CONSOLIDATION PROCESSING OF PVD Ti-6Al-4V COATED SiC FIBER COMPOSITES


3M Company, St. Paul, Minnesota, USA, *Department of Materials Science & Engineering, University of Virginia, Charlottesville, Virginia, USA

Fiber fracture during the consolidation processing of Ti-6Al-4V physical vapor deposition (PVD) coated SiC fibers has been studied. Metallographic observations of samples consolidated by both hot isostatic and vacuum hot pressing revealed that the fiber damage depended strongly on the geometry of fiber packing and the conditions of processing. In particular, damage was found to be a result of fiber crossing; fiber segments were subjected to bending by loads concentrated at the fiber crossover points. Higher processing temperatures and lower applied stresses (and rates of application) resulted in less damage. A cellular solids model is used to describe the constitutive response of an array of misaligned coated fibers subjected to consolidation stresses. Densification is allowed to occur by a combination of matrix (superplastic) creep and fiber elastic deflection into interfiber voids. The incorporation of this latter mechanism enables prediction of the process-dependent accumulation of fiber damage. The model is used here to explore “optimal” process cycles that could lead to full densification of the preform while minimizing or eliminating fiber damage. Process conditions which exploit the enhanced superplastic properties of the PVD processed Ti-6Al-4V matrix are identified with the model and have been experimentally confirmed.

1. INTRODUCTION

The attractive combination of high strength and stiffness together with reduced weight has led to increased interest in continuous fiber reinforced titanium alloy matrix composites as replacements for, or inserts within, conventional monolithic materials in many aerospace structural applications. Current research is focussing on the development of low-cost processing techniques for titanium-based and other metal matrix composites (MMC's). Efforts to produce MMC's with mechanical properties approaching (theoretical) predicted values have been only partially successful because of a limited understanding of the effects of processing on performance.²

Today, both electron beam and sputtering techniques can be used to evaporate and to deposit metal (alloy) coatings onto fibers (Figure 1) at relatively low cost. The physical vapor deposition of metallic coatings on ceramic fibers followed by consolidation is thus a potentially low cost fabrication technique for continuously reinforced MMC's.³ A number of factors contribute to this potential high-yield, low cost approach: the matrix material can be in inexpensive bar form, which is significantly cheaper than
Figure 1 PVD metal coated fibers (Ti-6Al-4V/Sigma 1240 SiC) are a potentially low-cost precursor for the fabrication of advanced metal matrix composites.

Figure 2 An array of PVD metal coated fibers prior to consolidation.

either the powders used in plasma spray deposition⁴ / tape casting,⁵ or the thin foil used in the foil/fiber technique⁶. The relatively high initial density of a well-aligned array of fibers (upto 90%, compared to 50-70% for other processes) requires smaller matrix deformations to fill in voids (Figure 2). This means less time is required to con-
solidate the composite and the yield can be high. Vapor deposition can be conducted over a wide range of fiber temperatures and does not create large temperature gradients within the fiber and so avoids the thermal shock characteristic of plasma spray processes. It is the most promising route for achieving uniform infiltration of small diameter fiber tows. Finally, the capability of metal coated fibers to withstand handling and bending may often make them more suitable precursors for near net shape forming compared to the monotapes of plasma spraying or tape casting.

The compositing of metal coated fibers has not been widely investigated as yet, however this approach is particularly attractive for titanium-based MMC’s because of titanium’s high reactivity. This was confirmed by Ward-Close and Partridge, who used EB evaporation to coat SiC fibers at low deposition temperatures with a variety of titanium alloys and found no observable interfacial reaction.\textsuperscript{7} In addition to control of fiber/matrix reactivity, the low deposition temperatures which can be achieved during PVD can also be exploited to produce very fine (nanocrystalline) grain sizes which can lead to enhanced (superplastic) creep behavior.\textsuperscript{8} Previous studies have shown that the consolidation process step following the production of the composite precursor is critical in determining final composite quality.\textsuperscript{9} Process conditions during consolidation determine the final density (i.e. residual porosity), fiber/matrix reactivity and fiber damage due to mechanical forces imposed on the fibers as matrix material is forced into interfiber voids.

Here, we experimentally explore the influence of consolidation processing conditions on the evolution of density and fiber damage in PVD Ti-6Al-4V-coated SiC fiber tows. Of particular interest is a dependence of the relative density on the applied stress and temperature (which is affected by the enhanced superplastic properties of vapor deposited microstructures) and the occurrence of fiber fracture in regions of the compact where fibers are not perfectly aligned (i.e. parallel to one another). The SiC (Sigma 1240) fiber was chosen as a model system to investigate the effects of fiber misalignment; its thick carbon/TiB\textsubscript{2} coating minimizes fiber strength loss at high consolidation temperatures due to reactions with the matrix. The experimental observations of fracture are rationalized using a “cellular solids” model of the consolidation process.

2. EXPERIMENTAL

Metal coated fibers were produced by sputtering ELI-grade Ti-6Al-4V on BP’s Sigma 1240 SiC fibers using a two-pass method. Sputtering or electron beam evaporation produces a fine grain structure with an average grain size of 0.1 \( \mu m \) or less. Chemical analysis after sputtering gave 89.8 wt\%Ti, 5.7 wt\%Al, 3.9 wt\%V. The matrix deposition usually exhibits some elliptical shape due to variations in the time each fiber surface faced the source during deposition. The Sigma 1240 SiC fibers (100 \( \mu m \))
diameter) were protected with a 2 μm thick C/TiB₂ coating. The average thickness of the metal matrix deposition was ~20 μm, giving a matrix volume fraction of approximately 50%.

Bundles of these coated fibers were consolidated either by VHP or HIP. Vacuum hot pressing was conducted at 10⁻⁶ Torr. The coated fibers (200-300) were placed in a 50 mm long x 6 mm wide and 6 mm deep, rectangular graphite channel die and hot pressed at temperatures ranging from 650°C to 950°C and at applied stresses ranging from 0.1 to 20 MPa. Pressure, time and ram displacements were continuously recorded during the consolidation cycle. Displacements were measured with a strain gage extensometer attached between the compression ram and a reference point. The fibers were deliberately placed in the die with no particular attention given to their alignment. Although the theoretical relative density of uniformly coated, aligned fibers is about 90%, actual initial densities ranged from 50-60%. This is due both to the nonuniformity of the sputtered coatings and to misalignment of the fibers.

HIP experiments were conducted by encapsulating the fibers within an evacuated (10⁻⁶ Torr) steel cannister (100x10x2 mm). The interior surfaces of the cannister were coated with yttrium oxide to prevent reaction with the titanium. Pressures ranged from 7 to 100 MPa with temperatures of 800-900°C. Applied pressure and temperature were measured throughout the HIP cycles.

The degree of misalignment (Figure 3) exerts a strong influence on both the densification response and the likelihood of fiber damage during consolidation. Careful attention was therefore paid to quantifying the degree of misalignment. The misalign

Figure 3. Light micrograph showing misalignment of metal coated fibers prior to consolidation.
ment of a given fiber can be determined with respect to the centerline of the die and because it varies from fiber to fiber, is treated as a random variable. Typically, the fiber misalignment angle (φ) was found to be normally distributed with a mean value of about 5°.

Densities of consolidated samples were measured by immersion (Archimedes' method). Fiber damage during consolidation was measured by dissolving the matrix material (using 10% hydrofluoric acid) from a specified volume of composite. The lengths of a significant number of fiber segments (>400) from within the test volume were measured from assembled SEM photographs and the degree of damage characterized by determining the distribution of the ratios of the fiber segment lengths to the length of an undamaged fiber. From this, the average fiber segment length, \( \bar{L} \), was calculated.

3. RESULTS

Table 1 presents the results of HIP consolidation experiments. In HIP cycles 1-6, the pressure was increased quickly to its soak value and then held constant (soak) for the remainder of the test whereas for 7-10, the pressure was slowly increased (ramped). The two samples which were consolidated to high relative densities using a constant pressure (nos. 3 & 6) also suffered extensive fiber damage, as evidenced by the low value of \( L/L_0 \) (where \( L_0 \) is the undamaged fiber's original length). Three of the tests in which the pressure was gradually increased (nos. 8, 9 & 10) achieved densities in excess of 90% while maintaining fibers at greater than 90% of their undamaged lengths. The conditions used for HIP-10 (two ramps, first from 0 to 10 MPa and then from 10 to 100 MPa, each over a two-hour period at 900°C) were the most successful, reaching full density with practically no fiber damage.

The results of the VHP tests are summarized in Table 2. Measurement of the compression fixture displacement permitted continuous recording of the density vs time during VHP tests. Figure 4 shows the evolution of density with time for VHP tests 1-6. The influence of increasing temperature and pressure on the amount of densification is clearly seen.

The densification and damage results given in Table 2 are best summarized in the form of consolidation maps. Figure 5 is such a map showing results for consolidation tests at 800°C. The left ordinate shows the relative density while the right shows the fiber damage parameter. The solid lines (with positive slope) indicate the density achieved for a after a given time; the lower line (t = 0) represents the density achieved immediately on application of the load, which is due only to plastic yielding. Further densification can only occur with time (creep and diffusive mechanisms); the density when these are active is indicated by the remaining contours (for times of 0.25, 1, 2, 4
The dashed curve (with negative slope) shows the fiber damage after consolidation for 8 h. Increasing the applied pressure results in accelerated densification, but at the expense of increasing fiber damage. For example, if a density of 95% or higher is

<table>
<thead>
<tr>
<th>TABLE 1. HIP CONSOLIDATION RESULTS</th>
</tr>
</thead>
<tbody>
<tr>
<td>CONSOLIDATION</td>
</tr>
<tr>
<td>------------------------------------</td>
</tr>
<tr>
<td><strong>Cycle Number</strong></td>
</tr>
<tr>
<td>HIP-1</td>
</tr>
<tr>
<td>HIP-2</td>
</tr>
<tr>
<td>HIP-3</td>
</tr>
<tr>
<td>HIP-4</td>
</tr>
<tr>
<td>HIP-5</td>
</tr>
<tr>
<td>HIP-6</td>
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</tr>
<tr>
<td>HIP-9</td>
</tr>
<tr>
<td>HIP-10</td>
</tr>
</tbody>
</table>

Figure 4. Densification response of metal coated fiber tows under various processing conditions.


Table 2. VHP CONSOLIDATION RESULTS

<table>
<thead>
<tr>
<th>Cycle Number</th>
<th>Temperature [°C]</th>
<th>Pressure [MPa]</th>
<th>Time [h]</th>
<th>Sample Relative Density</th>
<th>Fiber Damage (\frac{L}{L_0})</th>
</tr>
</thead>
<tbody>
<tr>
<td>VHP-1</td>
<td>700</td>
<td>10</td>
<td>8</td>
<td>0.92</td>
<td>0.5</td>
</tr>
<tr>
<td>VHP-2</td>
<td>800</td>
<td>1</td>
<td>8</td>
<td>0.7</td>
<td>0.94</td>
</tr>
<tr>
<td>VHP-3</td>
<td>800</td>
<td>10</td>
<td>8</td>
<td>0.94</td>
<td>0.82</td>
</tr>
<tr>
<td>VHP-4</td>
<td>800</td>
<td>12.5</td>
<td>8</td>
<td>0.98</td>
<td>0.71</td>
</tr>
<tr>
<td>VHP-5</td>
<td>800</td>
<td>17</td>
<td>8</td>
<td>0.99</td>
<td>0.72</td>
</tr>
<tr>
<td>VHP-6</td>
<td>900</td>
<td>10</td>
<td>8</td>
<td>0.99</td>
<td>0.98</td>
</tr>
<tr>
<td>VHP-7</td>
<td>950</td>
<td>1</td>
<td>8</td>
<td>0.73</td>
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<tr>
<td>VHP-8</td>
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<td>0.88</td>
<td>0.93</td>
</tr>
<tr>
<td>VHP-9</td>
<td>950</td>
<td>17</td>
<td>8</td>
<td>1</td>
<td>0.56</td>
</tr>
<tr>
<td>VHP-10</td>
<td>950</td>
<td>34</td>
<td>2</td>
<td>1</td>
<td>0.74</td>
</tr>
<tr>
<td>VHP-11</td>
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<td>8</td>
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<td>VHP-12</td>
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</tr>
<tr>
<td>VHP-13</td>
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<tr>
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<td>8</td>
<td>0.82</td>
<td>0.82</td>
</tr>
<tr>
<td>VHP-15</td>
<td>900</td>
<td>3</td>
<td>8</td>
<td>0.91</td>
<td>0.83</td>
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<tr>
<td>VHP-16</td>
<td>900</td>
<td>5</td>
<td>0.33</td>
<td>0.79</td>
<td>0.99</td>
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<tr>
<td>VHP-17</td>
<td>900</td>
<td>10</td>
<td>8</td>
<td>0.98</td>
<td>NA</td>
</tr>
<tr>
<td>VHP-18</td>
<td>900</td>
<td>15</td>
<td>2</td>
<td>0.9</td>
<td>0.92</td>
</tr>
<tr>
<td>VHP-19</td>
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<td>17</td>
<td>8</td>
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<tr>
<td>VHP-20</td>
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<td>0.25</td>
<td>1</td>
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<tr>
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<td>7</td>
<td>0.33</td>
<td>0.93</td>
<td>0.94</td>
</tr>
<tr>
<td>VHP-22</td>
<td>900</td>
<td>17</td>
<td>8</td>
<td>1</td>
<td>0.86</td>
</tr>
</tbody>
</table>

to be achieved within 8 h, the map indicates that the average intact fiber length will be about 80% of the undamaged length.

Figure 6 shows the results obtained at 900°C. A damage contour at \(t = 0.5\) h is shown in addition to one at 8 h. At this temperature, a relative density of 95% can be reached within 8 h while maintaining fiber lengths at 90% of the undamaged length or greater. The contour at 0.5 h indicates that practically no fiber damage accumulated this early in the process for applied stresses below 10 MPa.

Figure 7 shows a consolidation map for a constant applied stress (10 MPa); the density and fiber damage are shown as functions of temperature and time. An interesting feature of the map is the drop in density achieved (at 10 MPa) as the temperature is increased from 900 to 950°C. This anomaly implies an increased resistance to deformation at 950°C and is also reflected in an increase in fiber damage. The data
Figure 5. Consolidation map showing contours of constant density and fiber damage (T = 800°C).

Figure 6. Consolidation map at 900°C.
4. DISCUSSION

The experimental results presented in section 3 offer insight into how process conditions (applied stress and temperature) affect the evolution of density and fiber damage during consolidation of metal coated fiber arrays. The consolidation maps (Figures 5-7) clearly illustrate that the conditions needed to achieve high densities often lead to increased fiber damage; conditions must therefore be sought which allow the highest possible densities to be achieved while minimizing damage to the fibers. Our experimental study could be extended to determine these optimal processing conditions, but even for only two material state variables (density and fiber damage) and a single composite system, this would be a time consuming and costly task and would have to be repeated each time the matrix or fiber was changed. As an alternative, the mechanisms by which densification and fiber damage occur can be identified and dependence of this on the applied stress and temperature can be investigated using process models.

Fiber fracture during consolidation processing of MMC's is now a well-known phenomenon and has been observed in powder/fiber systems$^{11}$, PVD coated...
so long as the macroscopic body is large relative to microstructural features (e.g. fiber spacing, diameter, etc.) affecting the constitutive response.

The macroscopic constitutive response can be analyzed by viewing the porous composite as a cellular solid\(^{15}\) (i.e. a continuum made up of porous cells). As shown in Figure 8b, each cell is comprised of a segment of fiber and its contacts

![Diagram](image)

Figure 8. (a) 3-D architecture of misaligned fiber array. (b) Cellular solids unit cell used to analyze the densification and fiber bending response.

with three adjacent fibers which cross through the plane of the fiber segment. The length of the cell and the area of each fiber-fiber contact depend on the spatial distribution of fibers and the angle at which fibers cross over. Each plane section perpendicular to the applied stress (Figure 9) is made up of many such cells of varying compressive strength undergoing uniaxial compression. Since all plane sections have identical properties and undergo the same displacement, it is only necessary to consider one of these to determine the overall response. The (known) applied force \(F_a\) must be balanced by the sum of forces supported by the unit cells:

\[
F_a = \sum_{i=1}^{N} v_i F_i
\]  

(1)

where \(N\) is the total number of cells in the plane, \(v_i\) is the number fraction of \(i^{th}\) cells and \(F_i\) is the force carried by the \(i^{th}\) cell.
Figure 9. Macroscopically, each plane perpendicular to the applied stress is made up of unit cells all undergoing a uniform compressive strain.

The force needed to compress a given cell arises due to the resistance of the fiber segment within the cell to bending and of the contacts to inelastic deformation. The bending analysis is simplified by neglecting the contribution of the matrix coating to the stiffness (the stiffness of the fiber is, after all, much greater than that of the matrix at consolidation temperatures) and by assuming that the fiber remains elastic during consolidation. The force required to deflect a cylindrical, elastic beam loaded pointwise at its center is

\[ F = k\Delta \]  

(2)

where \( \Delta \) is the midspan deflection and \( k \) is the bend stiffness given by \( k = 3\pi E_f (d_f^4/l^3) \) where \( E_f \) and \( d_f \) are the fiber's Young's modulus and diameter. The length, \( l \), of fiber spanning the distance between two contacts decreases as the contacts deform and spread laterally so that the stiffness increases as the cell is compacted. Though not included here for the sake of brevity, this effect has been taken into account in the model.
The actual deformation occurring at the contact of two crossing metal coated fibers is quite complex. In the model, the contact is idealized as a rectangular region of matrix material of length, \( y_c(t) \) and height, \( z_c(t) \) undergoing plane strain deformation (the depth of the contact is just the fiber diameter and is assumed not to change). The matrix material is assumed to deform either superplastically or by dislocation creep, with the strain rate described in either case by a power-law expression:

\[
\dot{\varepsilon} = B_0 \exp \left[ \frac{-Q}{RT} \right] \frac{\sigma^n}{d^p}
\]

where \( \sigma \) is the stress acting on the contact, \( T \) is the process temperature, \( d \) is the grain size as a function of time, \( Q \) is the activation energy and \( B_0 \), \( n \) (stress exponent) and \( P \) (grain size dependence coefficient) are material constants. The evolution of grain size was described by an expression of the form, \( d = d_0 + kt^a \), where \( d_0 \) is the initial grain size, \( k \) the grain growth factor and \( a \) is the grain growth exponent.

By writing the uniaxial strain rate as \( \dot{\varepsilon} = \dot{z}_c/z_c \) and the contact stress as \( \sigma = F_c/a_c \), the force required to deform the contact at a given rate and temperature can be obtained. Since the height of the unit cell (Figure 8b) is \( h_i = 2z_c(t) + d_f - \Delta(t) \), the rate of change of cell height given an applied load and temperature is

\[
\dot{h}_i = 2\dot{z}_c + \dot{\Delta}
\]

with \( \dot{\Delta} \) and \( \dot{z}_c \) obtained from equations 2 and 3, respectively. The response of each unit cell, as given by equation 4, combined with the equilibrium equation (1) and the isostrain condition that all cells in a given plane section have the same deformed height (\( h_1 = h_2 = \ldots = h_i \)) are solved simultaneously to obtain the thickness of the removed plane section, i.e. \( h \). With \( h \) known, the density of each cell can be determined and the overall density obtained from a rule-of-mixtures based on the known number fraction of each cell.

The cells within a plane section vary in length (and therefore in stiffness) because of the irregularity in fiber alignment and spacing. If two parallel fibers (spaced apart by a distance \( s_q \)), cross a third fiber at an angle \( \varphi_p \) (Figure 9), then the length of fiber segment spanning the two crossing fibers is \( l = s_q/(\sin\varphi_p) \). If the distribution of crossover angles can be described by a probability density function, \( P(\varphi_p) \) and that of fiber spacing by \( P(s_q) \), then the probability of finding a cell whose length lies between \( l \) and \( l + dl \) is \( P(l)_{q,p} = P(s_q) \cdot P(\varphi_p) \). The PDF's, \( P(\varphi_p) \) and \( P(s_q) \), are both obtained from detailed metallographics analysis of the metallized fiber compact.
prior to consolidation. Knowing \( P(l) \) allows the number fraction of cells of a given size and the initial relative density of the cell to be determined.

With the deflection of each fiber segment known from the densification analysis, the stresses within the (elastic) fibers can be predicted (again using simple beam theory). If the peak tensile stress in the fiber (occurring at midspan opposite the applied force) is inserted into the probability function describing the distribution of fiber strengths, the probability the fiber will fail is obtained. The strength distribution of the SiC fibers considered here is well described by a Weibull function

\[
\Psi = 1 - \left\{ \exp \left[ -k \frac{l}{l_0} \left( \frac{\sigma_f}{\sigma_0} \right)^m \right] \right\}
\]  

(5)

where \( \Psi \) is the cumulative probability of failure \( (0 < \Psi \leq 1) \), \( k \) is the load factor relating the strength obtained in tensile tests to that obtained in bending, \( \sigma_0 \) is a reference strength obtained at a reference length, \( l_0 \), \( \sigma_f \) is the peak tensile stress in the fiber and \( m \) is the Weibull modulus. Summing the probabilities of failure for all cells at any time gives the number of fibers failed (per unit length of fiber).

Calculations were done using 12 unit cells of various lengths; the number fraction of each cell was chosen to reflect the experimentally observed distribution of fiber bend lengths. Ref [16] provides a detailed explanation of the procedure for determining the distribution of span lengths and its discrete approximation.

Figure 10 illustrates how fiber damage is predicted to depend on the processing temperature and the rate at which stress is applied; the damage, as given by \( \Phi \), the fraction of cells with broken fibers, is seen to increase with increasing stress rate and decreasing process temperature. At \( 750^\circ \text{C} \), it is no longer possible to avoid damage even for very low stress rates (i.e. less than 1 MPa/h). In contrast, at \( 900^\circ \text{C} \), the Ti-6Al-4V matrix creeps readily so that densification is achieved by matrix flow instead of fiber bending; as a result, higher stress rates may be applied without incurring damage. These results are in agreement with the observed influence of processing conditions as given in Tables 1 and 2, and Figs. 5-7. Comparing the results of experiment no. HIP-3 with those of HIP-10 illustrate the effect of stress rate (see Table 1): rapid loading (> 100 MPa/h) to 100 MPa followed by a 2-hour hold resulted in complete densification,

but substantial fiber damage. Ramping slowly to the same peak pressure in a two-step ramp (0-10 MPa at 5 MPa/h followed by 10-100 MPa at 45 MPa/h) allowed the same density to be reached, but with practically no damage to the fibers. Both samples were consolidated at \( 900^\circ \text{C} \).
5. CONCLUDING REMARKS

Fiber damage during consolidation can be a severe problem when reinforcing metal matrices with continuous ceramic fiber since slightly misaligned fibers are prone to break in bending. Model experiments have been conducted to search the pressure, time, temperature cycles that will eliminate fiber breaks and to provide an experimental basis for predictive models.

Qualitative trends in fiber damage as a function of packing geometry, and hot pressing condition, (time, temperature and pressure) are adequately predicted by a process model that incorporates packing geometry, the creep properties of the matrix and the statistics of fiber strength. The experiment and model indicate that fiber damage can be suppressed by consolidation below a threshold pressurization rate that is a function of matrix properties, statistics of fiber strength, packing geometry and relative packing density. This threshold identifies the regime where matrix creeping is the main deformation mechanism as opposed to elastic bending of the reinforcing fiber. Knowledge of this threshold is important to minimize consolidation cycle times, for economic and thermodynamic reasons, while avoiding fiber failure.

Another important aspect is the identification of a critical consolidation temperature, below which fiber damage is not avoidable (i.e. 750°C in our example). The upper bound (950°C in our example) is determined by the kinetics of interface reactions.

Figure 10. Predicted cumulative fiber damage (at D = 0.75) as a function of process temperature and rate of applied pressure.
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