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ROOM TEMPERATURE CREEP OF SIC/SIC COMPOSITES

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ABSTRACT

During a recent experimental study, time dependent deformation was observed for a damaged Hi-Nicalon™ reinforced, BN interphase, chemically vapor infiltrated SiC matrix composites subjected to static loading at room temperature. The static load curves resembled primary creep curves. In addition, acoustic emission was monitored during the test and significant AE activity was recorded while maintaining a constant load, which suggested matrix cracking or interfacial sliding. For similar composites with carbon interphases, little or no time dependent deformation was observed. Evidently, exposure of the BN interphase to the ambient environment resulted in a reduction in the interfacial mechanical properties, i.e. interfacial shear strength and/or debond energy. These results were in qualitative agreement with observations made by Eldridge of a reduction in interfacial shear stress with time at room temperature as measured by fiber push-in experiments.

INTRODUCTION

Time dependent crack growth of non-oxide ceramic matrix composites occurs at elevated temperatures due to the reduction of bridging tractions in a fiber-bridged matrix crack. This results from the oxidation of the interphase and/or fiber creep [1] that is indicative of time-dependent deformation at elevated temperatures. These materials are usually considered to be stable both microstructurally and mechanically at room temperature. However, in some recent experiments conducted at room temperature on woven SiC/SiC composites aimed at measuring the acousto-ultrasonic properties over a range of applied static stresses, time dependent strain accumulation resembling transient creep curves [2] was measured for composites with BN interphases. Time-dependent strain accumulation was not observed for C interphase composites. In this study, this apparent room temperature creep of BN interphase SiC/SiC composites, and the lack thereof for C interphase SiC/SiC composites, is presented and discussed in light of the possible mechanisms that could cause such behavior.

EXPERIMENTAL

Several different chemically vapor infiltrated (CVI) SiC matrix composite systems were studied. All of the composites were reinforced with eight-harness satin, woven Hi-Nicalon™ SiC (Nippon Carbon, Tokyo, Japan) fibers. The composites either possessed interphases consisting of

CVI BN or carbon and matrices consisting of CVI SiC only or B_4C enhanced CVI SiC. In other words, four different composite systems were examined; however, the matrix enhancement had no effect on the accumulation of room temperature time-dependent strain. All the composites were manufactured by Honneywell Advanced Composites (formerly Dupont-Lanxide Advanced Composites, Newark, DE).

Tensile monotonic and unload-reload tests were performed at room temperature on a servo-hydraulic, uniaxial universal test system (Instron Model # 8500, Canton, Mass.). The loading rate was 70 MPa per minute. Strain measurements were made using a room temperature, clip on strain extensometer. Specimen gripping was accomplished through the use of hydraulic wedge grips. In-house software was utilized to run the load-controlled tests. The gage length of the extensometer was 12.7 mm and the strain range was ± 5 percent. During the tensile tests for loading or unloading conditions, load holds were required in order to conduct acousto-ultrasonic (AU) measurements [3]. During the static load condition, measurable time-dependent strain was recorded for some of the composite systems and not for others. Acoustic emission (AE) was monitored [4] with a Fracture Wave Detector (Digital Wave Corporation, Englewood CO) system utilizing wide band (50 to 2000 khz) sensors (Model B1025, Digital Wave Corp.). The AE system was actively monitoring the specimens at all times except for when AU measurements were performed. The AU measurements took less than one minute and were performed after the time dependent strain rate had slowed considerably. The time at a given static stress condition ranged from one to five minutes.

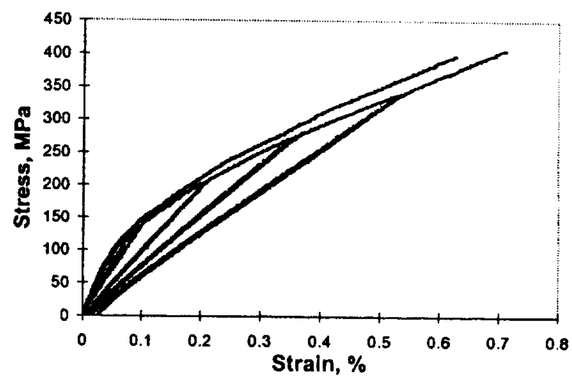

Figure 1: One monotonic and two unload-reload tensile stress-strain curves for C interphase, enhanced CVI SiC matrix composite.

RESULTS

Monotonic and unload-reload tensile stress-strain data for a carbon interphase composite system and a BN interphase composite system are shown in Figures 1 and 2, respectively. For each of the stress-strain curves in Figures 1 and 2, the stress was held constant (for up to five minutes) at 70 MPa increments (70 MPa, 140 MPa, 210 MPa...) until failure except where the experiment was interrupted prior to composite failure. Note the strain accumulation that occurred during the stress hold for the BN interphase composite (Figure 2), i.e. the horizontal part of the stress-strain curves at various stresses, and the absence of such behavior for the C interphase composites (Figure 1). For the BN interphase composites, the periods of time-dependent strain accumulation only occurred when the

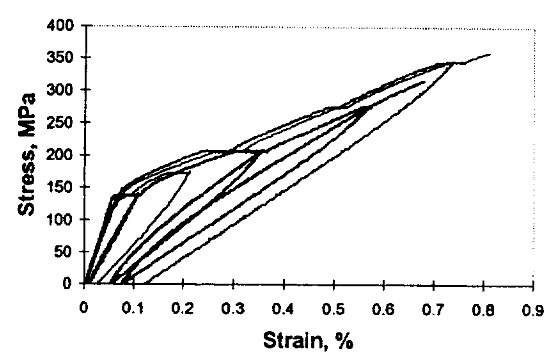

Figure 2: Four monotonic and one unload-reload tensile stress-strain curves for BN intephase, CVI SiC matrix composite.

previous maximum stress was surpassed. Therefore, time-dependent strain accumulation was not observed during the load-holds conducted while unloading the specimens or upon reloading at static stresses below the previous maximum stress condition.

The time-dependent strain accumulation is plotted as strain versus time in Figures 3 and 4 for the applied static stress of 210 MPa for both C interphase and BN interphase composites, respectively. The data plotted in Figures 3 and 4 were recorded utilizing the AE system. However, when AU measurements were taking place, the AE system was inactivated in order to prevent an overload of data (note where this is indicated in Figures 3 and 4). AU testing took from 50 to 150 seconds depending on the experiment.

Little deformation was measured for the C interphase composites (~ 0.005%). This is in contrast to the BN interphase composites, where considerable time-dependent strain accumulation was measured (~ 0.05% within the first three minutes at a static stress of 210 MPa). Similar time-dependent strain behavior was observed for other peak applied static stresses (140 to 350 MPa – Figures 5 and 6); however, the largest magnitude of time-dependent strain recorded for BN

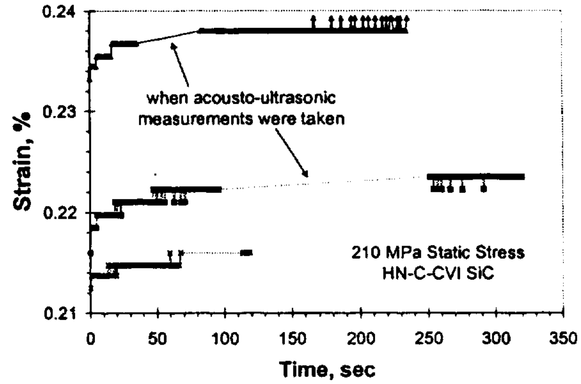

Figure 3: Time dependent strain accumulation for 210 MPa peak applied stress for three different tensile specimens of C interphase CVI SiC matrix composites.

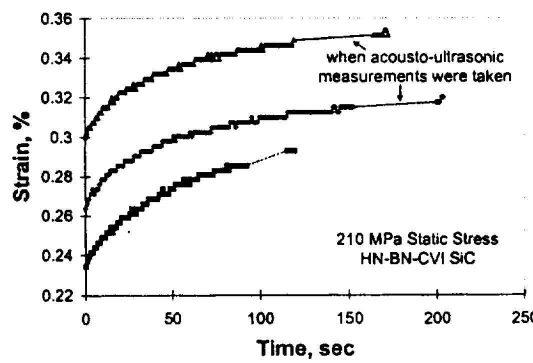

Figure 4: Time dependent strain accumulation for 210 MPa peak applied stress for three different tensile specimens of BN interphase CVI SiC matrix composites. The strain is the total tensile strain of the tensile test.

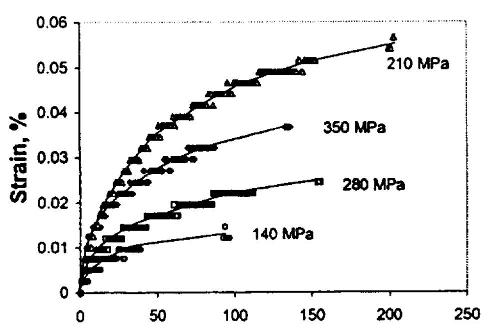

Figure 5: Time-dependent strain recorded at different stress holds during one tensile monotonic test for a BN interphase CVI SiC matrix composite. The strain plotted is the time dependent strain only.

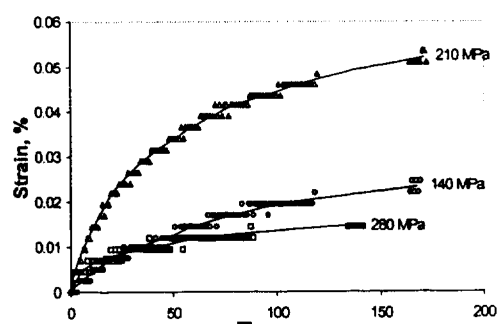

Figure 6: Time-dependent strain recorded at different stress holds during one tensile unload-reload test for a BN interphase CVI SiC matrix composite. The strain plotted is the time dependent strain only.

interphase composites was always the 210 MPa static stress condition (Figure 5 and 6). Note that for Figures 3 through 6, a time t = 0 corresponds to the beginning of the stress hold.

AE was occurring throughout the static stress holds. The AE activity recorded during the static stress conditions of 210 and 350 MPa are shown in Figure 7. The cumulative AE events and the cumulative AE energy versus time at each particular static stress were analyzed. In earlier studies, the proportion of cumulative AE energy had been shown to directly correspond to the crack density for SiC/SiC composites [4,5]. AE events that were the highest order of magnitude in energy were found to occur for the most part at stresses just before, during and just after the "knee" in the stress-strain curve. The AE energy from these "loud events" composed 80 to 90 % of the total cumulative AE energy. In other words, high AE energy events can be assumed to correspond to transverse matrix cracks. Lower AE energy events may correspond to smaller matrix cracks, fiber breaks, or sliding events. Note that only a few high-energy events occurred for the 210 MPa static stress condition and no high-energy events occurred during the 350 MPa static stress condition (high energy events were those that caused a sharp increase in AE energy as apparent in Figure 7). It was evident from the stress-strain curve (Figure 8) that the 210 MPa condition was at a stress significantly larger than the stress at which the "knee" in the stress-strain curve occurs (~ 145 MPa). By 210 MPa, 80% of the AE events had occurred as well as 95 % of the cumulative AE energy (Figure 8), which infers that matrix cracking was nearly saturated prior to achieving a stress of 210 MPa. Therefore, it appeared that most of the AE activity and time-dependent strain accumulation were not associated with large-scale crack growth.

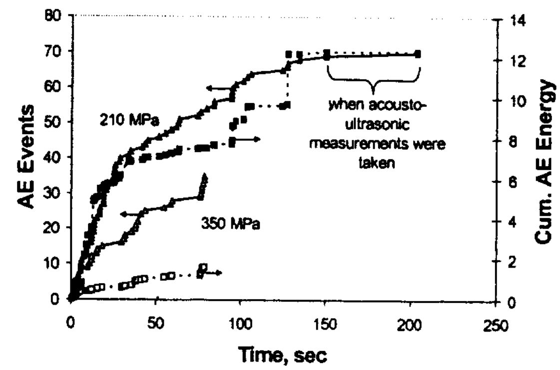

Figure 7: Acoustic emission activity for the 210 MPa and 350 MPa static stress condition of a BN interphase composite monotonic tensile test.

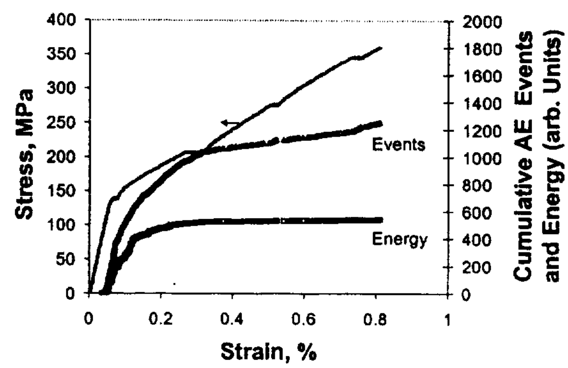

Figure 8: Monotonic (with holds) stress-strain curve and AE activity for a BN interphase CVI SiC matrix composite.

DISCUSSION

Presumably, the time-dependent strain must be due to the lowering of the interfacial properties, i.e. interfacial shear (sliding) stress, τ, and/or interfacial debond energy with the result of matrix crack growth, matrix crack opening, and/or increased fiber sliding lengths with time. It does not appear that substantial matrix cracking occurs given the low AE energy produced during the static stress condition (i.e., load holds) and the fact that matrix-crack saturation had most probably already occurred.

Eldridge [6,7] has demonstrated that the environment affects τ for both C and BN interphase composites at room temperature. For composites with C interphases [6-7], it was found that lower τ values were measured for fiber push-out and push-in experiments that were performed in room air compared to dry-air, nitrogen, or vacuum environments. When a number of fiber push-in cycles were performed consecutively with no time in between each cycle in room air for a C interphase SiC/SiC composite, it was found that each successive push-in cycle yielded a lower τ measurement (longer fiber sliding lengths) [7]. After about ten cycles a minimum τ was attained that remained constant with subsequent cycles. The reduced τ was attributed to the well-known improvement in lubricity of carbon when water and oxygen are adsorbed at the debonded interface, the major factor being water vapor [7,8].

For BN interphase composites, Eldridge [7] found that consecutive push-in cycles did not lower τ. However, for individual fibers that were initially pushed-in and then subjected to room air for 8 weeks τ was dramatically reduced compared to the initial measured value of τ. Eldridge has since found that a reduction in τ can be measured on already pushed-in fibers after an exposure period of five minutes in room-air [9]. Evidently, more time is needed, in comparison to C interphase composites, for a reduction in τ to occur for BN interphase composites. This may be due to a reaction or phase transformation process at the sliding interface (Eldridge [7] speculated that one possible mechanism was the condensation of water at the sliding surfaces [10]).

In this study, the effect of static stress on time-dependent strain accumulation was very small for C interphase composites. One might expect a greater effect given the reduction in τ measured by Eldridge [6,7] for C interphase composites from consecutive push-in experiments. However, Eldridge's findings were for cyclic tests and not static, as were performed in this study. Also, because the environmental effect appears to be very rapid for C interphase composites, within the time it takes to push a fiber in, and given the relatively slow loading rate used in this study, it was likely that the sliding interfaces were exposed to ambient air during loading once matrix cracking had occurred. In other words, by the time the static stress was reached, τ had been nearly fully reduced.

The results of this study certainly confirm the time-dependent reduction in τ for BN observed by Eldridge [7]; however, the time to observe an effect under static loading was more rapid than had been observed by Eldridge for push-in experiments [9]. The tensile stress-condition would cause the fiber to contract due to the Poisson effect, increasing the interfacial gap width. This would presumably have sped up the kinetics for environmental access along the sliding interface within the BN or between the BN and the fiber in contrast to a push-in test where fiber expansion would occur in the radial direction reducing environmental access.

Although these interface effects do not seem to harm the ultimate strength properties of these composites, they could lead to crack growth at room temperatures in humid environments if these types of composites are subjected to tensile stresses at room temperature. Also, whether or not these environmental effects reduce the durability of BN interphases at intermediate temperatures or have any bearing on intermediate or elevated temperature properties of SiC/SiC composites is unknown and should be further studied.

CONCLUSIONS

Static stress conditions were applied to SiC/SiC composites with C and BN interphases at room temperature. Significant time-dependent deformation was measured for BN interphase composites but little if any time-dependent deformation was measured for C interphase composites. The lack of a real time-dependent effect in C interphase composites appeared to be due to the rapid adsorption of water vapor at the sliding interface as was found by Eldridge [7] for similar composites. An environmental process also occurred for BN interphases in water containing environments that resulted in a lowering of interfacial shear strength with exposure time. This behavior also has been observed by Eldridge [7,9].

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