CREEP TESTING OF HIGH PERFORMANCE MATERIALS FOR INFLATABLE STRUCTURES

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ABSTRACT

Inflatable structures made from flexible coated fabrics are currently used in many applications including airships, aerostats, and radomes where the materials are under constant long term loading. Future applications, where the material will be under similar loading conditions, include inflatable habitats for the Moon and the exploration of Mars. To prevent premature failure of these types of structures, the effects of long term tensile loading must be taken into account. Creep testing was performed by ILC Dover using a similar method as that used in geotextile engineering to establish a protocol for textile based inflatable structures manufactured from high performance fibers. This paper will discuss creep testing methodology and present data for two typical textiles used in inflatable structures: polyester and Vectran™. The materials were shown to follow Arrhenius and/or Williams-Landel-Ferry (WLF) behavior depending on their crystallinity. This testing methodology can be used as a guideline to determine the appropriate design safety factors once the system level temperature, time, and loading requirements are known.

KEYWORDS: Advanced Composite Materials / Structures, Applications – Aerospace, Viscoelasticity / Creep Behavior

1. INTRODUCTION

One of the more challenging aspects of designing inflatable structures is determining the optimal stress levels that the material can be allowed to experience during long-term operation to minimize system level mass. The results that are obtained from short duration tests for the ultimate tensile strength (UTS) of the material are only part of the story. For example, if a structure is designed to be loaded at 100% of the UTS of the material, creep rupture would occur almost instantaneously upon initial loading. If the same structure was loaded to 50% UTS of the material, then it would take longer for creep rupture to occur. Creep rupture occurs after a viscoelastic material has been under constant load for a period of time. Viscoelastic materials exhibit elastic responses when subjected to rapid loading (like a solid) but they have a viscous response when subjected to long-term loads (like a high viscosity liquid). Real-time tests could be performed to determine how long the material will last at 50% UTS, but for some materials it might take years to obtain results. Instead, the lifetime can be predicted by using time-temperature superposition to accelerate the test.

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The overall goal of this type of test is to accelerate the creep of the material by increasing the temperature periodically so that creep rupture is forced to occur within one day. This data can then be analyzed to provide a lifetime prediction for the material at a predetermined load and temperature. This is the major advantage of this test because it allows the engineer to predict the performance of a material which may last years or decades under constant load in a short time period (1). This test is used in geotextile engineering where materials are used to stabilize slopes and provide earthquake protection. In these instances, the materials have design lifetimes over 100 years which again is why this test is of great utility.

Inflatable structures made from textile based materials can also benefit from this test. For example, aerostats, high altitude balloons, airships, radomes, and habitats have design lifetimes ranging from 1 to 20 years (2, 3, 4, 5, 6). In some cases these structures will be inflated during their entire lifetime which means that the material will see constant stress. As opposed to geotextile engineered structures, the stress in inflatable structures may not be held at a constant value because of the diurnal cycle. Therefore when applying the time-temperature superposition technique, it is important to utilize the worst case loading conditions (maximum time and temperature) to determine the optimal stress in the material. This optimal stress is determined based on a trade between reliability, safety, and system mass. Examples of inflatable structures that will see high stress for long time periods are shown in Figures 1-3. Figure 1 shows a 30.5-m (100-ft) diameter radome, Figure 2 shows a prototype lunar habitat, and 11,893m³ (420K ft³) volume tethered aerostat is shown in Figure 3.

**Figure 1. Sea Based Inflatable Radome**

**Figure 2. Lunar Inflatable Habitat**

**Figure 3. Inflatable Tethered Aerostat**
It is important to note the difference between creep rupture and creep failure. Creep rupture is the time when the material actually breaks due to long term loading and is what most creep tests measure. Creep failure, as defined by Zornberg, can be determined by drawing a best fit line through the initial set of strain data (1). The point at which the shifted strain data begins to deviate from this best fit line is considered the creep failure time (1). At this point, the material will show significant permanent deformation even when the load is removed. Creep failure does not result in a catastrophic failure of the structure, but the material is now well on its way to creep rupture. As lighter-than-air (LTA) inflatables are concerned, creep failure must be considered because it will impact both aerodynamic performance and overall volume. Recent creep testing was performed using time-temperature superposition and is discussed in the following sections.

2. THEORETICAL DISCUSSION

The creep test is predicated on the Boltzman superposition principle. This theory states that the strains that occur in a material from different loading events are additive (1). In the case of the creep test, this means that the creep strain that occurs at the periodic temperature steps constitute different loading events and therefore can be added together to determine the total strain (1). Time and temperature data from the one day creep test are related using either the Arrhenius (eq. 1) or the WLF equations (eq. 2).

\[
\text{Log}(aT) := \frac{Ea}{2.303R} \left( \frac{1}{T} - \frac{1}{T_r} \right) \quad (eq. 1), (7)
\]

Where \(aT\) is the time shift factor, \(Ea\) is the activation energy, \(R\) is the Universal Gas Constant, \(T\) is the test temperature, and \(T_r\) is the reference temperature.

\[
\text{Log}(aT) := \frac{-c1 \cdot (T - T_r)}{c2 + T - T_r} \quad (eq. 2), (8)
\]

Where all of the variables were previously defined except for \(c1\) and \(c2\). If \(T_r = T_g\) then \(c1\) and \(c2\) are “universal constants” and for most polymers are 17.4 and 51.6, respectively (8). Otherwise, \(c1\) and \(c2\) must be calculated using equations 3 and 4.

\[
c1 := \frac{[(17.4) \cdot (51.6)]}{(17.4 + T_g - T_r)} \quad (eq. 3), (7)
\]

\[
c2 := 51.6 + T_g - T_r \quad (eq. 4), (7)
\]

These equations are used to check the correlation between the data and the theory for the materials that are tested and can also be used to predict the lifetime of the material at different temperatures. The validity of these two equations depends on the viscoelastic region of the
polymer where the test is performed. Semi-crystalline materials, such as nylon and polyester, have a glassy, a rubbery, and liquid regions. The point below which the semi-crystalline material becomes stiff (i.e. glassy) is termed the glass transition temperature (Tg). When performing this test on a semi-crystalline material it is important not to test through the Tg because specific volume is discontinuous at that point, which will adversely affect the results of the test (8). Because of this, it is important to determine the Tg of the material prior to beginning any creep test program. Crystalline materials, such as Vectran™, ultra high molecular weight polyethylene (UHMWPE) and carbon, do not exhibit a glass transition temperature and upon heating directly turn into liquids (or burn) from their solid states at their melting temperature. The regions of the polymer where the WLF and Arrhenius equations are valid are summarized in Table 1.

<table>
<thead>
<tr>
<th>Equation</th>
<th>Material</th>
<th>Valid Regions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arrhenius</td>
<td>Semi-Crystalline</td>
<td>Below Tg; &gt;Tg+100°C</td>
</tr>
<tr>
<td></td>
<td>Crystalline</td>
<td>All Regions</td>
</tr>
<tr>
<td>WLF</td>
<td>Semi-Crystalline</td>
<td>Tg to 100°C + Tg</td>
</tr>
</tbody>
</table>

3. MATERIALS AND TESTING

Multiple materials were tested during this study in order to compare different creep responses including polyester laminates, UHMWPE, Vectran™ fabrics, and Vectran™ laminates. The results from the testing of two of these materials are presented here: a commercially available polyester laminate and a custom woven Vectran™ fabric. The polyester laminate consisted of three layers: a polyester fabric, an adhesive, and a 0.0127-mm (0.5-mil) thick polyester film. In this case the term polyester, refers to poly(ethylene terephthalate) (PET) which is semicrystalline and has a glass transition temperature (Tg) of approximately 70°C (10). The Vectran™ material is a woven fabric, with no coatings or films attached to it. The fiber used in the fabric is Vectran™ HS, which is a liquid crystal polymer with a melting point of approximately 330°C (11). Since Vectran™ is a crystalline material, it does not have a Tg. These two materials were chosen to examine the different responses of semi-crystalline and crystalline materials during the test. They were also chosen to determine if both fabrics and textile laminates could be tested using this protocol and the results reported here demonstrate this capability.

3.1 Test Procedure The test is performed in a two step manner by first determining the ultimate tensile strength (UTS) of the material in a Fed. Std. 191-5104 tensile test and then performing the creep test at some percentage of the UTS. In the case of the results presented here, the UTS was determined by testing the samples at 304.8-mm/minute (12-inches/minute) on 50.8-mm (2-inch) wide samples for the polyester laminate and 25.4-mm (1-inch) wide samples for the Vectran™ fabric.

The creep test is performed using an environmental chamber which is set to a predetermined start temperature and relative humidity. The test is performed on samples in series, which eliminates sample to sample variability (1). During the test, the creep of the material is accelerated by increasing the temperature in the environmental chamber by approximately 10°C every 2 hours until failure (1, 12). Strain, time, and temperature are recorded during the entire test. This procedure is referred to as the stepped isothermal method (1). A 0-50% MTS extensometer was
used to measure the strain of the polyester laminate while a 0-9% MTS extensometer was used for the Vectran™ fabric. This is due to the fact that Vectran™ fiber has a considerably lower elongation to failure (~3.3%) than polyester fiber (>12%). After the test, the data is plotted and an appropriate time shift factor, \(aT\), is determined. The data is then shifted horizontally on the time axis by \(t^* = t/aT\) to create master curves which are used to predict the lifetime of the material (1, 7, 8). The data did not require vertical shifting as is referenced in the literature (7). The data can be compared to the WLF or Arrhenius equations as appropriate, depending on the crystallinity of the material.

For the results presented here, almost all of the samples were tested at a relative humidity of 20%. Some initial tests were performed at lower percent relative humidity (~5%) but it was found that the temperature variation in the environmental chamber was more difficult to control at lower humidity. Maintaining the relative humidity at 20% also minimizes the moisture adsorption affect of hydroscopic materials such as nylon and aramids. The end-use application environment of the system may help the engineer select the appropriate humidity level for the test. The predetermined starting temperature was generally 25\(^\circ\)C. However, in some cases the starting temperature was increased in order to further accelerate the creep test. The loads were varied between 30 and 80\% UTS. When using this test protocol, it is imperative to consider the operational profile of the system to select the time at temperature and the load to meet the requirements. The test set-up is shown in Figure 4. Note that prior to attaching the extensometer a 1% load is applied to the sample in order to provide some pre-tension in the material to remove some of the initial crimp in the fabric. Bollard grips were used to test all of the samples to minimize stress concentrations at the grips. None of the samples failed at the grips.

3.2 Data Analysis and Model Correlation
The raw data from a creep test that was performed on the polyester laminate at 60\% UTS is shown in Figure 5. Strain is plotted on the primary Y axis, while temperature is plotted on the secondary Y axis. The three distinct temperature increases can clearly be seen in the data. Temperature equilibration occurred in approximately 4
minutes. There is some variation in the temperature during the test because of the cycling of the environmental chamber, but the standard deviation was approximately +/-1.5 to 2.2°C for the results presented. As seen in the chart, the strain increases during each temperature ramp. This data is superimposed to determine the design lifetime (1, 7, 8).

**Figure 5. Raw Creep Data from the Polyester Laminate at 60% UTS**

In Figure 6, the raw creep data has been plotted on a semi-log plot and the time has been zeroed at each test temperature. The data shown includes both non-steady state and steady state creep. In order to horizontally shift the strain data, the final slope of the first set of data is matched with the initial slope of the next set of data and the non-steady state creep data is removed (1, 7, 8). The data is shifted to the first test temperature which in this case is 22°C.

**Figure 6. Strain Rate Data at Different Temperatures for Polyester Laminate at 60% UTS**
In the process of horizontally shifting the data, the values of aT for each different test temperature are determined (8). Upon review of equation 1 and assuming that the Arrhenius equation is valid in this region, a plot of the log of aT vs. \((1/T - 1/T_r)\) should be a straight line. A sample of one of these plots is shown in Figure 7.

![Arrhenius Plot](image)

**Figure 7. Arrhenius Plot for Polyester Laminate at 60% UTS**

Since the correlation factor is high \((R^2=0.991)\) in Figure 7, it can be concluded that the data does fit the Arrhenius equation. From the slope of the line in Figure 7 and after reviewing Equation 1 the activation energy can be determined. This is important because once the activation energy is known, the strain data can be shifted to another reference temperature as will be discussed later. Using equations 1–4, Log aT can be calculated and compared to the test data as shown in Figure 8 for the polyester laminate.

![Data and Model Comparison Plot](image)

**Figure 8. Data, Arrhenius, and WLF Comparison for Polyester Laminate at 60% UTS**
The correlation between the data and both models is excellent in this region, which indicates that the test procedure used to collect the data is valid. It is interesting to note the correlation between the data and the WLF equation in this region, which is near the Tg of PET, but still below it. This is a region where the WLF equation is generally not valid. However the fact that this material is a laminate and not pure PET could be the root cause of this unexpected correlation between the data and both models. Similar results were seen for all of the data collected on the polyester laminate, but not with the uncoated Vectran™ fabric. In the case of the Vectran™ fabric only the Arrhenius equation showed good correlation with the data, as would be expected with this highly crystalline material.

3.3 Polyester Laminate Master Curves The master curve for the polyester laminate at 60% UTS is shown in Figure 9. This curve was generated using the information shown in Figures 5-8 (1, 7, 8). Note that all of the samples in this study were tested in the warp direction.

The remainder of the master curves for the polyester laminate are shown in Figures 10-12 for the tests that were performed at 30, 50, and 70% UTS. As would be expected, the data show that as the %UTS is decreased, the creep failure time increases considerably. For example, at 30% UTS, the polyester laminate would not see creep failure for approximately 88,500 hours or 10 years.

During the 50% UTS creep test the temperature was raised above the Tg of PET (70°C). Since the Tg was traversed, these two strain data sets (80.7°C and 91.7°C) should not be used to determine the creep failure time. A significant slope change can be seen, especially in the 97.1°C strain data illustrating the fact that specific volume versus temperature is not continuous through the Tg of the material. However, creep failure occurred during the 67.2°C isotherm temperature, which is still below the Tg and therefore this creep failure time prediction is valid.
In most cases, with the polyester laminate, creep rupture was not seen within the 1 day test. However, at 70% UTS, creep rupture was witnessed during the test and is predicted to occur after approximately 1,000 hours (Figure 12).
3.4 Polyester Laminate Creep Failure Curve

Once enough data is gathered at various percentages of the UTS, a plot of percent loading versus creep failure or creep rupture time can be created for each material tested (1). This is shown in Figure 13 for the polyester laminate. In this case, each data point only represents one sample and therefore the data is not statistically significant. However, a definitive trend can be seen in the initial data and once it is filled in with more data, a plot like this would be of great use to the design team. It allows the team to determine the highest possible load that the material can support for a given time and temperature. For inflatable applications, like stratospheric balloons and airships, this data may allow a safety factor decrease, which in turn reduces overall system weight.
3.5 Vectran™ Fabric Master Curves The Vectran™ fabric was tested in a similar fashion as the polyester laminate in order to create a similar creep failure curve that is shown in Figure 13. For the Vectran™ fabric, the testing was performed until creep rupture occurred. This is mainly because it was much more difficult to determine where the creep failure point occurred in the Vectran™ fabric as compared to the polyester laminate. Whereas the polyester material began to drift above the line of best fit when creep failure was occurring, Vectran™ did not generally exhibit that behavior and in fact in some cases the strain data began to drift below the best fit line. This behavior may be described by the log model discussed by Fette and Sovinski (11).

Although the polyester laminate shows good creep resistance, the Vectran™ fabric exhibited an even higher degree of creep resistance. In order to force a creep rupture failure in the Vectran™ fabric at 40% UTS, three tests had to be performed. The first test began with a starting temperature of 24°C, but rupture was not seen (Figure 14). For the second test, a starting temperature of 46°C was used, but again rupture was not recorded during the test (Figure 15). Finally, creep rupture at 40% UTS was recorded when the starting temperature was increased to 80°C (Figure 16). Being able to increase the starting temperature of the test in order to force a failure or rupture in the test is a major benefit of this testing protocol. Because the strain data at 80°C followed Arrhenius behavior, it was able to be super-positioned to a lower reference temperature. In this case, this lower temperature was 25°C (Figure 17.).

![Figure 14. Vectran™ Fabric Master Curve at 40% UTS, T_r=24°C](image-url)
Figure 15. Vectran™ Fabric Master Curve at 40% UTS, T_r=46°C

No Failure was seen at this starting temperature

Figure 16. Vectran™ Fabric Master Curve at 40% UTS, T_r=80°C

Creep Failure

Creep Rupture
The creep rupture data for the Vectran fabric at 25°C was more easily obtained as the load was increased for the remaining tests at 50, 60, and 80% UTS (Figures 18-20). All of these tests ruptured within four isotherms. In fact the 80% UTS test experience rupture within the first isotherm in about 0.04 hours so a creep master curve was not able to be created (Figure 20).
3.6 Vectran™ Fabric Creep Failure / Creep Rupture Curve A similar curve as that constructed for the polyester laminate is shown in Figure 21 for the Vectran™ fabric. Creep rupture and creep failure times are plotted against the %UTS. The creep rupture times are easily selected from the individual master curves, while creep failure was more subjective for Vectran™. Although these data points only include one sample at each percentage of UTS and is therefore not statistically significant, there is still a clear trend in the data. Once enough data is gathered, charts such as these can assist engineers in setting design allowables for specific applications.
Figure 21. Creep Failure / Creep Rupture for Vectran at T\textsubscript{r}~25°C

4. SUMMARY

Time-temperature superposition can be used to predict the lifetime of flexible composite materials. This reliable and repeatable short duration accelerated creep test has been developed to replace long term real-time testing. The test allows the study of creep failure and creep rupture whereas traditional tests only demonstrate creep rupture. The data can be shifted to different reference temperatures (higher or lower than the initial test temperature) in order to predict the material lifetime in other application environments. The percent relative humidity can be varied during the test depending on the application requirements. Vectran\textsuperscript{TM} demonstrates excellent creep resistance at room temperature while polyester displays very good resistance. Both materials will show increased creep resistance as the temperature is decreased, while they will creep more at higher temperatures. The polyester laminate followed Arrhenius and WLF behavior, while the Vectran\textsuperscript{TM} fabric displayed Arrhenius behavior only. Many other types of flexible composite materials can be tested using this method to determine the appropriate factors of safety in design. It is important to be aware of the system level requirements for time and temperature in order to determine the allowable maximum stress in the material. This test has significant implications for the design of inflatable high altitude LTA vehicles, radomes, and habitats. Specifically it allows engineers to select more aggressive safety factors than traditionally used, resulting in lower system mass and packing volume while not compromising safety and reliability.

5. ACKNOWLEDGEMENTS

The authors thank the following people for their advice and test support at ILC Dover: Gume Rodriguez, Sunil Inamdar, John Balcerak, Rich Bork, and Bill Ayrey.
6. REFERENCES