STRESS RUPTURE OF UNIDIRECTIONAL POLYMER MATRIX COMPOSITES IN BENDING AT ELEVATED TEMPERATURES

by

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(ABSTRACT)

A new method for stress-rupture experiments in bending has been developed and used to characterize unidirectional polymer matrix composites. The method, which makes use of very simple fixtures, led to coherent results. These results have been modeled using the large deflection of buckled bars theory (elastica) and it is possible to predict with good accuracy the strain at each point of the specimen if the end-to-end distance is known. The failure process has been experimentally characterized. The formation and propagation of microbuckles leads to a compressive failure. Based on the elastica and the classical lamination theory, a model for the distribution of the Young’s modulus along the length of the specimen has been established. Three different micromechanical models have been applied to analyze the time-to-failure versus strain behavior at two temperatures - one below and one above the glass transition. The first micromechanical model considers the nucleation of the microbuckles as the main cause of failure. In addition, the stiffness and stress distributions at any time before failure are calculated based upon the rotation of the fibers in the damaged region. The second and
last models, respectively based upon a Paris Law and energy considerations relate the
time-to-failure to the propagation of the main microbuckle. For this last model, a good
correlation between experimental and theoretical data has been obtained. Finally the
influence of the temperature on these models has been studied.
DEDICATION

This work is dedicated to my parents: Francis and Guitty Mahieux.
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1 INTRODUCTION AND LITERATURE REVIEW

1.1 Introduction

Engineering composite materials can be subjected to bending situations during manufacturing or service. Composite materials are sometimes stocked in rolled tapes. As a result, it is important to determine the influence of the environment on the mechanical properties of the material over long periods of storage time. During service, this need becomes even greater. The case of composite materials used to reinforce flexible pipes is representative of this need. Indeed, the composite used in a pipe-type geometry is subjected to a quasi-static bending load and may be subjected to various dynamic loads. If this pipe is used in an aggressive environment (wet, salt water, elevated temperature...), the combination of the environmental effects and the bending loads can be dramatic.

The rupture, after a period of time, of a material subjected to a static load is regarded as “stress rupture”. The stress rupture of many homogeneous materials has been studied in great detail. Surprisingly, a very small number of studies deal with the case of polymer matrix composites. Even though the mechanical behavior of a composite material in the fiber direction is “fiber controlled”, we would intuitively expect the presence of a viscoelastic matrix to contribute to the global behavior of the composite.
Consequently, the behavior of unidirectional polymer matrix composites and the possibility that these materials can undergo stress rupture under a quasi-static load must be explored.

As previously mentioned, fiber direction stress rupture in bending for polymer matrix composites has not been directly studied, and the literature concerning these issues is very sparse. The first part of the present literature review deals with bending of beams and the theory of elastica (large deflections of buckled bars [Ref. 1]). Then compressive models for polymer matrix composites and viscoelastic models for microbuckling are presented. Alternative models are also introduced to characterize the damage accumulation in composites under quasi-static loads. Examples of pre-existing models for ceramic matrix composites are summarized. The last part of this literature review deals with the different attempts to describe the stress-rupture of polymer matrix composites and the influence of temperature.

1.2 Literature review

1.2.a Bending

The case of large deflections for beams has been analyzed in the elastica theory [Ref. 1, Ref. 2, Ref. 3]. A bar is considered to be fixed at one end and a force is applied as shown in Figure 1.
Fig. 1 Large deflection bending of a beam.

In the Timoshenko and Gear analysis [Ref. 1], the exact value for the curvature is used. Starting from the exact differential equation of the deflection curve, relations between $dy, ds$ (the curvilinear abscissa) and the curvature are derived. To obtain these relationships, complete elliptic integrals of the first and second kind are used. Equations are given to compute the maximum deflection of the specimen ($Y_a$) and the new end-to-end distance for the buckled bar ($X_a$) as a function of the deflection angle $\alpha$. It is also discussed that this analysis can be used in the case of a bar with hinged ends.

Fukuda et al. [Ref. 4] describe a new bending test method (compression bending) to obtain the bending characteristics of composites. Equations for the radius of curvature at the midpoint, the midspan deflection and the relative displacement of the ends of the specimen are derived based on the previous analysis. Experimental values are obtained
by a strain gauge method. Deflection versus end-displacement curves are used to compare experimental and theoretical data. A good correlation exists between experimental and theoretical results.

Lifshitz [Ref. 5] briefly deals with the possibility of applying a time-dependent model to a flexural composite system. He underlines the existence of both tensile and compressive contributions to the failure of the composite under quasi-static bend loading conditions, and the need for a compression model.

1.2.b Compression

Several models to characterize the compressive strength of unidirectional composites have been developed [Ref. 6, Ref. 7, Ref. 8]. The criteria are based on the constitutive properties of the fibers and matrix, and are time independent. Lo et al. [Ref. 8] derive a time-independent expression for the Young’s modulus in a microbuckled region.

A time independent model together with a Weibull analysis are used by Jelf and Fleck [Ref. 9] to study the failure of AS4 carbon fiber/PEEK composite under a compressive stress. Jelf and Fleck also summarize and characterize the different failure mechanisms for unidirectional composites under compressive loads. The four mechanisms are:

- elastic microbuckling (for composites with a linear elastic behavior of the matrix)
-fiber crushing (fiber failure by longitudinal splitting, plastic yielding, kinking within the fibers or plastic buckling of the microstructural units within each fiber)
-matrix failure (brittle crack propagation in the matrix material)
-plastic microbuckling.

This last failure mechanism is thought to be the most important type of failure for polymer matrix composites [Ref. 9, Ref. 11], but experimental verification of this claim has not been achieved.

1.2.c Viscoelastic microbuckling

Microbuckling in unidirectional fiber composites leads to the formation of a kink band (Figure 2).

![Fig. 2 Kink band.](image)

Steif [Ref. 10] derives a model for kink band formation based on energy considerations which assumes that the kink band is in mechanical equilibrium with the unkinked material. Typical values for carbon-reinforced composites are used to compute the characteristic angles $\alpha$ and $\beta$ of the kink band. The angle $\alpha$ is found to have a critical
value above which kinking is favored. The angle $\beta$ seems to be in a $15^\circ$-$30^\circ$ range of values [Ref. 10, Ref. 11] and $\alpha$ is slightly in excess of $2\beta$. Steif's analysis ignores the possibility of the initiation of the kink band at a free edge. Yang et al. [Ref. 12] analyze the elastic-plastic surface instability problem and define a critical compressive stress for the horizontal damage band as a function of characteristic shear moduli. This analysis predicts the formation of compressive damage bands near the free surface. Budiansky and Argon [Ref. 11, Ref. 17] identify the shear yield stress of the matrix material and the initial misalignment angle of the fibers as the preponderant parameters in the behavior of the kink band.

These models do not consider the time factor as influencing the development of the kink band. Schapery [Ref. 13] raises the problem of the time-dependent behavior of the material in the case of high temperatures or long loading times. Simple shear deformation and bending deformation models are used to provide a better understanding of the viscoelastic behavior. In the case of constant loading, the correlation between analytical and numerical results is very good. But these models are idealizations and the possibility of the presence of microbuckles is not considered.

There were other attempts to model the time-dependent behavior of the buckled region [Ref. 14, Ref. 15]. These analyses assume a linear viscoelastic behavior of the material. Slaugther and Fleck [Ref. 14] model the microbuckled region as a three parameter solid (or standard linear viscoelastic model) and a logarithmically creeping
material. The problem that arises from this analysis is the need for four non-dimensional material constants. No experimental or analytical method is given to obtain these material constants. For their computations, the authors assume plausible values. The theoretical results are not compared to experimental data.

Budiansky and Fleck [Ref. 15] summarize the different studies on compressive bending, starting from the critical kinking stress $\sigma_c = G$ ($G$, effective longitudinal shear modulus of the composite) suggested by Rosen [Ref. 16] that overestimates the compressive strength by a factor of 3 or 4. The consideration of plasticity (yield strength) and misalignment of the fibers as the preponderant factors in kinking was introduced by Argon [Ref. 17] in a simplified expression for the kinking stress $\sigma_c = \frac{\tau}{\bar{\phi}}$, where $\tau$ is the shearing yield stress and $\bar{\phi}$ is the initial maximum angular misalignment of the fibers. Budiansky and Fleck [Ref. 15] also present the viscoelastic and creep kinking model from Slaughter and Fleck [Ref. 18]. A relationship between time, the applied stress, and the fiber angle is given. Assuming that the creep kinking lifetime is reached for an infinite angle of misalignment, the lifetime can be calculated. The solution depends on the determination of values like the initial misalignment, the reference shear stress and the reference value of creep rate produced by the reference shear stress. In their computation, Budiansky and Fleck assume “plausible” values.
1.2.d Alternative time-dependent models for damage accumulation

An alternative approach to the stress rupture problem that can be found in the literature is the simulation of damage accumulation [Ref. 19, Ref. 20]. Bolotin [Ref. 19] derives a unified model of the breakdown of composites under a tensile load, based on the assumption that the process is a combination of single breaks of the fibers and defect formation in the matrix. The accumulation of damage by both processes is a function of time. At a given time (final breakdown), the density of defects reaches a critical level. Tsykalo [Ref. 20] uses this model to predict the damage accumulation in unidirectional composites under long-term static loading. The fibers are assumed to have a linear elastic behavior and the matrix to be perfectly elastoplastic. Long-term strength values are plotted as a function of time, but they are not compared to experimental data. Though the shape of the strength versus time curve seems to correspond to the one expected, several problems arise from this analysis. Some parameters are difficult to relate to the material properties; this is probably why the results are not compared to experimental values. The model is based on statistical failure and the viscoelastic characteristics of the matrix are ignored. Finally, this model is based on tensile experiments and seems to be difficult to extend to the bending case where the failure process involves compression.

1.2.e Stress rupture and creep of ceramic matrix composites

Stress rupture of metal matrix composites and ceramic matrix composites has been widely and successfully studied [Ref. 22, Ref. 23]. The behavior of metal matrix composites is too different from the behavior of polymer matrix composites to be
considered here. However, we may learn a great deal by studying the work which has been done in the field of ceramic matrix composites. Chermant et al. [Ref. 22] study the creep of ceramic fiber reinforced ceramic matrix composites. Creep mechanisms for different ceramic matrix composites are brought out and summarized: the fibers contribute to the creep of the composite by microcracking of the fibers and fiber creep. Fiber fracture and its effects in continuous fiber ceramic composites have been studied in detail [Ref. 21]. The matrix contributes to the creep of the composite in several ways: microcracking, elastic creep, grain boundary sliding of the crystals, cavitation in the matrix... The fiber matrix interface also plays a very important role in the creep of the composite, by the fiber-matrix decohesion due to the shear stress. Chermant et al. plot the stress and creep rates as a function of time for these different components. Equations for steady states are established for each process. Several composies are studied and the conclusion is developed that the creep behavior is dominated by the interface and the fiber creep, but no model is found to fit the experimental data.

Chuang et al. [Ref. 23] suggest a model for the creep rupture of a metal-matrix particulate composite. The specimens have been subjected to both tensile and flexural tests at high temperature. The tested specimens show a different behavior under tensile and compressive loading so the authors use a model previously established [Ref. 24] for asymmetric creep materials: the fibers are considered as a Maxwell element and “time-dependent stress and strain distribution are solved for a given bending moment”. A redistribution of the local bending stresses from a linear (elastic) to a final non-linear state
(quasi-steady state creep) occurs defining a material time constant. The rupture process is assumed to occur from the tensile side where a major crack will grow to the center. Based on these considerations, a relationship is established between the critical crack length at rupture and the outer fiber tensile strain. The time-to-failure is numerically computed and the applied stress is plotted as a function of the rupture time on a semi-logarithmic scale. A very good agreement between experimental and theoretical data can be observed. The applied stress and the logarithm of the rupture time are linearly related. These approaches pursue the same goal, i.e. the determination of the rupture time as a function of the applied load for stress rupture of the material under quasi-static bending load. These approaches have been fairly successful for ceramic and metal matrix composites. But these models can not be applied directly to polymer matrix composites as the main component of the creep behavior of the composite is generally the viscoelastic character of the matrix. The damage growth process seems also to be fundamentally different.

1.2f Stress rupture and creep of polymer matrix composites

Very few papers deal with stress rupture of polymer matrix composites and the suggested models are not as successful as in the case of ceramic matrix composites when compared to experimental data.

Gates [Ref. 25] models the behavior of polymer matrix composites by an elastic/viscoplastic constitutive model. By considering uniaxial loading of an off-axis
laminate, the load-strain relationships are established for four cases (quasistatic $\sigma_x = \sigma_x^*$, loading $\dot{\varepsilon}_x > 0$, stress relaxation $\dot{\varepsilon}_x = 0$ and creep $\dot{\sigma}_x = 0, \dot{\varepsilon}_x > 0$) under tension loading. The equations are derived assuming a quasi-static plastic strain-stress relation: $\varepsilon = A \sigma^n$. A problem is the need of the material constants A and n that are found by fitting "the power law to the effective stress, effective plastic strain data". Like the case of ceramic matrix composites, master curves for tension and compression of polymer matrix composites differ, which seems to indicate that the deformation mechanisms are different for tension and compression.

Christiensen [Ref. 27] derives a model still based on the viscoelastic creep of the material (the creep function is taken in the power law form) but he adds the concept of a crack generated surface layer. He makes a simplifying assumption that a unique initial flaw is present in the material. A kinetic criterion to describe the crack growth under given conditions of load and temperature is "derived from an application of the global balance-of-energy law". Time and stress are found to be linearly related on a log-log scale. This model was statistically generalized by assuming a Weibull distribution for the strength. But the analysis contains several limiting assumptions such as the use of a power-law creep function and the assumption that the crack and free edge do not interact.

The curve fitting problem when assuming a power-law behavior for the creep function is raised by Raghavan et al. [Ref. 26]. The authors suggest a model involving
the material properties and temperature. The model used to predict the creep behavior of the composite is a three parameter solid. The rupture criterion is given by the critical energy that is dependent on temperature and strain rate. The applied stress versus time to failure curve is plotted. But the shape of the calculated curve does not compare well to the shape of the experimental curve.

An alternative approach to the stress rupture of polymeric matrix composites has been derived by Lifshitz [Ref. 5] based on the concept of the fibers’ ineffective length: the local shear stresses in the matrix created by the fiber breaks induce a relaxation of the matrix. So a part of the fiber becomes ineffective as a load carrying agent. The stress distribution around the fiber near the broken end will change as a function of time due to the viscoelastic character of the matrix (the stress decreases as a function of time), which leads to an increase in the ineffective length. An expression for the time-dependent ineffective length is derived and is found to be proportional to the square root of the matrix creep compliance. The contribution of broken and unbroken fibers are used to compute the stress at a given time. The time when this stress reaches the “most probable elastic failure stress” defines the time-to-failure. The calculated values of time to failure overestimate the real values for two main reasons: the stress concentrations have not been considered and a single mode of failure has been assumed.

1.2.g The influence of temperature in the stress rupture of polymer matrix composite and the possibility of a time-temperature equivalence
The fracture strength of polymer composites and pure polymer (epoxy) materials has been shown to behave in a similar way [Ref. 26]. Even at a temperature well below the glass transition of the matrix, the composites show a significant degradation of strength and modulus as a function of time. According to Christensen’s [Ref. 27] model, when the temperature changes, the time-to-fracture versus applied stress curve obeys a horizontal shifting for the materials which allows a time temperature superposition. For materials that do not follow such a rule, the shape of the time-to-fracture curve will change as a function of temperature. A master curve of flexural static strength has been obtained for satin woven CFRP laminates [Ref. 28]. Using the static master curve and a linear cumulative damage law, the creep strength of the composite can be predicted. The experimental results show that the flexural strength remains time-dependent even far below the glass transition temperature. These results give us an indication that a time-temperature equivalence could be established for polymeric matrix composites, but these latter experiments were run on woven laminates and the fracture process for unidirectional fiber reinforced composite probably differs.

1.3 Goal of this research

The goal of this research is to study the bend-stress rupture of unidirectional polymer matrix composites. The influence of the temperature and the environment is more important in the case of bending than in the case of tension. Bending leads to a non-linear geometry. Therefore, the first step in the analysis will be to describe the geometry
of the bent specimen. As a result of the analysis, it will be possible to bend at controlled initial strain levels. Experiments will be performed to validate the fact that unidirectional polymer matrix composites undergo stress rupture as intuitively predicted.

The next step to this analysis is to derive a model for the viscoelastic behavior of unidirectional polymer matrix composites under bending at elevated temperature. Specifically, the experimental observations and theoretical treatments will require the study of the following points:

- characterization of the failure mode,

- analysis of the strain distribution along the length of the specimen,

- computation of the Young’s modulus and stress distribution along the length of the specimen.

- modeling of the compressive failure process.

This last point includes the study of the nucleation and propagation of the microbuckles. Ultimately these models will allow the computation of the life of the material under a given bending load.
2 EXPERIMENTAL CHARACTERIZATION

2.1 The material

The material used for the experimental work was polymer matrix/unidirectional continuous carbon fiber reinforced composite: Polyphenylene sulfide (PPS) based matrix/AS4 fibers. The matrix is a semi-crystalline thermoplastic. This material has been provided by Wellstream Inc. and very little information could be obtained about the material composition and properties. Table 1 summarizes some important mechanical characteristics of the material at room temperature [Ref. 33].

<table>
<thead>
<tr>
<th></th>
<th>% Fiber by Weight</th>
<th>Tensile strength (Ksi)</th>
<th>Tensile Modulus (Msi)</th>
<th>Strain to Failure (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average</td>
<td>51.35</td>
<td>186.1</td>
<td>14.3</td>
<td>1.41</td>
</tr>
<tr>
<td>Standard deviation</td>
<td>2.409</td>
<td>18.2</td>
<td>0.80</td>
<td>0.033</td>
</tr>
</tbody>
</table>

Table 2 gives some information on the material characteristics at elevated temperatures.

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Modulus (Msi)</th>
<th>Strength (Ksi)</th>
<th>Strain to Failure (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>90°C</td>
<td>13.6±.13</td>
<td>188±8.4</td>
<td>1.46±0.061</td>
</tr>
<tr>
<td>120°C</td>
<td>13.2±36</td>
<td>174±7.8</td>
<td>1.37±0.081</td>
</tr>
</tbody>
</table>
The temperatures of 90°C and 120°C where chosen based on the properties of the matrix: one temperature is around the usual Tg (89°C) given for PPS in the literature [Ref. 32] and one is above. To confirm these choices a Dynamic Mechanical Analysis (DMA) was performed on the composite in bending at fixed frequency 1Hz, 35-175°C, heating rate 1°C/min. The result is shown in Figure 3.

Fig. 3 DMA of AS4/PPS.
In Figure 3, based on the storage modulus versus temperature curve, the beginning of the glass transition is at 90°C. At the second test temperature, 120°C, the modulus of the matrix has already dropped a large amount. The maximum value for \( \tan \delta \) occurs at 130°C. This discrepancy between the glass transition temperature given in the literature and the one indicated by the DMA can be explained by the presence of other constituents than PPS present in the matrix. For complementary information the result of a differential scanning calorimetry is appended.

The material is supplied in a rolled tape. As a result the width and the thickness of the specimens are determined by the manufacturing process and are summarized in table 3. For the length, 6 inches will be arbitrarily chosen.

<table>
<thead>
<tr>
<th>Table 3. Geometrical properties of the specimens.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Width (mm)</td>
</tr>
<tr>
<td>------------</td>
</tr>
<tr>
<td>Average</td>
</tr>
<tr>
<td>Standard deviation</td>
</tr>
</tbody>
</table>
2.2 Preliminary experiments

Stress rupture of these materials under quasi-static bending load has never been experimentally observed. To confirm the fact that stress rupture will occur, some preliminary experiments were conducted. The specimens were bent manually around pipes of different diameters. During these manipulations, one could hear the specimens crack and pop due to fiber breakage, even for specimens that did not break. A minimum radius of non rupture was determined in this way. For this smaller diameter (3 inches), the specimen was attached around the pipe by a wire fastened to the two ends of the specimen. The specimen bent around the pipe was put in the oven at 90°C and a moment later, the specimen was observed to be broken. Though this experiment was not accurate or reproducible, this was encouraging in showing that this material undergoes stress rupture. Another important observation was that, for most of the specimens that were bent around the pipes, thin lines initiating at the edge of the specimen and propagating across the width of the specimen (perpendicular to the fibers direction) could be observed. These lines could be seen on the compressive side of the specimen and were located on only one of the two edges of the specimen, as shown in Figure 4.
Fig. 4 Example of damage locations on a specimen.

Several questions arose at this point:

- Are these lines (that we could see clearly with the eyes) microbuckles?

- If these lines located on the compressive side are microbuckles, then does the rupture process occur by a compressive mode of failure?

- Is the fact that these microbuckles are located only on one side of the specimen due to a non-axial loading (manual bending)?
2.3 Design of the fixture

The fixture used for the bending experiments was developed in support of the work reported by Blair Russell, in Reference 31. Several features and capabilities of the fixture are important. The fixture was adapted to the test of 6 inch specimens. First of all, it should allow a good axial loading. Also, hundreds of specimens had to be tested, so the bending process had to be fast and the fixture had to be small in order to fit a large number of specimens in the oven. Initially, two adjustable fixtures were designed. These fixtures were used to establish an end-to-end distance versus strain relationship. After this relationship was established, it was possible to create a fixture with fixed ends. These different fixtures are shown on Figure 5 (from Ref. 31).

![Fig. 5 Fixtures.](image-url)
For the fixed-end fixture, one end of the specimen is put in corner A. The bending is done progressively by sliding the other end against the fixture until its end reaches corner B. The length of the fixture corresponds to a given maximum strain at the center of the specimen. This kind of fixture allows a systematic study with a minimum amount of material.

2.4 First step of the analysis and limits of the fixture

At this point, the first theoretical treatment was needed. Based on the theory of the elastica [Ref.1] a relationship between the end-to-end distance of the bent specimen and the strain at the center of the specimen (point of maximum strain) can be derived. Figure 6 specifies the notations used in this analysis.

![Fig. 6 Geometry of the bent specimen.](image)

For the symmetrical case of a bent specimen at both ends the relationship between the total end-to-end distance $X$ and the angle $\alpha$ is given by equation (1) from Reference 2, assuming that the bending modulus is constant:
\[ X = 2L \times \left( 2 \frac{E(\alpha)}{K(\alpha)} - 1 \right) \]  

(1)

Where

\[ K(\alpha) = \int_0^\frac{\pi}{2} \frac{1}{\sqrt{1 - \sin^2(\frac{\alpha}{2}) \cdot \sin^2(\Phi)}} d\Phi \]  

(2)

is the complete elliptic integral of the first kind.

\[ E(\alpha) = \int_0^\frac{\pi}{2} \sqrt{1 - \sin^2(\frac{\alpha}{2}) \cdot \sin^2(\Phi)}} d\Phi \]  

(3)

is the complete elliptic integral of the second kind and \( 2L \) is the length of the specimen.

Table 4 has been obtained by varying the angle \( \alpha \) and shows tabulated corresponding values between the end-to-end distance and the angle at both ends of the specimen for a specimen of total length \( 2L \) of 6 inches.

**Table 4. Corresponding deflection angles and end-to-end distances.**

<table>
<thead>
<tr>
<th>( X ) (in)</th>
<th>6.000</th>
<th>5.954</th>
<th>5.818</th>
<th>5.595</th>
<th>5.287</th>
<th>4.902</th>
<th>4.446</th>
<th>3.928</th>
<th>3.356</th>
<th>2.742</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \alpha ) (deg)</td>
<td>0</td>
<td>10</td>
<td>20</td>
<td>30</td>
<td>40</td>
<td>50</td>
<td>60</td>
<td>70</td>
<td>80</td>
<td>90</td>
</tr>
</tbody>
</table>

For each value of \( X \), it is possible to obtain the radius of curvature at the midspan:

\[ \rho_\lambda = \frac{L}{2 \cdot p \cdot K(\alpha)} \]  

(4)

where

\[ p = \sin \frac{\alpha}{2} \]  

(5)

It is now possible to compute the strain \( \varepsilon \) at the center of the specimen. Assuming that the neutral axis is at the mid-thickness of the specimen (\( \varepsilon = 0 \) at \( t/2 \)) we get
\[ \varepsilon = \frac{t}{2 \cdot \rho_A} \]  \hspace{1cm} (6)

When damage occurs the neutral axis will be locally shifted.

Experiments were run and specimen were bent. Strain gauges were located at the middle of each specimen. Values of the end-to-end distances and the corresponding strain values were recorded. The following plot (Figure 7) shows the experimental and theoretical values calculated according to the previous equations. The strain is normalized by dividing it by the tensile failure strain.

![Graph showing strain vs. end-to-end distance](image)

**Fig. 7 maximum strain (center of the specimen) versus end-to-end distance.**

Calculated values and measured data seem to correspond quite well, the average error is 6\%. The calculated strains seem to overpredict the measured values. This can be explained in several ways:
-the maximum strain occurs at the center of the specimen. The strain gauge is not a point measurement but averages the strain on its surface. So a lower value than the maximum strain is actually measured.

-The previous equations assume a constant and uniform bending modulus. It is possible that the compressive and tensile bending moduli differ slightly, so that the ε=0 at t/2 assumption is not exact anymore.

But the correlation is good enough, and a 6% error seems difficult to improve upon.

By using this kind of fixture, some difficulties arise for the analysis: only the ends are fixed and when put in the oven the shape of the specimen will not be constrained to a given position. As a result, these experiments will not occur at constant strain nor constant stress. Therefore, it will not be possible to directly apply a creep or relaxation model. Moreover, the bending configuration will lead to a variation of the strain (and as a result, the stress) along the specimen. Consequently, this problem has to be modeled by a non-linear behavioral model applied on a non-linear geometry.

2.5 SEM observations

To further understand the lines which were observed to emanate from the edges of the specimens during the preliminary rupture tests, new adjustable fixtures were used to bend a specimen. The specimen was first bent at low strain. Then the ends were released and the specimen was observed in a scanning electron microscope. No significant
damage could be seen on the tensile side. In contrast, broken fibers near the edges of the specimen could be observed on the compression side. Then the specimen was reloaded, this time up to failure. The two parts of the specimen were examined in the scanning electron microscope. Figures 8 and 9 show the fracture location on the tensile and compressive side. Figures 10 and 11 were taken on the compressive side. Microbuckles are clearly present near the edge of the specimen. These observations seemed to confirm the fact that microbuckles and compressive damage processes play an important role in the failure processes.
Fig. 8 SEM picture of the fracture on the tensile side.
Fig. 9 SEM picture of the fracture on the compression side.
Fig. 10 SEM picture of the microbuckles on the compression side.
Fig. 11 SEM picture of the microbuckles on the compression side.
2.6 Stress rupture experiments

2.6.a Bending stress experiments at 90°C and 120°C

Next, systematic bending stress rupture experiments were conducted. The specimens were strain gauged and bent at different strain levels. The specimens were placed in the oven at 120°C. High strain levels were chosen first, as the time to failure was unknown and the fastest time-to-failure was preferred. The specimens bent at about 90% of their ultimate strains broke in a few seconds. Specimens bent at lower strain levels were put in the oven at the same temperature. It took them more time to break, up to runout experiments (unfilled squares in Figure 12) : the specimen had to be taken out of the oven after a long time, 10 hours to a couple of days, because the fixtures were needed for other experiments. The same experiments were run at 90°C. For the same strain level, it takes more time for the specimens to break at 90°C than 120°C, as expected. The results are summarized in Figure 12 and Figure 13 (the normalized strain corresponds to the experimental strain over the ultimate tensile strain-to-failure at room temperature).
Fig. 12 Time-to-failure versus normalized maximum strain at 90°C.

Fig. 13 Time-to-failure versus normalized maximum strain at 120°C.
As we can see on the previous plots, time-to-failure and strain level seem to be related. The data show very little scatter and the obtained curves look very coherent.

At high strain levels, when fracture occurs, the two pieces of the specimen fly apart. In contrast, at lower strain levels the two pieces of the specimen don’t separate. For this case, specimen failure was defined to occur when one microbuckle (usually close to the center of the specimen) propagates across the whole width of the specimen, leading to a dramatic change in the shape of the specimen.

2.6.b Thermocouple measurements

Another important fact was that the specimens bent at high strain levels broke in a very short time. They probably did not have the time to reach the temperature of the oven. To get some details on this point, a thermocouple was put on a specimen before being put in the oven. An insulating tape was used to attach the strain gauge in order to get a temperature record close to the temperature of the specimen (not the oven). The temperature was recorded every 5 seconds from the moment when the specimen was put in the oven until stabilization of the temperature values. As intuitively predicted, a ramp of temperature can be observed and the maximum temperature is not reached instantaneously by the specimen. But some problems arose at this point: the thermocouple when put alone in the oven takes also some time to reach the maximum value of the temperature. Moreover it never reaches the value of the oven temperature. The following plots (Figures 14 and 15) summarize these results.
Fig. 14 Temperature recorded by the thermocouple alone and the thermocouple on the specimen as a function of time when put in the oven at 90°C.

Fig. 15 Temperature recorded by the thermocouple alone and the thermocouple on the specimen as a function of time when put in the oven at 120°C.
These data then seem very difficult to interpret, but we have the certitude that at high strain levels, the specimens break before reaching the oven temperature.

**2.6.c Videotape**

Three specimens were videotaped in the oven at 90°C, in front of a grid. The first and last frame before failure were printed. By photocopying these pictures on slides and superimposing the pictures, the following observations have been made (see appendix). Three specimens were tested. The first one was strain gauged and bent at 80% of the ultimate strain to failure. The second one was not strain gauged. Using the end-to-end measurement and the previous analysis, it was also bent at 80% strain to failure. The last specimen was not strain gauged. It was bent at 65% strain to failure. For the first and last specimens a change of shape between the first and the last frame could be observed. The curvature of these two specimens became flatter near the ends and sharper at the center. It is also noticeable that the strain gauge on the first specimen seems to delay the fracture a small amount. All the specimens that were strain gauged for the other experiments fractured at one end or the other of the strain gauge. The adhesive used to adhere the strain gauge to the specimen seems to slightly reinforce the composite.

For the second specimen, no change of shape occurred until fracture. It could be observed that the first and last specimens exhibited the presence of microbuckles while the second didn’t. So, the microbuckles seem to be responsible for a change in the curvature of the specimen at each point, that modifies the local stress concentration.
2.6.4 Microbuckles

Since the microbuckles seemed to play a very important role in the failure process, a better understanding of the phenomenon was necessary. Where, when, and why does microbuckling occur, and how do the microbuckles propagate were the main questions.

To have a better understanding of how the microbuckles nucleate and propagate, the underneath of the specimen was videotaped while in the oven. The specimen was bent at 50% strain-to-failure and placed in the oven at 120°C. After one minute in the oven, the first microbuckles initiated. The first microbuckle appeared on one edge but was not located at the middle of the specimen (highest stress level). Then these first microbuckles stopped propagating and other microbuckles appeared on both edges of the specimen. During the last second, one of the microbuckles, located at a point of higher stress level (middle of the specimen) grew slowly, stopped for a short moment, then propagated very rapidly through the whole width of the specimen inducing fracture.

The broken specimens always showed the presence of more microbuckles on one edge than on the other, although now the axial loading process is better than in the preliminary experiments. The microbuckles were unequally spaced. It seems that (unlike matrix cracks in these materials) there are no interactions between the microbuckles. However, the microbuckles are more concentrated at the middle of the specimen. But the microbuckles closest to the center of the specimen (highest stress) are not really much longer than further from the midpoint. An attempt was made to identify a trend in the
formation of the microbuckles by mapping the position of microbuckles on several specimens bent at several strain levels. As shown in the next figures (Figures 16 and 17) no real trend can be identified.

The first attempt to identify a trend was made by plotting the average spacing between microbuckles on one of the edges as a function of the total number of microbuckles on this same edge. The result doesn’t seem to follow a trend (Figure 16).

![Graph showing average interspace between microbuckles as a function of the number of microbuckles on one edge.](image)

**Fig. 16** Average interspace between microbuckles as a function of the number of microbuckles on one edge.

Another attempt was made to plot the average spacing between microbuckles as a function of the strain level. But as we can see in Figure 17, no trend can be identified here, either.
Fig. 17 Average interspace between microbuckles as a function of maximum strain.

More encouraging data can be plotted. Indeed if we plot the total number of microbuckles versus the time-to-failure, we can observe a more regular increase (quasi linear on a logarithmic scale, Figure 18). The same trend can be observed at 90°C.

Fig. 18 Number of microbuckles versus time-to-failure at 120°C.
From this it can be concluded that microbuckles don’t seem to interact and the spacing between them doesn’t seem to be driven by any external parameter. However, the number of microbuckles seems to be related to the time to failure of the specimen, which in turn, is determined by the quasi-static applied strain level.
3 ANALYSIS

All of these observations had to be explained, and if possible, modelled and generalized.

3.1 Determination of the equations of shape for the bent specimen

The following picture (Figure 19) and equations describe what was stated in part 2.4.

For the symmetrical case of a bent specimen at both ends, the relationship between the total end-to-end distance $X$ and the angle $\alpha$ is:

$$ X = 2L \times \left( 2 \frac{E(\alpha)}{K(\alpha)} - 1 \right) $$

(1)
Where

$$K(\alpha) = \int_{0}^{\pi/2} \frac{1}{\sqrt{1 - \sin(\frac{\alpha}{2})^2 \sin(\Phi)^2}} d\Phi$$  \hspace{1cm} (2)$$

is the complete elliptic integral of the first kind,

$$E(\alpha) = \int_{0}^{\pi/2} \sqrt{1 - \sin(\frac{\alpha}{2})^2 \sin(\Phi)^2} d\Phi$$  \hspace{1cm} (3)$$

is the complete elliptic integral of the second kind and $2L$ is the length of the specimen.

On the previous case, we only dealt with the point at the centre of the specimen. We will now obtain the strain of the material as a function of the different coordinates $x$, $y$ and $s$, the curvilinear abscissa.

For a given angle $\alpha$, we want to get the correspondence between the different coordinates at each point of the bent specimen. For the following computations, we will now consider one half of the specimen as shown in Figure 20.

![Fig. 20 Bending of the half specimen.](image)
The y coordinate varies from 0 to the maximum deflection at the opposite end of the specimen. This value can be easily derived from Ref. 1:

\[ y_a = \frac{2 \cdot \sin(\frac{\alpha}{2}) \cdot L}{K(\alpha)} \quad (7) \]

Timoshenko and Gere start from the exact differential equation \( EI \frac{d\theta}{ds} = -P_y \). Knowing that the radius of curvature can be written as \( \rho = -\frac{ds}{d\theta} \) and after some mathematical manipulations, we obtain the following expression for the exact curvature radius:

\[ \rho = \frac{EI}{P_y} \]

\[ k^2 = \frac{P}{EI} \]

then

\[ \rho = \frac{1}{k^2 y} \]

\[ k = \frac{K(\alpha)}{L} \quad (8) \]

Finally

\[ \rho(y) = \frac{L^2}{K(\alpha)^2 \cdot y} \quad (9) \]

This assumes that the bending modulus is constant through the whole specimen.

\( \theta \) is the angle defined by the tangent to the bent specimen at one point and the vertical.

This angle can be computed for each value of \( y \):

\[ \theta = \arccos \left( \frac{K(\alpha)^2 \cdot y^2}{2 \cdot L^2} + \cos(\alpha) \right) \quad (10) \]
\( ds \) can be expressed as a function of the \( \alpha \) and \( \theta \) angles by
\[
 ds = \frac{1}{k \cdot \sqrt{2(\cos \theta - \cos \alpha)}} \, d\theta
\]
(11).

Assuming that we can approximate \( dx \) by \( \cos \theta \cdot ds \) and \( dy \) by \( \sin \theta \cdot ds \), it is now possible to establish the equations to compute \( x \) and \( s \), the curvilinear abscissa for a given \( y \):
\[
x = Xa + \frac{1}{\sqrt{2 \cdot k}} \int_0^\theta \frac{-\cos(\theta)}{\sqrt{\cos(\theta) - \cos(\alpha)}} \, d\theta
\]
(12)
\[
s = L + \frac{1}{\sqrt{2 \cdot k}} \int_0^\theta \frac{-1}{\sqrt{\cos(\theta) - \cos(\alpha)}} \, d\theta
\]
(13)

Table 5 shows some tabulated results of corresponding values of \( y \), \( x \), \( s \) and \( \theta \) for a specimen of half length 3 inches and \( \alpha \) equals 40 degrees (corresponding to an end-to-end distance \( 2Xa \) of 5.3 inches).

<table>
<thead>
<tr>
<th>Table 5. Corresponding values of ( y ), ( x ), ( s ) and ( \theta ).</th>
</tr>
</thead>
<tbody>
<tr>
<td>( y ) (inches)</td>
</tr>
<tr>
<td>( x ) (inches)</td>
</tr>
<tr>
<td>( s ) (inches)</td>
</tr>
<tr>
<td>( \theta ) (rad)</td>
</tr>
</tbody>
</table>

3.2 Strain distribution along the length of the specimen

It is now possible to compute the maximum strain \( \varepsilon \) at each point along the length of the specimen. As established in part 2.4, assuming that the neutral axis is at the mid-thickness of the specimen (\( \varepsilon=0 \) at \( t/2 \)) we get:

42
\[ \varepsilon = \frac{t}{2 \cdot \rho} \]  \hspace{1cm} (14)

with \( t \) the total thickness of the specimen. The curvature radius \( \rho \) differs for each point of the specimen. The latter assumption is no more exact when damage occurs: the neutral axis will be slightly shifted. But as the change of shape of the specimen will be neglected as a function of time, we will also assume that the local shift of the neutral axis can be neglected. Inserting equation (8) in this last equation we get an expression of the strain as a function of \( y \):

\[ \varepsilon = \frac{t \cdot K(\alpha)^2}{2 \cdot L^2} \cdot y \]  \hspace{1cm} (15)

For a 3 inch half length specimen (0.5 inches width, 0.04 inches thickness) and for an angle \( \alpha \) equal to 40 degrees we obtain the following strain versus coordinate curves (Figures 21, 22, 23). On the following plots, the absolute value of the strain is given for the compression or tension side (at \( t = \pm0.02 \), at the mid thickness \( t = 0, \varepsilon = 0 \)).

\[ \text{Fig. 21 Strain versus } y \text{ at } t=0.02. \]
Fig. 22 Strain versus $x$ at $t=0.02$.

Fig. 23 Strain versus $s$ at $t=0.02$. 
3.3 Computation of the Young’s modulus and stress distribution along the length of the specimen

Knowing with accuracy the shape of the bent specimen and the strain at each point, it is possible to compute the stiffness distribution along the specimen.

The stiffness in the fiber direction in the undamaged zones can be estimated by a rule of mixtures:

\[ E_{11} = E_f V_f + E_m V_m \]  \hspace{1cm} (16)

Where the subscripts \( m \) and \( f \) are for matrix and fibers respectively, \( E \) is the Young’s modulus and \( V \) the volume fraction of each phase.

In the damaged regions, where microbuckling occurred, the fibers are now misaligned. The stiffness in the initial axial direction [11] will then be reduced due to the angle between the fibers and their initial position. To compute the modulus in this direction, the classical lamination theory is used. The modulus in the direction transverse to the fibers can be approximated as:

\[ E_{22} = \frac{1}{\frac{V_f}{E_f} + \frac{V_m}{E_m}} \]  \hspace{1cm} (17)

The Poisson’s ratio can also be considered as following a rule of mixture:

\[ \nu_{12} = \nu_f V_f + \nu_m V_m \]  \hspace{1cm} (18)

The shear moduli can be computed in both phases by:
\[ G = \frac{E}{2(1 + \nu)} \]  \hspace{1cm} (19)

Similarly to the transverse Young's modulus, the shear modulus in the composite can be written as:

\[ G_{12} = \frac{1}{V_f} \frac{V_m}{G_f + \frac{V_m}{G_m}} \]  \hspace{1cm} (20)

One should note that these are the simplest but not necessarily the most accurate micromechanics models for the computation of the elastic constants of the composite.

The stiffness matrix, \( Q \), is given by:

\[
Q = \begin{pmatrix}
E_{11} & \frac{v_{21} \cdot E_{11}}{1 - v_{12} \cdot v_{21}} & 0 \\
\frac{v_{12} \cdot E_{22}}{1 - v_{12} \cdot v_{21}} & E_{22} & 0 \\
\frac{v_{21}}{1 - v_{12} \cdot v_{21}} & \frac{v_{21}}{1 - v_{12} \cdot v_{21}} & G_{12}
\end{pmatrix}
\]  \hspace{1cm} (21)

Let \( \alpha_b \) be the rotation angle of the fibers in the kinked region as shown in Figure 24.

![Fig. 24 Kink band.](image-url)
The transformation matrix for this angle is:

\[
T = \begin{pmatrix}
    m^2 & n^2 & 2 \cdot m \cdot n \\
    n^2 & m^2 & -2 \cdot m \cdot n \\
    -m \cdot n & m \cdot n & (m^2 - n^2)
\end{pmatrix}
\]

(22)

with \( m = \cos(\alpha_b) \) (23) and \( n = \sin(\alpha_b) \) (24).

The transformed stiffness matrix can then be calculated:

\[
\overline{Q} = T^{-1} \cdot Q \cdot R \cdot T \cdot R^{-1}
\]

(25)

Where R is the Reuter matrix:

\[
R = \begin{pmatrix}
    1 & 0 & 0 \\
    0 & 1 & 0 \\
    0 & 0 & 2
\end{pmatrix}
\]

(26)

The stiffness in the buckled region is given by:

\[
E_{11} = \overline{Q}_{11} \cdot (1 - v_{12} \cdot v_{21})
\]

(27)

Knowing the positions of the microbuckles and their width \( \delta \) it is possible to obtain the stiffness distribution along the specimen by introducing Heaviside functions. The following equation is an example that can be used for any number of microbuckles. In this equation the 2 microbuckles are located at \( s_1 \) and \( s_2 \), where \( s \) is the curvilinear abscissa.
\[ E(s) = E_{11} \cdot H(s - s) + E_{\text{w,buckle11}} \cdot H(s - s) - E_{\text{buckle11}} \cdot H(s - (s_1 + w_{\text{buckle}})) + E_{11} \cdot H(s - (s_1 + w_{\text{buckle}})) \\
+ E_{11} \cdot H(s - (s_1 + w_{\text{buckle}})) - E_{11} \cdot H(s - s_2) + E_{\text{buckle11}} \cdot H(s - s_2) - E_{\text{buckle11}} \cdot H(s - (s_2 - w_{\text{buckle}})) \\
+ E_{11} \cdot H(s - (s_2 + w_{\text{buckle}})) \]  

(28)

Using the following plausible values at 120°C, it is possible to plot the variations of stiffness along the length on the damaged edge (compression side):  
\[ E_t=178.10^9 \text{ Pa}, \]
\[ E_m=3.10^9 \text{ Pa}, \]
\[ \nu=0.3, \quad \nu_m=0.4, \quad V_t=0.5135, \quad s_1=1.073 \text{ inches} \]
\[ s_2=2.395 \text{ inches}. \]

The width \( w \) of the microbuckles was taken very large (\( w=0.04 \text{ inches} \)) to allow a good representation of the local drops on graphs 25 and 26. The angle of the fibers in the kink band \( \alpha_0 \), is taken to be equal to 52 degrees, after measurement of the angle the SEM pictures. This value seems to agree with the experimental values given in Reference 10.

![Graph](image)  

**Fig. 25 Young’s modulus versus s.**
Finally, by multiplying this stiffness by the strain at each point, the stress distribution along the length of the damaged edge (compression side) can be obtained as shown in the Figure 26.

![Graph of stress versus s.](image)

**Fig. 26 Stress versus s.**

The last step to this analysis, but probably the most complicated, is the prediction of the nucleation and propagation of the microbuckles. As it has been seen previously, it is necessary to know the position and the number of microbuckles to be able to apply the previous analysis. Moreover, as described in part 2.6.a, the specimen was considered to be broken when the microbuckles cross the whole width of the specimen. The propagation of the microbuckle (length as a function of time) is then very important to know.
3.4 Nucleation of the microbuckles

Fleck, Budiansky and Slaughter [Ref. 13, 14, 15] work on the nucleation problem in the same way: a three-parameter constitutive equation relating shear strain rate and shear stress

$$\frac{\dot{\gamma}}{\dot{\gamma}_{\text{ref}}} = \left(\frac{\tau}{\tau_{\text{ref}}}\right)^M$$

(29)

is assumed for the material, where $\tau_{\text{ref}}$ (a reference shear stress) produces $\dot{\gamma}_{\text{ref}}$. In Reference 15, the viscoelastic character of the kink band is taken into account. A relationship between time (t), applied compressive stress ($\sigma$), the initial misalignment ($\bar{\phi}$) and the additional rotation ($\phi$) is established:

$$t = \left(\frac{1}{M-1}\right) \left(\frac{1}{\dot{\gamma}_{\text{ref}}}\right) \left(\frac{\tau^*_{\text{ref}}}{\sigma}\right)^M \left[\frac{1}{\bar{\phi}^{M-1}} - \frac{1}{(\bar{\phi} + \phi)^{M-1}}\right]$$

(30)

Where $\dot{\gamma}^*_{\text{ref}} = \frac{\dot{\gamma}_{\text{ref}}}{\alpha}$, $\tau^*_{\text{ref}} = \alpha \tau_{\text{ref}}$, $\alpha = \sqrt{1 + R^2 \tan^2 \beta}$, $R$ is a parameter relating the transverse to shear creep strength and $\beta$ is the kink angle as defined in Figure 27.

![Fig. 27 Kink band angles.](image)

50
The creep-kinking life time is defined as a time when the total rotation \((\phi + \bar{\phi})\) is infinite (upper bound approximation). This criterion can be written as:

\[
t_f = \left[ (M - 1)\dot{\gamma}_{ref} \phi^{M-1} \left( \frac{\sigma}{\tau_{ref}} \right)^M \right]^{-1}
\]

For the present data, the following values are found to fit the experimental data:

- \(M = 1.1\)
- \(\dot{\gamma}_{ref} = 10^{-7} \text{ sec}^{-1}\)
- \(\tau_{ref} = 0.0015 \text{ MPa}\)
- \(\alpha = 1\)
- \(\bar{\phi} = 0.05236 \text{ rad (3 degrees)}\)
- \(E = 9.26 \times 10^4 \text{ MPa at 120°C}\).

The applied stress is taken as the maximum local stress (at the center of the specimen), as computed in the previous analysis.

The calculated values fit the experimental data quite well as shown in Figure 28 with an average relative error of 20% (all the error computations have been made eliminating the worst point: outlier).
Fig. 28 Experimental and calculated time-to-failures versus maximum strain (at the center of the composite) at 120°C.

Using this model, it is possible to determine the fiber angle within the microbuckles as a function of time. This value can be used in the previous analysis to compute the Young’s modulus along the length as a function of time. However, the previous failure criterion (equation 31) has to be modified. The assumption that failure occurs when the misalignment angle is infinite would lead to absurd values. The maximum reasonable value for $(\phi + \bar{\phi})$ is 90 degrees. At this value the modulus reaches its minimum value ($E_{22}$). So the time-to-failure for this new criterion can be written as:

$$t_f = \frac{1}{(M-1) \cdot \gamma_{re} \left( \frac{\sigma_{\text{max}}}{	au_{ref}} \right)^M} \left[ \frac{1}{\bar{\phi}} \left( \frac{\pi}{2} \right)^{M-1} - \frac{1}{\bar{\phi}} \right]$$

(31)

For any time $t$ before failure it is possible to know the total rotation angle of the fibers $(\phi + \bar{\phi})$. For the two microbuckles considered in equation (28), at the same time $t$, the
angles will be different due to the difference in the local stresses. By inverting equation (30), we get a relationship that allows us to compute the rotation angle:

\[
\alpha_s(t) = \phi(t) + \bar{\phi}
\]

(32)

\[
\alpha_s(t) = \frac{1}{\left( \frac{1}{\phi_{M-1}} - (M - 1)(\gamma_{ys}) \left( \varepsilon(y) \cdot \frac{E_{11}}{\tau_{ref}} \right)^{M-1} \right)} \cdot t
\]

(33)

If we apply the classical lamination theory for this angle \(\alpha_s(t)\), the variations of the modulus in the microbuckled region can be computed as a function of time. Figures 29 and 30 show the drop of modulus in the microbuckled regions (located at \(s_1\) and \(s_2\)) as a function of time.

![Graph of Modulus versus time](image)

**Fig. 29** Modulus versus time in the microbuckled region located at \(s_1\).
Fig. 30 Modulus versus time in the microbuckled region located at $s_2$.

For a given time, it is now possible to get the distribution of the modulus along the length of the specimen including in the microbuckled regions (Figure 31).

Fig. 31 Modulus versus $s$ at $t=t_f/2$. 
The stress distribution along the length of the specimen at a given time can be obtained by multiplying the computed modulus at each point for this time by the computed strain at each point. This assumes that the change of shape of the specimen (and the change of curvature) can be neglected. Figures 32, 33 and 34, show the stress distribution versus the different coordinate systems at $t=t_d/2$.

![Graph showing stress versus y (inches) at $t=t_d/2$.]
Fig. 33 Stress versus $x$ (inches) at $t=t/2$.

Fig. 34 Stress versus $s$ (inches) at $t=t/2$. 
But this model includes some limitations. First, the changes in shape of the bent specimen are neglected. The second problem is that the reference values have been obtained by curve fitting. It is then very difficult to generalize these results. The influence of temperature is embedded in these coefficients and does not appear analytically. One way to obtain these values experimentally would be to conduct a creep test. The last problem is that the geometry of the specimen is never taken into account. We have seen previously that the chosen failure criterion is the time when the microbuckle will reach the other side of the specimen. The propagation or the length of the kink band should then be considered.

3.5 Propagation of the microbuckles

The propagation of the kink band across the width of the specimen can be represented, to a first approximation, as a thin crack propagating across the fibers and the matrix. In this case, a Paris law can be used.

\[
\frac{dc}{dt} = A \cdot K^n
\]

(34)

\[
K = \sigma_{app} \sqrt{\pi c}
\]

\[
\frac{dc}{dt} = A \sigma_{app}^n \pi^{n/2} c^{n/2}
\]
\[ \int_{c_0}^{w} \frac{dc}{dt} = \int_0^{t_f} A \sigma_{app}^n \pi^{n/2} c^{n/2} \]

\[
\frac{w}{c_0} = \left[ 1 - \left( \frac{n}{2} - 1 \right) A \sigma_{app}^n \pi^{n/2} c_0^{n/2} c_c^{n-1} t_f \right]^{\frac{1}{n-1}}
\]

Finally the time-to-failure is given by:

\[
t_f = \frac{1 - \left( \frac{w}{c_0} \right)^{\frac{n}{2}-1}}{\left( \frac{n}{2} - 1 \right) A \sigma_{app}^n \pi^{2-n}}
\]

(35)

Where \(c_0\) is the length of the initial flaw, \(w\) is the width of the specimen, \(A\) and \(n\) are two material parameters.

A fairly good fit (average error of 34%) is obtained for \(c_0\) equals to 0.001 mm and \(n=1\) as shown on Figure 35.

![Graph](image)

**Fig. 35 Time-to-failure versus maximum strain at 120°C.**
Equation 35 is very similar to Kachanov’s model mathematically derived in a more general case [Ref. 29]:

\[ t_j = \frac{1}{(n + 1)Ae_i^a} \]  

(36)

This relation takes into account the growth of the kink band across the width but it results from a curve fit and again, the temperature only intervenes in the material parameters A and n. Moreover, no description (angle) of the kink band can be obtained.

In most of the models existing in the literature, the problem of the nucleation of the microbuckles at the free edges are not explained nor taken into account. This problem and the one of the geometry are studied in one paper. Reference 12 suggests a model based on elastic-plastic surface instability. The total energy dissipated from the matrix material within the kink band of half width w and horizontal projection a can be written as:

\[ E = 2waV_m \int_0^a |\sigma_m| d|\varepsilon| + E_s \]  

(37)

The first term represents the energy dissipation in the matrix of the damaged kink band and Es is the energy needed for the creation of a free surface. From this point the J integral is calculated. But the obtained expression is time independent and shear values are needed that are very difficult to obtain experimentally. An easier energy model can be obtained by using the Griffith criterion modified by Kanninen [Ref. 30]:
\[
G = \frac{1}{2} P^2 \frac{dS}{da}
\]  
(38)

where \( G \) is the strain energy release or the crack driving force, \( P \) is the applied load and \( S \) is the compliance.

Assuming that \( G \) is time independent, we can define \( G_{\text{critical}} \) as the strain energy release defined when the microbuckle crosses the whole width of the specimen.

\[
G_c = \frac{P^2}{2w} \left( S_{\text{Damaged}} - S_{\text{Undamaged}} \right)
\]  
(39)

Starting from equation : 

\[
EI \frac{d\theta}{ds} = -P_y
\]

\( P_{\text{max}} \) can be obtained:

\[
P_{\text{max}} = \frac{EI}{\rho_a Y_u}
\]  
(40)

Where

\[
I = \frac{wt^3}{12}
\]  
(41)

Then

\[
G_c = \frac{wE^2t^6}{288\rho_a^2Y_a^2} \left( S_{\text{Damaged}} - S_{\text{Undamaged}} \right).
\]  
(42)

The compliances can be obtained by the classical lamination theory. The compliance in the undamaged region is only the inverse of the stiffness:

\[
S_{\text{Undamaged}} = \frac{1}{E_{11}}
\]  
(43)

In the kink bands the compliance will increase. The compliance matrix is given by the CLT:
\[ S = \begin{pmatrix} \frac{1}{E_{11}} & -\frac{v_{21}}{E_{22}} & 0 \\ -\frac{v_{12}}{E_{11}} & \frac{1}{E_{22}} & 0 \\ 0 & 0 & \frac{1}{G_{12}} \end{pmatrix} \] (44)

Then the compliance in the microbuckled region is given by:

\[ S_{Damaged} = S_{11}m^4 + (2S_{12} + S_{33})n^2m^2 + S_{22}n^4 \] (45)

Considering that the rotation angle reaches a maximum value \( \alpha_b \) as assumed in a first place (\( \alpha_b = 52 \) deg), then \( m = \cos(\alpha_b) \) (23) and \( n = \sin(\alpha_b) \) (24).

As in the case of the Paris Law, but this time in the general case, it is possible to relate the microbuckling growth to the total strain energy release rate:

\[ \frac{dc}{dt} = AG^B \] (46)

For each strain level, \( G \) can be computed then the time to failure is given by:

\[ t_f = \frac{w}{AG^B} \] (47)

where \( A \) and \( B \) are material parameters (\( G \) is still assumed to be time independent).

The variations of \( Gc \) as a function of the strain level is shown in Figure 36 and the time to failure versus the strain level at 120°C is plotted in Figure 37. The best fit is given for \( w/A=12 \) and \( B=6.5 \).
Fig. 36 Strain energy release versus calculated strain.

Fig. 37 Time-to-failure versus calculated strain.
This last model is the best: no assumption concerning the shape of the kink band has been made. Equations 42 and 47 have been derived for the general case and the correlation between calculated and experimental data seems good (average error: 14%).

3.6 The influence of temperature

The previous analysis takes into account the viscoelastic character of the composite. Intuitively, as in the case of polymers, a correspondence between time and temperature should exist. The previous parts dealt mostly with time and it is now interesting to analyze the influence of temperature on the fracture of the specimen. Six specimens were bent at 90% strain-to-failure and the time-to-failure was recorded for different temperatures. Figure 38 shows the results. The time-to-failure increases significantly as the temperature decreases. One should remember when analyzing this plot that the glass transition starts at 100°C (based on the DMA, Fig.3).

![Graph showing temperature versus time-to-failure for ε/ε_{ultimate}=90%](image.png)

*Fig. 38 Temperature versus time-to-failure for ε/ε_{ultimate}=90%.*
As in the case of polymers, a high temperature corresponds to short times and vice-versa. If we try to apply the previous models to the 90°C data, the values are harder to curve-fit because the curves are very steep. For the Budiansky and Fleck model, for example, the time to failure increases quickly as the strain decreases. In Figure 39, the curve fit (average error: 38%) is shown for a value of $\tau_{ref}$ around 30 MPa and 10 for the power coefficient M (instead of 1.1 for the 120°C case).

![Graph showing time-to-failure vs strain](image.png)

**Fig. 39 Experimental and calculated time-to-failures versus maximum strain**

(at the center of the composite) at 90°C.

The same kind of observation can be made if we try to apply the Paris law. The coefficient A in equation (35) becomes very small (the order of $10^{-29}$) and the power coefficient becomes very large ($n=10$) at 90°C. The curve-fit is shown in Figure 40 (average error 35%).
Fig. 40 Time-to-failure versus maximum strain at 90°C.

Finally, we can apply the same treatment to the energy method, the w/A coefficient in equation 47 becomes 0.27 and B becomes 32.3. For the considered points, the following strain energy release versus calculated strain is obtained (Figure 41).

Fig. 41 Strain energy release versus calculated strain.
By plotting the time-to-failure versus calculated strain at 90°C, a problem arises. The correlation between calculated strain and the measured strain is not as good for the experiments at 90°C as for the experiments at 120°C. If one plots the time to failure versus experimental strain, the calculated time-to-failure curve exhibits some scattered data (Figure 42).

![Graph showing time-to-failure versus experimental strain at 90°C.](image)

**Fig. 42 Time-to-failure versus experimental strain at 90°C.**

On another hand, if we plot the time-to-failure versus calculated strain, the experimental data exhibit some scatter, while the calculated curve is smooth (Figure 43) (average error 41%).
Fig. 43 Time-to-failure versus calculated strain at 90°C.
4 RESULTS AND CONCLUSIONS

4.1 Summary

A new method for stress-rupture experiments has been developed. The use of very simple fixtures led to coherent results. These results have been modelled and equations have been derived to predict the strain at each point of the specimen if the end-to-end distance is known.

Several experiments enabled the observations of the stress rupture of unidirectional polymer matrix composite specimens and the characterization of the failure process under bending. The failure appears to be compressive, driven by the presence of microbuckles. These microbuckles do not seem to interact with each other, but their number is related to the time-to-failure of the specimen.

Considering the local drop in modulus in the damaged regions, the distribution of the Young’s modulus and then the stress is given along the length of the specimen based on the elastica and classical lamination theories.

Three models have been applied to model the time-to-failure versus strain behavior at 90°C and 120°C. In the first micromechanical model, the nucleation of the microbuckles is considered as the main cause of fracture. The rotation of the fibers in the
damaged region as a function of time is used to compute the stiffness and stress distributions at any time before failure. Two other models are developed using the idea that the propagation of the largest microbuckle determines the time-to-failure of the whole specimen. In a first approximation a Paris-type law is used. Then a more accurate model is derived, based on energy considerations. For this last model, no assumption is made concerning the damage geometry, and the correlation between experimental and calculated data is good. Temperature effects are included only in the material parameters.

4.2 Recommendations

There are several ways to further develop the analysis. The first way would be to investigate the influence of the geometry by testing specimens with various dimensions. If this influence is significant, the energy analysis becomes the most favored model. The effect of edges and surfaces on the formation of the microbuckles also needs further investigation.

The possibility of establishing a time-temperature equivalence such as the WLF equation for polymers needs to be explored. This would enable the computation of the time-to-failure of the material at any temperature from a single time-to-failure versus strain reference curve. The influence of the glass transition can be a limitation to this approach and also needs further investigation.
It also seems desirable to relate the curve-fitting parameters of the three models to the mechanical properties of the material, so that it will be possible to predict the time-to-failure without requiring a great deal of experimental data. However, this would probably require preliminary experiments (creep tests). The residual strength of the broken specimens can be given by post-failure experiments. Another polymer matrix composite can also be studied. The differences between the results of the materials can be related to their mechanical properties. Ultimately, it would be interesting to integrate these micromechanics models into the MRLife software.
APPENDIX

DSC.

Change of shape of the specimen, pictures of the videotape.
DSC of the composite.
First specimen: bent at 80% strain to failure, first frame.
First specimen: bent at 80% strain to failure, last frame before failure.
Second specimen: bent at 80% strain to failure, first frame.
Second specimen: bent at 80% strain to failure, last frame before failure.
Third specimen: bent at 65% strain to failure, first frame.
Third specimen: bent at 65% strain to failure, last frame before failure.
REFERENCES


Ref. 33: Baycomp. Private communication.
VITA

Céline A. Mahieux, daughter of Guitty Fakhr and Francis Mahieux, was born on February 23, 1973 in Teheran, Iran. She grew up in Argenteuil, a northern suburb of Paris, France. She attended Middle school and High school at the College et Lycee Notre Dame de Bury, France. After graduation she attended the Université de Technologie de Compiègne (UTC, France). After two years of general engineering she entered the department of Mechanical Engineering and specialized in Materials and Technologic Innovation. She was then selected for an exchange program between UTC and Virginia Polytechnic Institute and State University (VPI, USA). In August 1995, she moved to Blacksburg to enroll the Materials Science and Engineering Master’s program and in January 1996, she started her research on composite materials in the Material Response Group of the Engineering Science and Mechanics department. Céline plans to pursue her study of composite materials in the Materials Response Group as a Ph. D. student.