HTCMC5 Abstract

Creep and Stress-Strain Behavior after Creep for SiC fiber reinforced, Melt-
infiltrated SiC Matrix Composites

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Silicon carbide fiber (Hi-Nicalon Type S, Nippon Carbon) reinforced silicon
carbide matrix composites containing melt-infiltrated Si were subjected to
creep at 1315°C for a number of different stress conditions. This study is
aimed at understanding the time-dependent creep behavior of CMCs for desired
use-conditions, and also more importantly, how the stress-strain response
changes as a result of the time-temperature-stress history of the crept
material. For the specimens that did not rupture, fast fracture experiments
were performed at 1315°C or at room temperature immediately following
tensile creep. In many cases, the stress-strain response and the resulting
matrix cracking stress of the composite change due to stress-redistribution
between composite constituents during tensile creep. The paper will discuss
these results and its implications on applications of these materials for
turbine engine components.
CREEP AND STRESS-STRAIN BEHAVIOR AFTER CREEP FOR SIC FIBER REINFORCED, MELT-INFILTRATED SIC MATRIX COMPOSITES

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Objective

- To determine the creep behavior of the Hi-Nicalon Type S*, slurry-cast melt-infiltrated (MI) composite system including the retained stress-strain behavior after prolonged tensile creep
  - History dependent $\sigma/\varepsilon$ for modeling purposes
  - Creep-induced matrix compression to raise "proportional limit" stress [1,2]

* Nippon Carbon Co., Japan
Hi-Nicalon Type S, Melt-Infiltrated Composite System

- A very creep resistant and relatively strong fiber
- A relatively high matrix cracking matrix stress composite
  - Slurry SiC + melt Si fills in most of open porosity remaining after CVI SiC
  - Removes (fills in) stress concentrators present in CVI SiC matrix systems
  - Adds compressive stress to matrix due to volume expansion of Si on cooling and thermal expansion difference between Si (~3x10^{-6}/°C) and SiC (~4.5x10^{-6}/°C)

Experimental: Composite Processing

- Composite processing consisted of the following steps:
  - Stacking and BN CVI (interphase) of eight plies of woven fabric
  - SiC CVI in order to rigidize the preform and protect the fibers from melt-infiltration
  - Slurry-infiltration of SiC particles
  - Melt-infiltration of molten Si
- Two different matrix compositions were processed [3]:
  - High CVI SiC & low Si content (A Panels)
  - Low CVI SiC & high Si content (B Panel)
Experimental: Mechanical Behavior

- Tensile tests was performed on 152 mm long dogbone specimens (12.6 mm in grip and 10 mm in gage)
- Unload, reload tensile hysteresis tests were performed at room temperature to determine the residual stress in the matrix
- Elevated temperature tests were loaded monotonically (0.25 mm/min) to failure for fast fracture or to the predetermined load for tensile creep
- If tensile creep survived 100 hours, the specimen was unloaded to zero stress and either reloaded immediately to failure at 1315°C or cooled to room temperature and tensile hysteresis to failure was performed
- Elastic modulus was determined from linear regression of the 5 to 50 MPa portion of initial loading of o/ε curve.
- The 0.002% offset stress was used to characterize non-linearity (proportional limit)

Figure 1: Tensile Creep Curves
Figure 2: Strain Rate vs. Time

Table I: Tensile Data

<table>
<thead>
<tr>
<th>Panel</th>
<th>Experiment</th>
<th>Creep Stress, MPa</th>
<th>E(RT), GPa</th>
<th>E(1315) Initial Loading, GPa</th>
<th>E(1315) After Creep, GPa</th>
<th>E(RT) after creep</th>
<th>Minimum Strain Rate, sec⁻¹</th>
<th>Creep Strain, %</th>
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* Tested at 1315°C;  ** Did not fail in hot zone;  *** Prior to the onset of tertiary creep

FF = fast fracture;  R = tested at room temperature
Results

- Creep failure within 100 hours only occurred for the 172 MPa condition
- Primary creep dominated the creep-time curves (Figures 1 and 2). Perhaps a steady-state was reached for the 138 MPa creep condition.
- Retained strength after 100 hour creep was essentially equal to or greater than the 1315°C fast-fracture strength, even for specimens that exhibited 0.3% time-dependent strain. Although, fast-fracture of as-produced specimens never failed in hot zone for 1315°C testing whereas fast-fracture after creep usually did.

Figure 3: Stress-Strain History for Panel A2
### Table II: Offset Stress

<table>
<thead>
<tr>
<th>Panel</th>
<th>Experiment</th>
<th>Creep Stress, MPa</th>
<th>0.002% offset stress</th>
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- There is considerable non-linearity in 1315°C loading curve at relatively low stress (> 100 MPa) for the applied loading rate (see Table II). This is believed to be due primarily to creep during loading and not matrix cracking due to the absence of significant matrix crack formation (below).
- After tensile creep, reloading specimens resulted in a lower E; however, the "offset stress" was significantly increased (Table II), especially for A panels with the greater CVI SiC content.
- The time-dependent creep strain was essentially identical with the permanent strain measured after unloading from the creep condition and the difference in strain from after-creep fast-fracture and as-produced fast-fracture (see double-sided arrows in Figure 3).
Figure 4: Room-temperature tensile after creep (panel A2)

- Room temperature tensile behavior of after-creep specimen (Figure 4) exhibited all the qualities of increased compression in the matrix: higher "proportional limit" stress, higher AE activity stress, and higher residual compressive stress [4].

- Note that very little matrix cracking occurred during creep. For 103 MPa crept specimens, no creep-induced* matrix cracking could be discerned. For 138 MPa crept specimens, a few surface creep-induced microcracks were observed (Figure 5a). For the 172 MPa crept specimen, six periodic (spaced ~ 2 mm apart) internal microcracks were observed that resided in the same longitudinal tow three plies from the surface (Figure 5b). Several of these caused fiber failure; however, the matrix cracking seemed to not extend beyond the neighboring plies.

* Creep-induced cracking was easy to distinguish from matrix cracking caused during fast fracture. Matrix cracking due to fast-fracture was difficult if not impossible to observe without the aid of plasma etching. Creep-induced matrix cracking exhibited significant opening and oxidation for surface-exposed cracks.
Figure 5: Micrographs of crept specimens.

Panel A2
138 Mpa creep; Post-fracture at room temperature after creep.

Panel A2
172 MPA Creep

a. Surface microcrack in 138 MPa crept specimen.  
b. Internal unbridged microcracks in 172 MPa crept specimen.

Summary and Conclusions

- Woven Hi-Nicalon Type S MI composites exhibit excellent creep resistance at 1315°C and excellent retained properties after 100 hour creep for applied creep stresses up to 138 MPa.
- Tensile creep induced compression in the matrix, presumably due to the relaxation of the MI portion of the matrix which resulted in a higher matrix cracking stress composite.
- It may be possible to take advantage of this creep-induced phenomena for certain high stress applications, e.g., a turbine blade.
References


