels such as constituent percentages could not be readily measured. The overall thickness of the hybrid PMC/CMC panels was about 5 mm. The average thickness of the CMC portion was ≈ 1 mm, whereas the average thickness of the PMC portion was ≈ 4 mm. Thus, the plane of CMC/PMC co-cure was not positioned at the mid-plane of the hybrid 2D PMC/CMC composite.

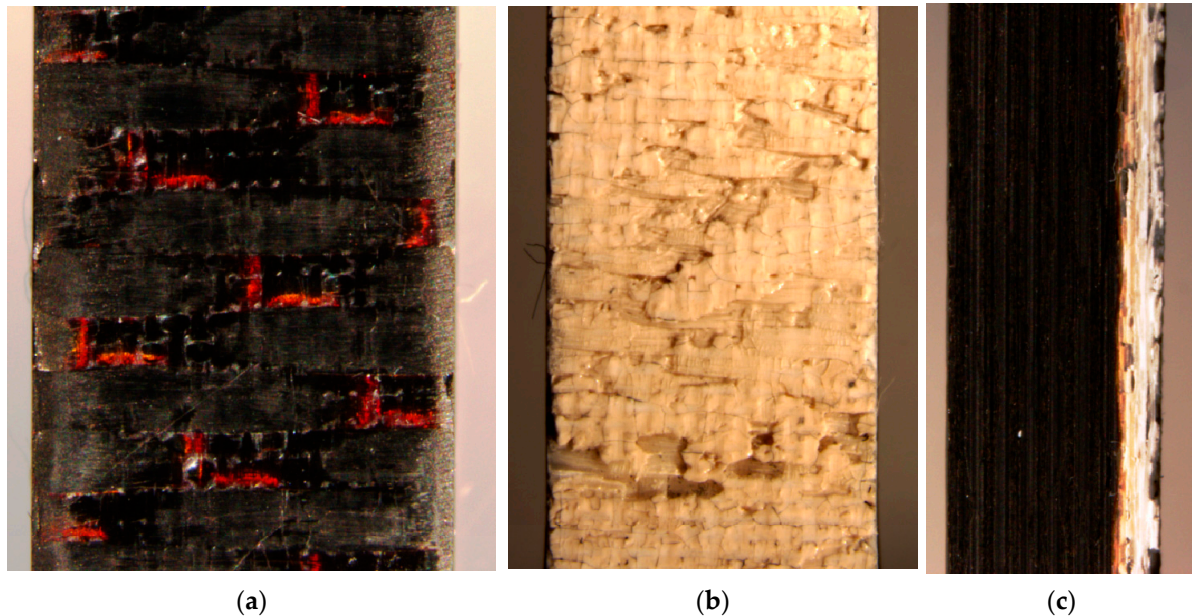
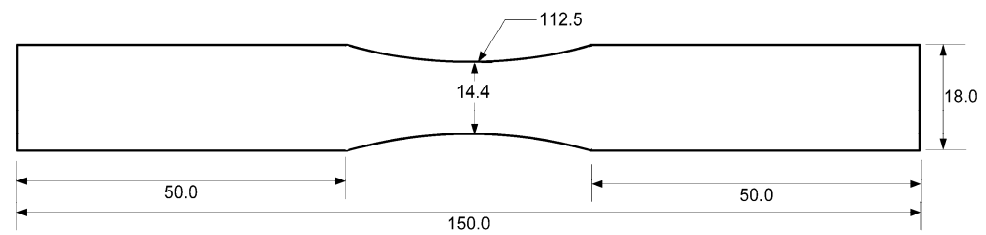


Figure 1. Two-dimensional unitized polymer/ceramic composite (2D PMC/CMC). (a) PMC face, (b) CMC face, (c) side view.

By definition, tension–compression fatigue involves compressive loading; hence, the potential for buckling failure modes exists and must be addressed. For that reason, buckling-resistant specimens (Figure 2) with an hourglass-shaped gage section were employed in all tension–compression fatigue tests.

Specimens with hourglass-shaped gage section were successfully used by Owen and Smith [16–18] to test chopped-strand-mat/polyester resin laminates in tension–compression cyclic fatigue. Additionally, other researchers successfully used specimens with hourglass-shaped gage sections for tension–compression fatigue testing of carbon fiber-reinforced PMCs [19,20] and for tension–compression fatigue testing of CMCs at elevated temperature [21,22]. Stress concentration intrinsic to an hourglass test specimen was evaluated. Finite-element analysis of the hourglass specimen showed that the maximum axial stress occurring at the edges in the middle of the hourglass section was only about 3% higher than the average axial stress.

The unitized PMC/CMC material system was designed specifically to perform as a thermal protection system (TPS) with a CMC layer serving as a thermal barrier for the PMC. The present study is a pilot effort to assess the fatigue durability as well as fitness of the co-cured 2D PMC/CMC for service in aerospace components designed to surround high-temperature environments. In order to imitate the realistic TPS operating conditions in the laboratory, mechanical tests were carried out with the CMC side of the specimen at 329 °C while the PMC side stayed open to laboratory air.



Dimensions in mm

All tolerances are ± 0.025 mm.

Figure 2. A tension–compression fatigue test specimen with an hourglass-shaped gage section. Note: this specimen design was successfully used in prior work presented in Refs. [19,21,22].

A detailed account of the experimental arrangements is given elsewhere [23,24]. For the readers' convenience, an abbreviated description is provided here. A servo-controlled MTS mechanical testing machine furnished with hydraulic water-cooled wedge grips, a small resistance-heated furnace, and a temperature controller was utilized in this study. An MTS Flex Test 60 digital controller was utilized to program the loading histories and to acquire data. Strain was measured with an MTS air-cooled high-temperature uniaxial extensometer (MTS model 632.53E-14) with a 12.5 mm gage length. In all high-temperature tests, the CMC (or the right) side of the test specimen was heated to 329°C as the PMC (or the left) side was exposed to laboratory air. In this study, elevated temperature is defined as $T_{\text{right}} = 329^\circ\text{C}$. Supplemental insulation inserts congruent with the shape of the test specimen were added to ensure that only one (CMC or right) side of the specimen was exposed to 329°C while the other (PMC or left) side stayed open to laboratory air. Steps taken to prepare the furnace insulation for testing are shown in Figure 3.

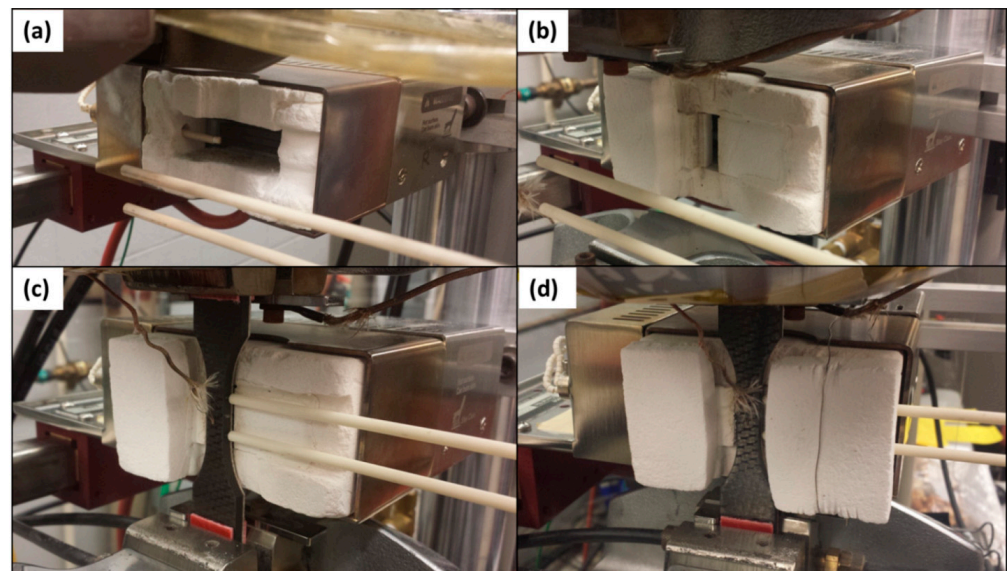


Figure 3. Setting up furnace insulation: (a) furnace interior, (b) furnace interior with insulation inserts, (c) test specimen is secured in the grips and extensometer extension rods are placed in contact with the specimen, (d) insulation blocks are added—specimen is prepared for testing.

To calibrate the furnace and the temperature controller for testing at elevated temperature, two thermocouples (K type) were attached to the specimen gage section. The temperature controller (utilizing non-contacting thermocouples exposed to the high-temperature environment in the vicinity of the test specimen) was adjusted to determine the setting needed to achieve the target temperature of the hot side of the test specimen. Once established, the controller setting was used for high-temperature testing. In all high-temperature

experiments, the CMC (or right) side of the specimen was heated to 329 °C at 10 °C/min, and kept at 329 °C for at least 45 min before mechanical loading was applied. Tested specimens were examined with an optical microscope (Zeiss Discovery V12 equipped with a Zeiss AxioCam HR digital camera).

In addition to monotonic compression tests at 23 °C and at $T_{\text{right}} = 329$ °C the experimental program included tension–compression cyclic tests with zero mean stress at $T_{\text{right}} = 329$ °C. Monotonic compression tests were performed with a constant displacement rate of 0.025 mm/s. Fully reversed tension–compression cyclic fatigue tests were performed at 1.0 Hz in load control with zero mean stress (i.e., ratio of minimum stress to maximum stress, $R = -1.0$). Fatigue runout was defined as 2×10^5 cycles without failure. All specimens that survived 2×10^5 cycles were tested in tension to failure at $T_{\text{right}} = 329$ °C to measure the retained tensile properties. Note that all test specimens had 0/90 fiber orientation, i.e., in all tests, the load was applied along the fiber direction.

3. Results and Discussion

3.1. Compression Properties and Stress–Strain Behavior

For the 2D PMC/CMC, room-temperature tensile properties were established from eight tension tests, and compressive properties were established from six compression tests. The tensile and compressive properties of the 2D PMC/CMC at $T_{\text{right}} = 329$ °C were also established from eight tension and six compression tests. Average tensile and compressive properties of the 2D PMC/CMC at 23 °C and at $T_{\text{right}} = 329$ °C are summarized in Table 1 with the corresponding percent coefficients of variation (COV) given in parentheses. Figure 4 shows typical stress–strain curves of the 2D PMC/CMC in tension and compression at 23 °C and at $T_{\text{right}} = 329$ °C. Given that the 2D PMC/CMC contains a 2D PMC portion, it is informative to compare and contrast the mechanical behavior and properties of the 2D PMC/CMC and those of a 2D PMC with the same constituents and fiber architecture. Therefore, the results of previous efforts [9,19] for a 2D PMC comprising 15 plies of de-sized Cytec T650-35 carbon fiber 8HSW fabric in a P2SI[®] NRPE polyimide matrix are included in Figure 4 and Table 1 for comparison.

Table 1. Tensile and compressive properties of the 2D PMC/CMC and 2D PMC at $T = 23$ °C and at $T_{\text{right}} = 329$ °C in laboratory air. Tensile data from Ref. [9].

	Tensile Strength (MPa)	E (GPa)	Compressive Strength (MPa)	E _C (GPa)
T = 23 °C				
2D PMC/CMC	682 (6.08)	56.8 (2.31)	176 (4.59)	51.4 (7.08)
2D PMC	838 (8.10)	57.3 (2.54)	422 (6.34)	51.8 (9.71)
$T_{\text{right}} = 329$ °C				
2D PMC/CMC	664 (6.51)	57.8 (3.98)	157 (2.09)	50.8 (6.72)
2D PMC	822 (6.88)	59.9 (3.39)	405 (8.87)	53.1 (8.37)

The results in Figure 4 and Table 1 reveal that the compressive strength of the 2D PMC/CMC is significantly lower than its tensile strength, while the compressive modulus appears to be close to the tensile modulus. At 23 °C, the compressive strength is only about 25% of the tensile strength. In contrast, the compressive modulus constitutes about 88–90% of the tensile modulus at both test temperatures. Similarly, compressive strength of the 2D PMC is lower than its tensile strength, but the difference is not as dramatic as in the case of the 2D PMC/CMC. The average compressive strength of the 2D PMC is close to 50% of its tensile strength. Compressive modulus of the 2D PMC represents about 90% of its tensile modulus, as was the case with the 2D PMC/CMC. Notably, high-temperature compressive strength and modulus obtained for the 2D PMC/CMC and the 2D PMC are close to the room-temperature values. Evidently, temperature has a minor effect on compressive properties of both composites.

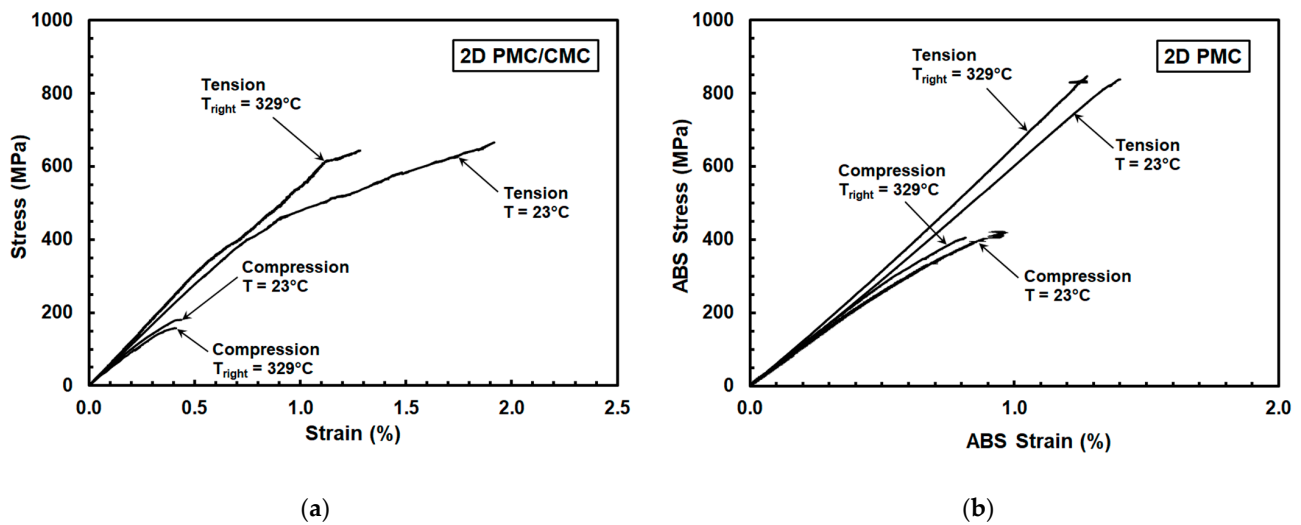


Figure 4. Tension and compression stress–strain curves produced at $T = 23^{\circ}\text{C}$ and at $T_{right} = 329^{\circ}\text{C}$ for (a) the 2D PMC/CMC and (b) the 2D PMC. ABS denotes absolute value. Data for the 2D PMC from prior work presented in Refs. [9,19].

The tensile and compressive moduli of the 2D PMC/CMC are very close to the respective properties of the 2D PMC. In contrast, the tensile and compressive strength of the 2D PMC/CMC are considerably lower than the corresponding strength values of the 2D PMC. Tensile strength of the 2D PMC/CMC is some 20% lower than the tensile strength of the 2D PMC, whereas compressive strength of the 2D PMC/CMC is about 60% below the compressive strength of the 2D PMC. To gain insight into such dramatic decrease in tensile and compressive strengths we examine the stress–strain behavior and aggressive delamination of the 2D PMC/CMC during monotonic tension and compression tests to failure (Figure 5).

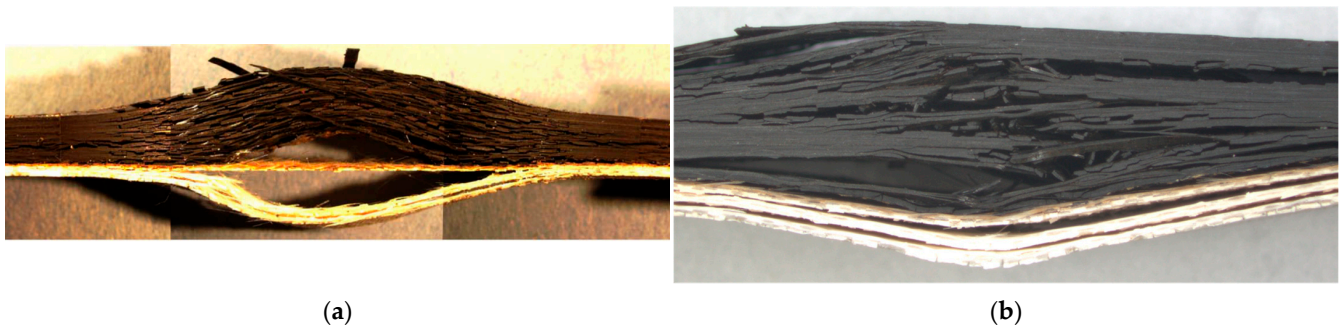


Figure 5. Typical non-uniform deformation of the 2D PMC/CMC in tension test (a) and compression test (b).

In a tension test, delamination of the test specimen becomes noticeable as the tensile stress reaches and goes above 300 MPa. Delamination progresses and becomes extreme with increasing tensile stress. The CMC and PMC plies curve outwards on the sides of the specimen (Figure 5a). It is noteworthy that while all PMC plies arch out, the CMC ply located at the co-cure plane stays reasonably flat. It is possible that the co-cure process raised the stiffness of this CMC ply. We also note that the PMC part of the specimen is noticeably thicker than the CMC part. Thus, the co-cure plane does not coincide with the mid-plane of the 2D PMC/CMC. As a result, the outward arching of the PMC and CMC plies is not symmetric relative to the mid-plane of the specimen. Such non-uniform deformation of the specimen is bound to add bending stresses to the applied tensile stress. Ply delamination together with non-uniform deformation spread well past the specimen

gage section. We attribute the lower tensile strength of the 2D PMC/CMC to extensive delamination and non-uniform deformation seen in tension test.

Damage and failure of composite materials under compression have been studied by many researchers [25–35]. By and large, compressive failure of a 2D composite is associated with fiber micro-buckling, fiber kinking and subsequent fiber fracture. Figure 6 shows a typical failure of a 2D PMC specimen in a compression test.

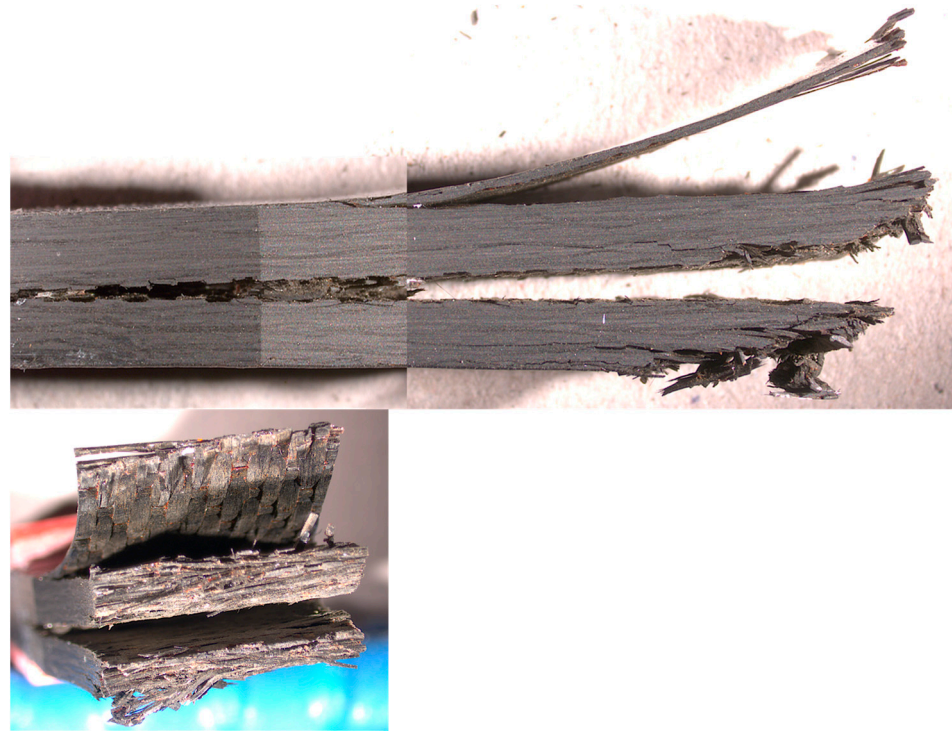


Figure 6. Typical failure of the 2D PMC in compression test, reproduced from Ref. [19].

Extensive ply delamination is evident. Delamination triggers formation of fiber kink bands across the entire specimen cross-section, followed by fiber fracture. In the case of the 2D PMC/CMC (Figure 5b), compressive damage also includes severe delamination. The fiber kink bands probably formed but did not encompass the entire specimen cross-sections. Fiber fracture is present but is not sufficient to cause the separation of the specimen into two distinct parts as in the case of the 2D PMC. In contrast, the non-uniform deformation noted in a tension test is also observed during a compression test. As in a tension test, the PMC plies and the CMC plies curve outwards under compressive loading. The arching of the plies is not symmetric relative to the specimen mid-plane. However, under compressive loading, none of the composite plies remain planar. Recall that under tensile loading, the CMC ply next to the co-cure plane stayed nearly planar. The non-uniform deformation of the 2D PMC/CMC observed under monotonic compression probably adds bending stresses to the applied compressive stress, resulting in a dramatically decreased compressive strength compared to that of the 2D PMC.

Compressive stress–strain behaviors of the 2D PMC/CMC and the 2D PMC are linear at first, but turn markedly nonlinear as the failure approaches. The 2D PMC fails in a catastrophic manner as compressive strain nearing 1%. In contrast, the 2D PMC/CMC fails at compressive strains not quite reaching 0.5%. Compressive failure of the 2D PMC/CMC does not have a catastrophic character, in the sense that the specimen does abruptly separate into two distinct parts. Under compression, the 2D PMC/CMC suffers massive delamination coupled with the non-uniform deformation. Compressive failure is signaled by the loss of load-carrying capacity.

3.2. Tension–Compression Cyclic Fatigue at Elevated Temperature

Fully reversed tension–compression fatigue tests were carried out at $T_{\text{right}} = 329^\circ\text{C}$ with zero mean stress (i.e., a ratio of minimum stress to maximum stress $R = -1$). Experimental results are presented in Figure 7 as fatigue (S–N) curves, where S is the maximum stress and N is the number of cycles to failure. Results of the tension–tension fatigue tests from an earlier effort [9] are plotted in Figure 7 for comparison. It is noteworthy that in tension–compression fatigue tests, all specimens failed during the compression segment of the fatigue cycle by a sharp drop in sustained load. Conversely, specimens tested in tension–tension fatigue failed by separation into two parts.

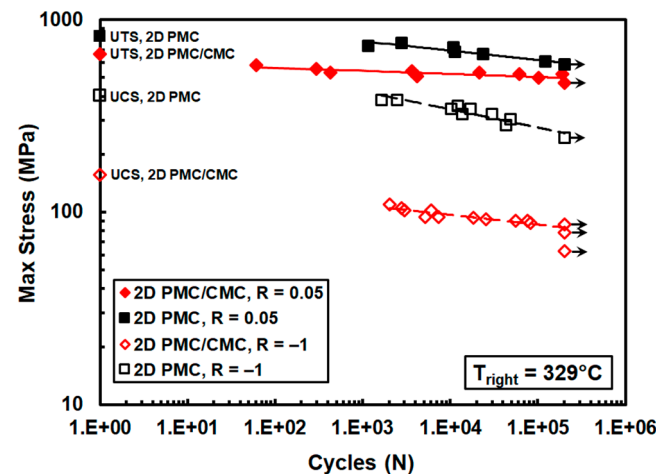


Figure 7. Fatigue S–N curves for the 2D PMC/CMC and the 2D PMC at $T_{\text{right}} = 329^\circ\text{C}$. Maximum stress, S_{max} , in cycle, serves as the basis for comparing fatigue S–N curves produced in two different mean-stress cycle types. UTS denotes ultimate tensile strength. UCS denotes ultimate compressive strength. Arrow denotes that specimen did not fail when the test was stopped. Tension–tension fatigue data for the 2D PMC from Ref. [9].

The results in Figure 7 demonstrate that tension–compression fatigue is significantly more destructive than tension–tension fatigue. In tension–compression fatigue tests, the 2D PMC/CMC achieved fatigue runout at 86.4 MPa, signifying that the fatigue limit lies between 86.4 and 90.3 MPa. Note that the fatigue runout stress of 86.4 MPa represents 55% of the composite’s compressive strength but only 13% of its tensile strength. In contrast, the runout stress obtained in tension–tension fatigue was 472 MPa (about 71% of the composite’s tensile strength), which is more than five times the runout stress of 86.4 MPa achieved in tension–compression fatigue. For any given cyclic life, the maximum stress in tension–tension fatigue is typically over five times that in tension–compression fatigue. Including compressive loading in a fatigue cycle drastically decreases the cyclic life and reduces the fatigue limit of the 2D PMC/CMC.

Examination of the S–N curves in Figure 7 reveals that the 2D PMC exhibits a much better tension–tension fatigue performance than the 2D PMC/CMC, achieving higher fatigue limit and longer fatigue lives for a given maximum stress. Notably, the tension–tension S–N curves obtained for the 2D PMC/CMC do not parallel those obtained for the 2D PMC. Hence, the reduced tension–tension fatigue performance of the 2D PMC/CMC is not exclusively due to its lower tensile strength caused by microstructural defects. The addition of the co-cured CMC plies influences the fatigue mechanism and accelerates the deterioration of fatigue strength with cycles. The 2D PMC also outperforms the 2D PMC/CMC in tension–compression fatigue, as is demonstrated by its higher fatigue limit. The 2D PMC achieves tension–compression fatigue runout at 243 MPa, which represents nearly 30% of the composite’s tensile strength and about 60% of its compressive strength. Tension–compression fatigue runout stress obtained for the 2D PMC also is about three times that obtained for the 2D PMC/CMC. Once again, the tension–compression S–N curves

for the two materials are not parallel. Thus, the degradation of tension–compression fatigue performance cannot be attributed exclusively to the lower strength of the 2D PMC/CMC. Once again, the co-cure of two dissimilar composites results in non-uniform deformation under tension–compression cycling, affecting the fatigue performance and accelerating fatigue failure.

Figure 7 presents the fatigue S–N curves for the two cycle types on the basis of the maximum stress, S_{max} , in the cycle. It is seen that S_{max} does not correlate in the results obtained for the 2D PMC/CMC composite well; the S–N curves obtained for the two different cycle types have different slopes. The same observation can be made regarding the fatigue S–N curves obtained for the 2D PMC. Figure 8 compares the S–N curves for the two cycle types with the stress amplitude (or alternating stress), S_a , as the correlating parameter. In the case of the 2D PMC, the results obtained for two cycle types now form a more compact group, but a noticeable difference in slopes persists. In the case of the 2D PMC/CMC, the two cycle types still yield somewhat different slopes and do not form a compact group. These results are not entirely surprising. Several researchers [16,36–38] have noted that results obtained for the woven 2D composites in tension–tension fatigue differed considerably from those obtained in tension–compression fatigue. The S–N curves exhibit different slopes. The failure modes are also different.

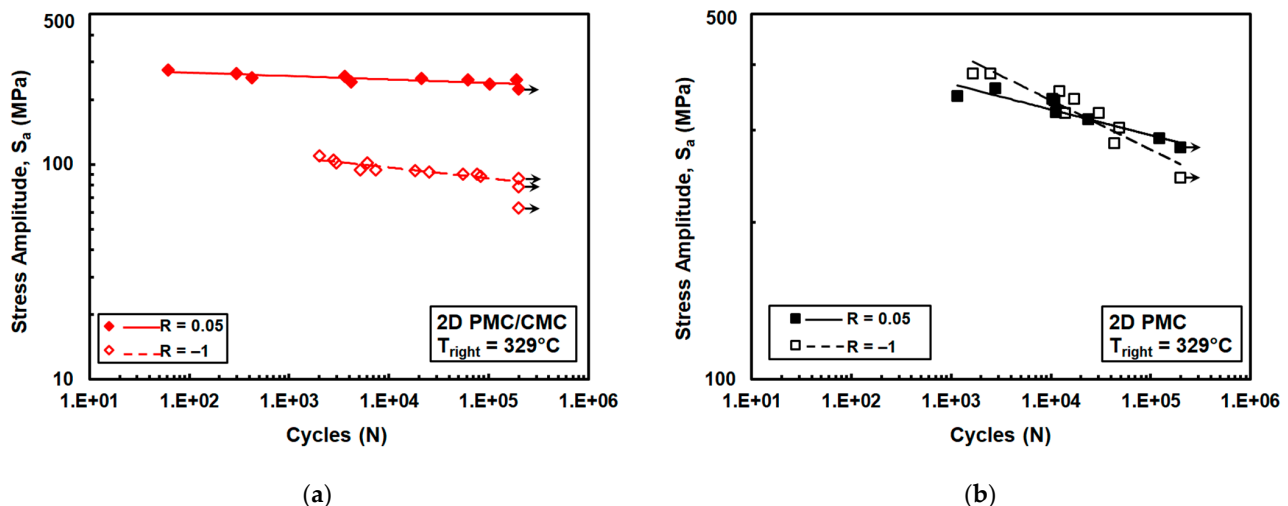


Figure 8. Fatigue S–N curves for the (a) 2D PMC/CMC and (b) the 2D PMC at $T_{right} = 329\text{ }^{\circ}\text{C}$. Stress amplitude, S_a , serves as the basis for comparing fatigue S–N curves produced in two different mean-stress cycle types. Arrow denotes that specimen did not fail when the test was stopped. Tension–tension fatigue data for the 2D PMC from Ref. [9]. Tension–compression fatigue data for the 2D PMC from Ref. [19].

Figures 9 and 10 present typical evolution of the stress–strain hysteresis response under tension–compression fatigue for the 2D PMC/CMC and the 2D PMC, respectively. In general, changing shape of the hysteresis behavior with cycles reflects changes in mechanical properties of the material. The area enclosed by the hysteresis loop represents a measure of energy dissipated in a cycle and can be associated with linear and/or nonlinear deformation. Results in Figs. 9a and 10a demonstrate that during fatigue tests with lower stress levels, both composites exhibit minimal strain ratchetting and nearly no change in stiffness. The hysteresis loops produced in these tests are very narrow; the composites' behavior is practically linear elastic. However, the hysteresis stress–strain loops produced by both composites are not entirely symmetric. In any given cycle, the largest compressive strain exceeds the largest tensile strain to some extent. This tendency is amplified with increasing fatigue stress, especially in the case of the 2D PMC/CMC (Figure 9b). Early in the test, the slopes of tension-going and compression-going parts of the fatigue cycle are approximately the same. As fatigue cycling progresses, the slope of the compression-going

part diminishes much faster than that of the tension-going part. The largest compressive strain becomes nearly two times the largest tensile strain in the cycle. Furthermore, the hysteresis loops produced later in test are markedly curved (see for example a stress–strain loop for cycle 6000 in Figure 9b). These phenomena signal a rapid damage buildup under compressive loading.

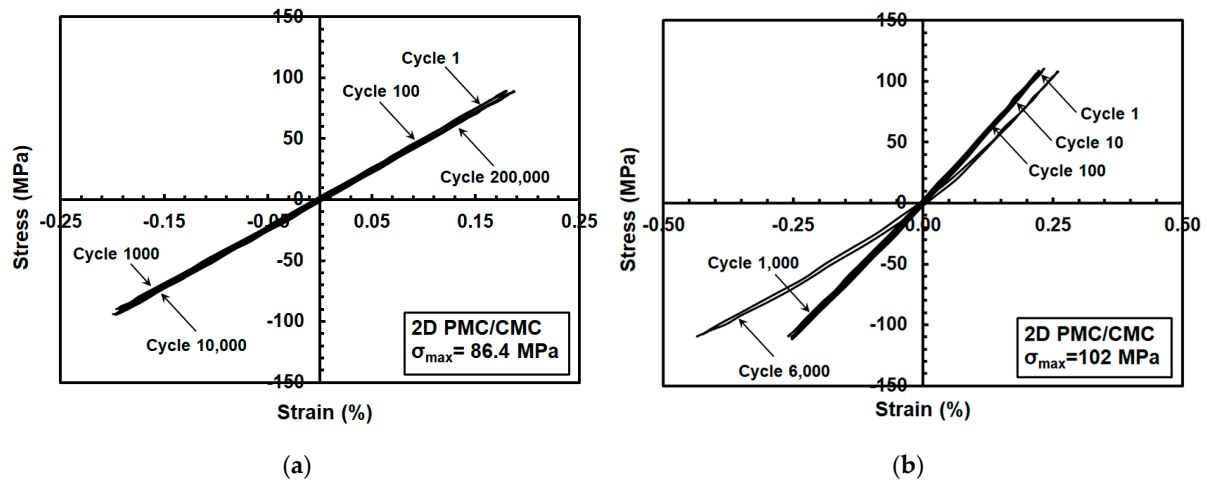


Figure 9. Typical development of stress–strain hysteresis behavior with fully reversed tension–compression cycling of the 2D PMC/CMC at $T_{\text{right}} = 329\text{ }^{\circ}\text{C}$: (a) $S_{\text{max}} = 86.4\text{ MPa}$, $N_f > 2 \times 10^5$, (b) $S_{\text{max}} = 110\text{ MPa}$, $N_f = 6063$.

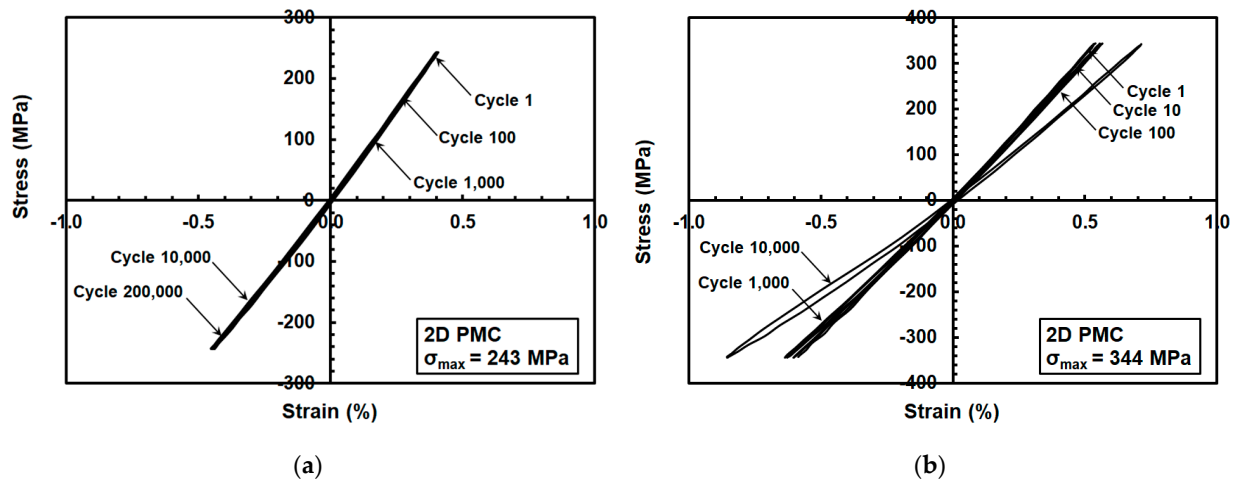


Figure 10. Typical development of stress–strain hysteresis behavior with fully reversed tension–compression cycling of the 2D PMC at $T_{\text{right}} = 329\text{ }^{\circ}\text{C}$: (a) $S_{\text{max}} = 243\text{ MPa}$, $N_f > 2 \times 10^5$, (b) $S_{\text{max}} = 344\text{ MPa}$, $N_f = 10,281$. Tension–compression fatigue data for the 2D PMC from Ref. [19].

Similar trends are observed for the 2D PMC (Figure 10b). The hysteresis loops become progressively less and less symmetric, with the slope of the compression-going part decreasing faster than that of the tension-going part. The trend, however, is not as pronounced as in the case of the 2D PMC/CMC. We attribute the rapid damage buildup in the 2D PMC/CMC to the non-uniform deformation of the test specimen. As mentioned earlier, the 2D PMC/CMC, which comprises two different composites co-cured together, produced non-uniform deformation in monotonic tension and monotonic compression tests. The PMC and CMC plies curved outwards. The CMC ply next to the co-cure plane remained virtually planar in a tension test, but curved outwards in a compression test. The 2D PMC/CMC produces similar non-uniform deformation under tension–compression fatigue. The CMC and the PMC plies curve outwards during tension-going part of the

cycle, regain their original shape as the load returns to zero, and then curve outwards during the compression part of the cycle, and once again regain their original shape at zero load (Figure 11). As the tension–compression fatigue tests continue, sizable tensile and especially compressive strains build up, and stiffness diminishes.

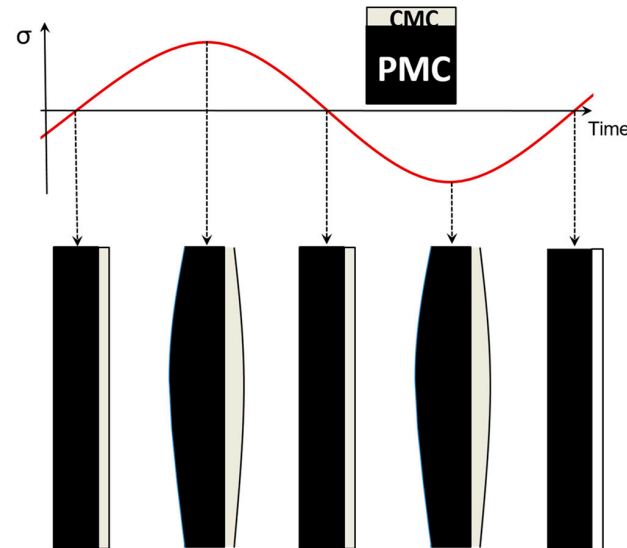


Figure 11. A schematic showing non-uniform deformation of the 2D PMC/CMC during tension–compression cycle.

Figure 12 presents the evolution of tensile and compressive strains with cycles for the 2D PMC/CMC and the 2D PMC. There is little accumulation of tensile or compressive strains in tension–compression tests conducted with lower fatigue stresses ($S_{\max} = 86.4$ MPa for the 2D PMC/CMC and $S_{\max} = 243$ MPa for the 2D PMC). Recall that these tests achieved runout completing 200,000 cycles without failure. Conversely, considerable accumulation of tensile and compressive strains is seen in tests conducted with higher fatigue stresses ($S_{\max} = 102$ MPa for the 2D PMC/CMC and $S_{\max} = 344$ MPa for the 2D PMC). For the 2D PMC/CMC (Figure 12a), compressive strain builds up faster than tensile strain. As seen in Figure 12a, tensile and compressive strains in cycle 1 are 0.21% and -0.26% , respectively. Tensile and compressive strains increase to 0.22% and -0.29% in cycle 1500, and to 0.26% and -0.44% in cycle 6000. Interestingly, tensile strains accumulated in tension–tension fatigue tests in prior work [9] are close to those accumulated in tension–compression tests. Of course, we note that the fatigue stresses (472–580 MPa) used in tension–tension fatigue tests [9] greatly exceed those used in tension–compression fatigue tests (63–110 MPa). For the 2D PMC (Figure 12b), compressive strain also grows faster with cycles than the tensile strain. For example, in the tension–compression fatigue test performed with $S_{\max} = 344$ MPa, tensile and compressive strains in cycle 1 were 0.49% and -0.60% . Tensile strain grew to 0.69%, whereas compressive strain reached -0.89% in cycle 10,000. The 2D PMC accumulated noticeably higher tensile strains in tension–compression fatigue compared to tension–tension fatigue. For instance, tensile and compressive strains accumulated in tension–compression fatigue tests conducted with $S_{\max} = 370$ MPa were 0.80% and -0.98% . Contrastingly, tensile strains accumulated in tension–tension fatigue tests with a higher $S_{\max} = 503$ MPa in an earlier effort [9] did not exceed 0.13%. These observations are hardly surprising. Specimens of the 2D PMC with 0/90 fiber orientation are highly resistant to tension–tension fatigue, hence the low strain accumulations. On the other hand, the 2D PMC/CMC exhibits non-uniform deformation with CMC and PMC plies delaminating and curving outwards under both tension–tension [9,23] and tension–compression cycling. As a result, similar tensile strains are accrued in tension–tension and in tension–compression fatigue tests.

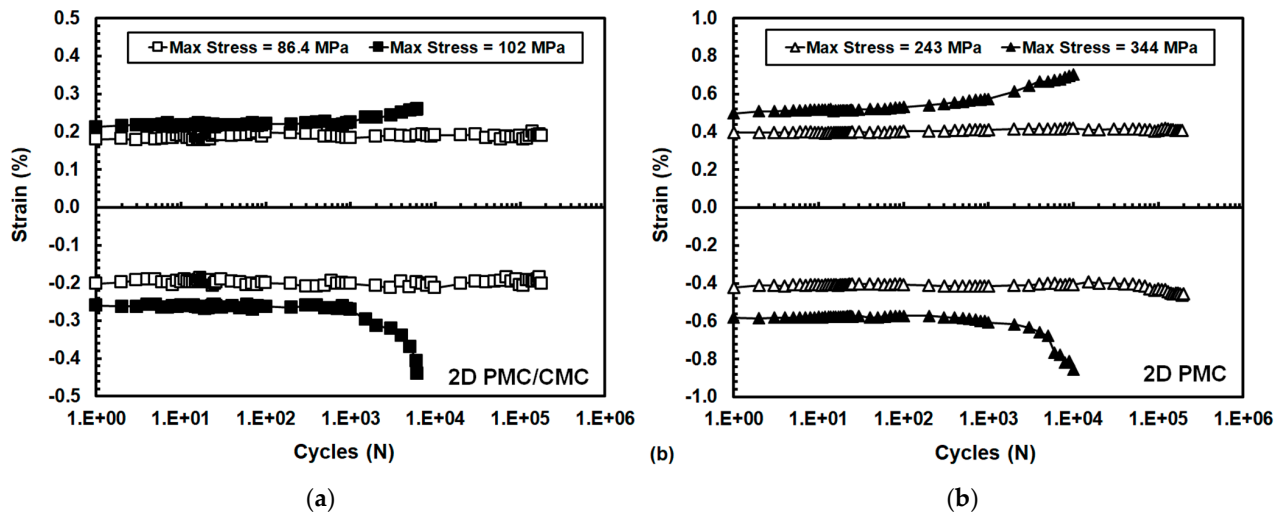


Figure 12. Maximum (or tensile) and minimum (or compressive) strains vs. tension–compression cycles at $T_{\text{right}} = 329\text{ }^{\circ}\text{C}$ for (a) the 2D PMC/CMC and (b) the 2D PMC.

It is instructive to examine the loss of stiffness with cycles (hysteresis modulus calculated using the maximum and minimum stress–strain data points in a load cycle), which reflects the damage development during fatigue. The change in normalized modulus (i.e., modulus normalized by the modulus measured in the first cycle) with fatigue cycles for the 2D PMC/CMC and the 2D PMC is presented in Figure 13.

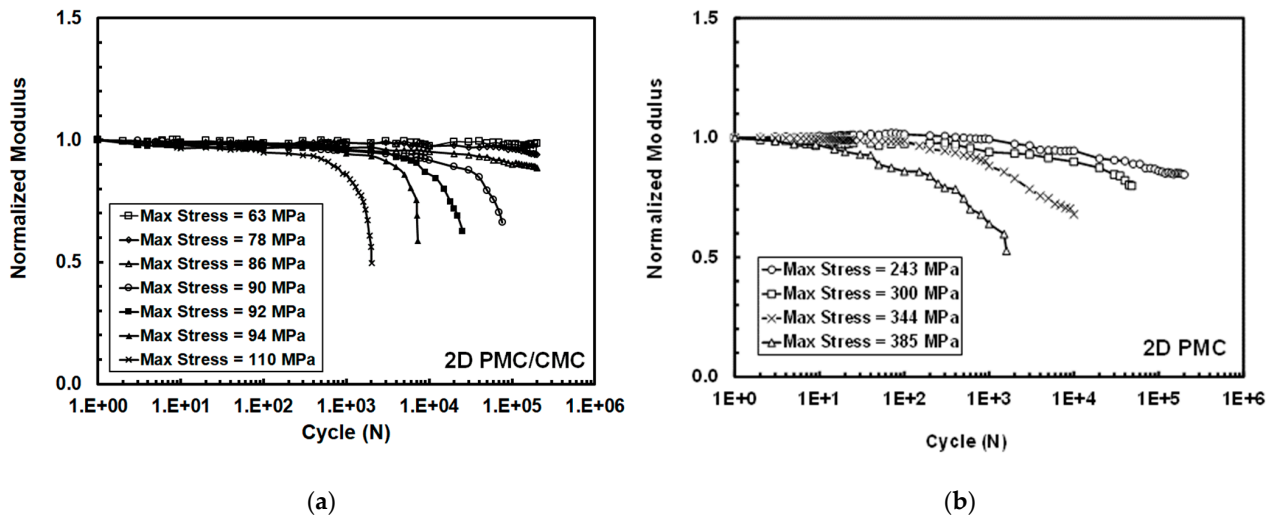


Figure 13. Normalized modulus vs. tension–compression cycles at $T_{\text{right}} = 329\text{ }^{\circ}\text{C}$ for (a) the 2D PMC/CMC and (b) the 2D PMC. Data for the 2D PMC from Ref. [19].

In tension–tension fatigue, the normalized modulus vs. cycles curves for both composites showed a classic pattern of gradual decrease followed by a rapid drop as the test specimen neared failure [9]. In the case of the 2D PMC/CMC the normalized modulus vs. cycles behavior adheres to the same pattern under tension–compression fatigue. In contrast, the normalized modulus of the 2D PMC diminishes continually with tension–compression cycles but does not drop sharply before failure. Predictably, for both composites, the normalized modulus decreases more rapidly in tests performed with higher fatigue stress. In runout tests where 200,000 cycles were completed without failure, the loss of normalized modulus was about 11% for the 2D PMC/CMC and about 15% for the 2D PMC. Compared to tension–tension fatigue, a greater drop in the normalized modulus is seen under tension–compression fatigue for both composites. Under tension–compression fatigue, the

modulus loss for the 2D PMC/CMC and the 2D PMC extends to 50%. In contrast, under tension–tension fatigue, the modulus loss did not exceed 40% for both composites [9].

All test specimens that achieved a fatigue runout of 200,000 cycles were tested in tension to failure to measure the retained tensile modulus and strength. The results in Figure 14 reveal that tension–compression fatigue is more damaging than tension–tension fatigue.

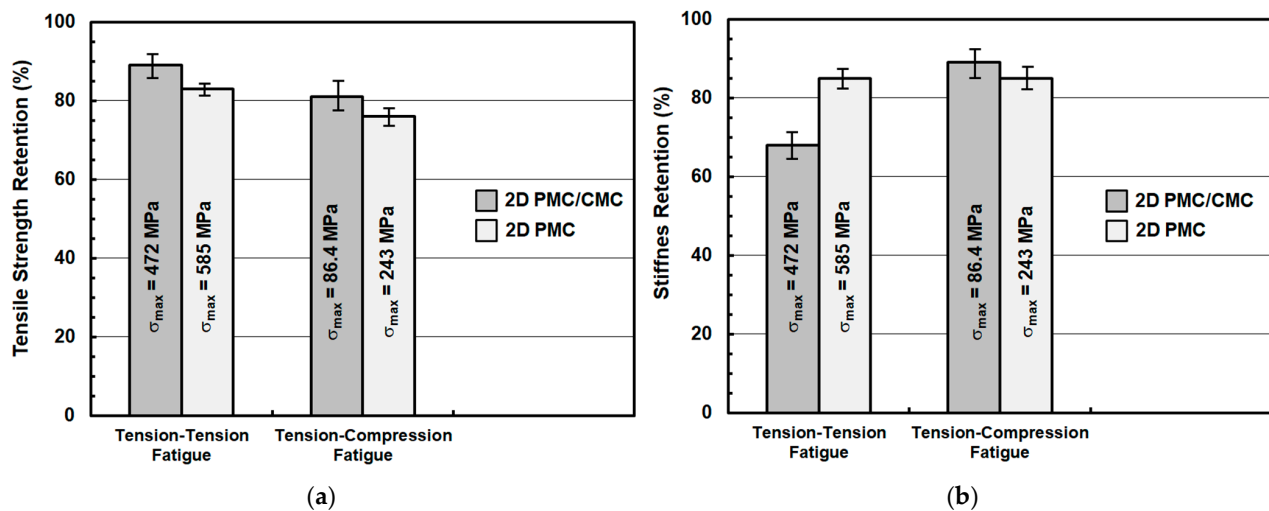


Figure 14. Retention of tensile strength (a) and modulus (b) of the 2D PMC/CMC and the 2D PMC specimens after 2×10^5 cycles at $T_{right} = 329$ °C. Tension–tension fatigue data for the 2D PMC from Ref. [9].

The 2D PMC/CMC retained 89% of its tensile strength following 200,000 tension–tension cycles with $S_{max} = 472$ MPa, but only 81% of its tensile strength after tension–compression cycles with a much lower $S_{max} = 86.4$ MPa. At first glance, the stiffness retention appears to show an opposite trend. The 2D PMC/CMC retains 68% of its tensile stiffness following tension–tension fatigue and 89% of its tensile stiffness after tension–compression fatigue. However, we note that the fatigue stress in tension–tension fatigue (472 MPa) was about five times that in tension–compression fatigue (86.4 MPa). It is reasonable to assume that stiffness retention would approach 100% in a tension–tension fatigue test performed with a low $S_{max} = 86.4$ MPa. The 2D PMC retains about 84% of its tensile strength following tension–tension fatigue with $S_{max} = 585$ MPa, but only about 75% of its tensile strength after tension–compression cycles with a much lower $S_{max} = 243$ MPa. Modulus retention is about 85% following either tension–tension or tension–compression fatigue. Once again, we note that the maximum stress was much higher in the case of the tension–tension fatigue than in the case of tension–compression fatigue. The addition of the co-cured CMC plies appears to be slightly beneficial—the 2D PMC/CMC exhibits better retention of its tensile strength after both tension–tension and tension–compression fatigue.

4. Composite Failure—Examination with Optical Microscopy

As noted earlier, the 2D PMC/CMC displayed non-uniform deformation under both tension and compression. Ply delamination is evident. The CMC and the PMC plies curve outwards under larger tensile or compressive loads (Figure 5). The 2D PMC subject to compressive loads also showed massive delamination. However, in this case, ply delamination led to formation of fiber kink bands spanning the whole specimen cross-section and eventual fiber fracture. The ultimate failure of the 2D PMC is synonymous with separation into two parts (Figure 6).

Typical failure of the 2D PMC/CMC in tension–tension fatigue (Figure 15a) involves the delamination of both PMC and CMC plies. In the case of the PMC part, widespread damage covers the entire specimen gage section. In the case of the CMC part, the damage is

more localized. The CMC part fractures at the bottom or top of the specimen gage section. The non-uniform deformation, repetitive curving and straightening of the PMC and the CMC plies result in additional bending stresses. We believe that these additional bending stresses are behind the localized failures in the CMC portion. Finally, the 2D PMC/CMC fails in tension by separation into two distinct parts.

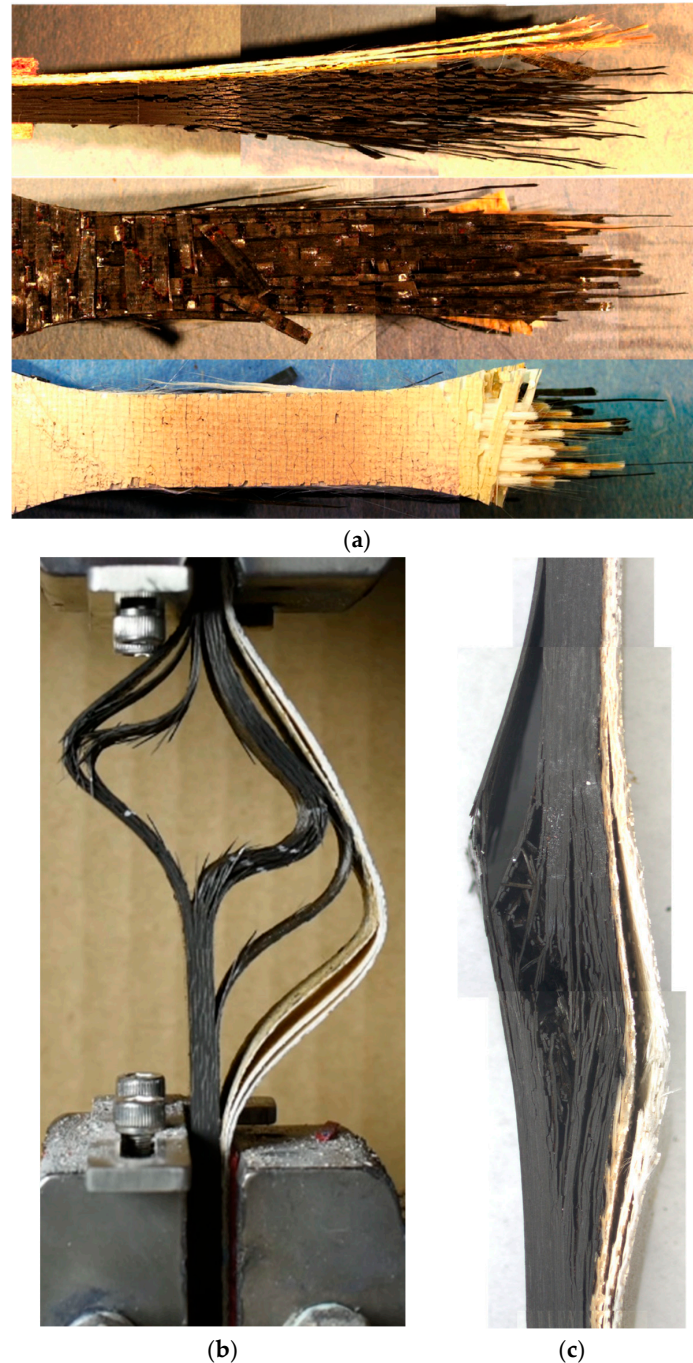


Figure 15. (a) Typical failure of the 2D PMC/CMC in tension–tension fatigue, reproduced from Ref. [9]. (b,c) Typical failure of the 2D PMC/CMC in tension–compression fatigue. (b) Failed specimen remains gripped in the testing machine. (c) Failed specimen is removed from the testing machine.

All specimens tested in tension–compression fatigue failed in compression. Figure 15b,c demonstrate that for the 2D PMC/CMC, the failure mode is similar to that observed in compression tests to failure (Figure 5b). Severe delamination of both PMC and CMC

portions is observed with the PMC portion showing more damage throughout the gage section than the PMC portion. Both PMC and CMC plies of the failed specimen are seen curving outwards. Bending of the CMC plies appears to proceed symmetrically, with the largest lateral deflection of the curved CMC plies occurring near the mid-length of the gage section. In contrast, delamination and subsequent bending of the PMC plies does not appear to be symmetric relative to the specimen mid-height. Larger lateral deflections of the PMC plies are seen in the upper half of the gage section. Once again, we attribute this phenomenon to the non-uniform deformation mode of the 2D PMC/CMC. Compression failure of the 2D PMC/CMC does not result in a separation of the specimens into two parts. Rather, compression failure manifests itself through a sudden drop in the load carrying capacity of the test specimen.

Typical failure of the 2D PMC in tension–compression fatigue is also different from that in tension–tension fatigue. Prevailing failure mechanisms in tension–tension fatigue are matrix cracking, ply delamination, fiber fracture and fiber pullout. In tension–compression fatigue, specimen failure takes place during the compression part of the cycle. The prevailing failure mechanism is ply delamination, which extends from the gage section of the specimen towards its gripping sections (Figure 16). Ply delamination probably stimulates formation of the fiber kink bands leading to shear failure of the fibers. Note that fracture planes form 45° with the loading direction (also the specimen axis), which also points to shear fiber failures.

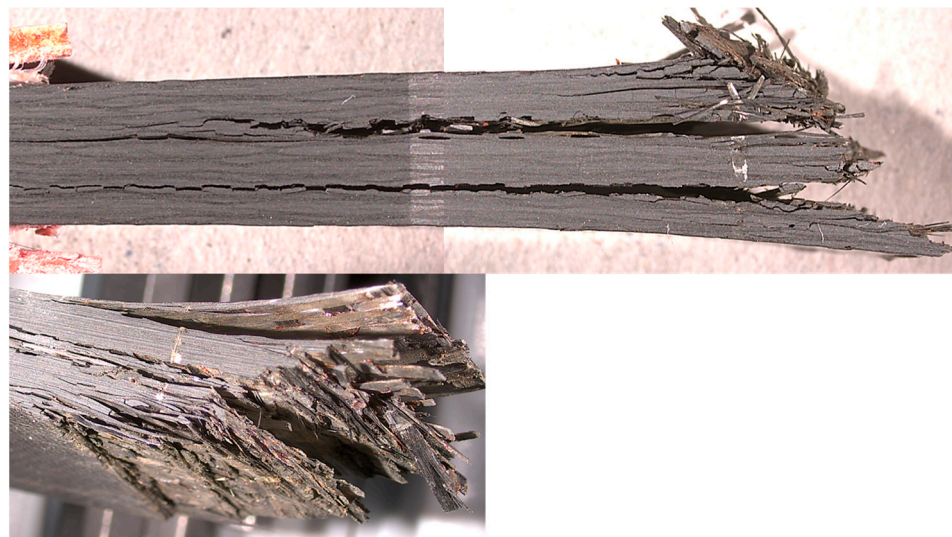


Figure 16. Typical failure of the 2D PMC in tension–compression fatigue, reproduced from Ref. [19].

5. Concluding Remarks

The compression stress–strain behavior of the 2D PMC/CMC and of the 2D PMC was investigated, and compression properties were measured at room and elevated temperature. Under compressive loading, the 2D PMC/CMC fabricated by co-curing two different materials displays massive delamination of both the CMC and PMC parts. Moreover, non-uniform deformation is seen throughout the gage section and beyond, with the CMC plies and the PMC plies arching outwards. At 23°C , the average compressive moduli of the 2D PMC/CMC and the 2D PMC are about 9% less than their tensile moduli. In contrast, the average compressive strengths of the two composites are at least 50% lower than their tensile strength. The compressive modulus of the 2D PMC/CMC is nearly the same as that of the 2D PMC. Conversely, the compressive strength of the 2D PMC/CMC is less than half of the compressive strength of the 2D PMC. We attribute this dramatic loss of compressive strength to the non-uniform deformation and massive delamination of the 2D PMC/CMC under compressive loading. Compressive stress–strain behavior of both composites is

markedly nonlinear. The compressive strength and stiffness of both composites are little influenced by the test temperature.

The tension–compression fatigue behavior of the 2D PMC/CMC and the 2D PMC was investigated at elevated temperature. All fatigue tests were carried out with the ratio of minimum to maximum stress $R = -1$ (i.e., zero mean stress). Tension–compression cyclic fatigue is substantively more damaging than tension–tension fatigue. Involving compression in the loading cycle drastically worsens the fatigue durability of both materials, the deterioration being much more extreme in the case of the 2D PMC/CMC. Generally, the 2D PMC delivers better tension–compression fatigue performance than the 2D PMC/CMC. Fatigue runout stress is 243 MPa (30%UTS) for the 2D PMC, but only 86.4 MPa (13%UTS) for the 2D PMC/CMC. Nevertheless, the fatigue runout stresses for the two composites represent approximately equal percentages of their respective compressive strength (55% for the 2D PMC/CMC and 60% for the 2D PMC). We believe that massive ply delamination and non-uniform deformation are behind the low compressive strength and the reduced tension–compression fatigue strength of the 2D PMC/CMC. Repetitive arching out and back of the delaminated CMC and PMC portions during tension–compression cycles add bending stresses to the applied axial stress. As a result, the 2D PMC/CMC is essentially experiencing combined tension/compression and bending. All specimens tested in tension–compression fatigue fail during the compression portion of the load cycle. The 2D PMC/CMC fails through extensive ply delamination and non-uniform deformation (i.e., arching of the CMC and PMC plies). The 2D PMC fails through matrix cracking, ply delamination and shear fiber failure. Neither the stress amplitude nor the maximum stress appear to correlate the fatigue S-N results obtained under tension–tension and tension–compression cycle types for both materials. We recognize that damage mechanisms operating in the 2D PMC/CMC under tension–tension or tension–compression cyclic loading are complex and call for further investigation.

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