Uniaxial compression and creep behavior of a porous state change material

Stacy Michelle Nelson
University of Nevada, Las Vegas

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UNIAXIAL COMPRESSION AND CREEP BEHAVIOR OF
A POROUS STATE CHANGE MATERIAL

by

Stacy Michelle Nelson
Bachelor of Science
University of Nevada, Las Vegas
2006

A thesis submitted in partial fulfillment
of the requirements for the

Master of Science Degree in Mechanical Engineering
Department of Mechanical Engineering
Howard R. Hughes College of Engineering

Graduate College
University of Nevada, Las Vegas
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The Thesis prepared by

Stacy Michelle Nelson

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By

Examination Committee Chair

Dean of the Graduate College

Examination Committee Member

Examination Committee Member

Graduate College Faculty Representative
ABSTRACT

Uniaxial Compression and Creep Behavior of a Porous State Change Material

by

Stacy Michelle Nelson

Dr. Brendan O'Toole, Examination Committee Chair
Associate Professor of Mechanical Engineering
University of Nevada, Las Vegas

A reconfigurable tooling system (RTS) has been developed for use in manufacturing composite parts and molding liquid polymers. The RTS utilizes a state-change material that may also be useful as a water-soluble tooling material. The state-change material transitions from a liquid state to a solid state and then back to a liquid state. The material in its liquid state consists of glass microspheres in a solution of water-based binder and water. Through the application of heat and vacuum, the material can be hardened. When the material is in its solid state, the glass microspheres are “glued” together by small amounts of binder remaining in capillarity. Previous studies have been conducted to determine certain mechanical properties of the material in its hardened state. The focus of this study is to characterize variations of the state-change material in uniaxial compression at both ambient and elevated temperatures. Additionally, the room temperature uniaxial compression data is applied to a uniaxial compression creep study. The results show that material variation and test temperature significantly affect the compression and creep properties.
# TABLE OF CONTENTS

**ABSTRACT** ....................................................................................................................... iii

**TABLE OF CONTENTS** ........................................................................................................ iv

**LIST OF FIGURES** ........................................................................................................ vi

**LIST OF TABLES** ........................................................................................................... viii

**ACKNOWLEDGEMENTS** ................................................................................................. ix

**CHAPTER 1  INTRODUCTION** ....................................................................................... 1
  1.1 The 2Phase Reconfigurable Tooling System (RTS).................................................... 1
  1.2 The 2Phase State Change Material for Water Soluble Tooling ................................. 3
  1.3 The 2Phase State Change Material ........................................................................ 4
  1.4 Literature Review....................................................................................................... 6
    1.4.1 Review of Recent Research Related to Fly Ash Concrete.................................. 6
    1.4.2 Review of Recent Research Related to Syntactic Foams .................................... 9
    1.4.3 Review of Recent Research Related to Cenosphere Characterization ............. 12
    1.4.4 Review of Research Conducted at UNLV Related to the 2Phase State Change Material ........................................................................................................................ 13
  1.5 Objectives .............................................................................................................. 17

**CHAPTER 2 EXPERIMENTAL** ...................................................................................... 19
  2.1 Overview of Experimental Work ............................................................................. 19
  2.2 Test Specimen Preparation ...................................................................................... 20
    2.2.1 Overview of Test Specimen Manufacturing ..................................................... 20
    2.2.2 Test Specimen Manufacturing ........................................................................ 21
    2.2.3 Test Specimen Preparation for Testing............................................................ 34
  2.3 Uniaxial Compression Characterization of the Tooling Material ............................ 37
    2.3.1 Material Testing System (MTS) ....................................................................... 37
      2.3.1.1 Strain Measurement Devices ...................................................................... 43
    2.3.2 Uniaxial Compression Test Procedures ........................................................... 45
    2.4 Uniaxial Compression Creep Characterization of the Tooling Material ............... 50

**CHAPTER 3 RESULTS AND DISCUSSION** .................................................................. 58
  3.1 Uniaxial Compression Results ................................................................................ 58
    3.1.1 Room Temperature Compression Results ....................................................... 58
    3.1.2 Elevated Temperature (177°C) Compression Results ........................................ 62
LIST OF FIGURES

Figure 1-1  a) Toolbed ready for use, b) Part master about to be copied, c) Hardened toolbed, d) Fabricated part from toolbed ...................................................... 3
Figure 1-2  Mandrel of state-change material for water-soluble tooling ......................... 4
Figure 2-1  Solid model rendering of Teflon mold (left) and actual machined part (right) .......................................................... 22
Figure 2-2  Teflon mold and steel plate before joining (left) and after (right) ............... 23
Figure 2-3  Teflon mold filled with a slurry of the liquid state change material .......... 25
Figure 2-4  Teflon mold covered with a layer of peel ply fabric .................................. 26
Figure 2-5  Vacuum bagged Teflon mold before (left) and after (right) the addition of bleeder/breather cloth ........................................................................... 28
Figure 2-6  Completed specimen molding vacuum bag setup ..................................... 29
Figure 2-7  Flow chart describing test specimen manufacturing setup ......................... 30
Figure 2-8  Actual laboratory setup for specimen manufacturing ............................... 31
Figure 2-9  Teflon mold with hardened state change material .................................... 32
Figure 2-10 Test specimen removal from the Teflon mold with a hand press .............. 33
Figure 2-11 Test specimens immediately after removal from the Teflon mold ............ 34
Figure 2-12 A cylindrical test specimen being sanded during the specimen preparation process ......................................................................................... 35
Figure 2-13 Test specimens after sanding .................................................................... 36
Figure 2-14 Strain gaged test specimens ready for testing .......................................... 37
Figure 2-15 Schematic of the MTS machine’s primary components ............................ 38
Figure 2-16 Diagram of the MTS machine’s load frame ............................................. 39
Figure 2-17 Schematic of pod describing its manual functions ................................. 40
Figure 2-18 The MTS machine’s grips as they are used to hold compression platens in place ........................................................................................................ 41
Figure 2-19 The MTS machine with the attached furnace ........................................... 42
Figure 2-20 Comparison of strain measurement devices ............................................. 44
Figure 2-21 Solid model depiction of the compression test fixtures ........................... 46
Figure 2-22 Compression fixtures, after machining, in the experimental setup .......... 46
Figure 2-23 Elevated temperature Experimental setup .............................................. 48
Figure 2-24 Depiction of determination of modulus from the initial linear portion of a stress-strain curve ........................................................................... 50
Figure 2-25 Solid model depiction of a creep fixture in the testing configuration ........ 54
Figure 3-1  Linear portion of an engineering stress versus axial curve for the 20% binder material test group ................................................................. 60
Figure 3-2  Linear portion of an engineering stress versus axial curve for the 77.7% binder material test group ................................................................. 60
LIST OF TABLES

Table 1-1 Properties for the baseline state-change tooling material ......................... 14
Table 1-2 Flexural properties found tooling material with various amounts of binder 15
Table 2-1 Test material recipes for the water soluble tooling and RTS applications ... 24
Table 2-2 Working specification of the MTS machine .............................................. 38
Table 2-3 Test matrix describing the completed compression tests ........................... 48
Table 2-4 Test matrix describing the completed creep tests ..................................... 52
Table 2-5 Actual stiffness values of the eight springs used for testing ..................... 55
Table 3-1 Room temperature compressive properties for two formulations of the 2Phase state change material ................................................................. 59
Table 3-2 Elevated temperature (177°C) compressive properties for two formulations of the 2Phase state change material ......................................................... 63
Table 3-3 Summary of compressive properties ......................................................... 69
Table 3-4 Room temperature creep properties for two formulations of the 2Phase state change material ................................................................. 74
Table 3-5 Elevated temperature (177°C) creep deformation for two formulations of the 2Phase state change material after 4 hours ........................................ 77
Table 3-6 Summary of room temperature creep deformations ................................. 79
Table 3-7 Summary of elevated temperature (177°C) creep deformation ................ 79
Table 4-1 Compressive properties of the 2Phase state change material .................... 87
Table 4-2 Creep deformation rates of the 2Phase state change material ................... 88
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CHAPTER 1

INTRODUCTION

There is a growing interest in composite materials. Composite materials, which are characterized by superior strength-to-weight ratios when compared to metals, lend themselves well to applications that require lightweight building materials. Several industries, including aerospace, automotive, and sports, have started moving toward the implementation of composites on a large scale. With the increasing applications for composite materials, there is a growing demand for alternatives to metallic tooling. Metallic tooling is expensive and often requires sophisticated and lengthy machining. Therefore, there is a need for tooling alternatives that are both inexpensive and that will produce prototypes quickly. However, aside from the low cost and time criteria, a good tooling material alternative needs to have good mechanical properties at both ambient and elevated temperatures. The tooling material should also be dimensionally stable. The 2Phase RTS and state-change material are intended to be inexpensive and less time consuming alternatives to the machining of metallic molds.

1.1 The 2Phase Reconfigurable Tooling System (RTS)

A reconfigurable tooling system has been created by 2Phase Technologies Inc. for use in the quick fabrication of molded composite and liquid polymer prototypes. With the
system, a low-production rate component can be created quickly and economically, and then the system can be reconfigured to allow for the fabrication of a different component.

The system utilizes a state-change material. The material consists of hollow glass microspheres in a slurry of water based binder and water. This slurry solution is enclosed within a metal frame with flexible silicon membranes on the top and bottom. The process for making a mold with the RTS is as follows: a master part is placed onto the top membrane and held in place with a vacuum cap. The vacuum cap uses vacuum to force the top silicon membrane and liquid tooling material to conform around the part master. Next, vacuum is applied to the liquid tooling material to remove excess liquid and to harden the tool. At this point the master part is removed and heat is applied to the semi-hard tooling material to produce a hard, stiff tool that can be used for making composite or polymer components. The resulting tool can be used repeatedly, but once it is no longer needed for part fabrication, moisture can be reintroduced into the tooling material, allowing the material to return to the original, liquid state. The process can now begin again, and a new mold can be made. Figure 1-1 shows this process at the laboratory scale [1].
1.2 The 2Phase State Change Material as Water Soluble Tooling

The 2Phase state-change material is useful for additional applications other than the RTS. The material, given its ability to transform from a liquid to a solid and then back to a liquid, may be functional for water-soluble tooling applications. Specifically, 2Phase Technologies, Inc. is investigating the material usefulness in the development of removable mandrels for the manufacturing of filament wound pressure vessels.

To create a mandrel shaped tool, a method similar to the one used to create a tool in the RTS would be used. While the RTS requires a male mold for a part master, the creation of a water-soluble tool would require a female mold that captures the complete outer shape of the desired mandrel. The liquid state 2Phase material would be positioned into the female mold. Heat and vacuum would then be applied to remove the excess binder solution and rigidize the tool. In this way, the female mold replaces the flexible
silicon membranes used by the RTS during the tool hardening process. Once removed from the female mold, the hardened tool would then be coated with additional layers of the water based binder. Coating the tool prior to composite lay up works to prevent resin infiltration into the hardened state-change material, as it is porous when in its solid state. Figure 1-2 shows a test mandrel created with the method described above.

![Figure 1-2: Mandrel of state-change material for water-soluble tooling](image)

After coating the material, composite lay up or filament winding could occur about the mandrel. Upon completion of the required composite cure cycle, the introduction of liquid to the tool would cause it to effectively dissolve out.

1.3 The 2Phase State Change Material

The state change material created by 2Phase Technologies Inc. consists of a liquid particulate mixture that can be transformed from a formable/liquid state to a force-resisting/solid state, and then back to a liquid formable state.

The particulate component of the tooling material consists of hollow glass microspheres. The specific microspheres used for this study were Sphere One
Extendospheres™ SLG Hollow Glass Spheres [2]. These spheres undergo a proprietary washing process prior to distribution. The microspheres, which occur in the fly ash byproduct of coal manufacturing, need to be cleaned prior to their introduction into the RTS to promote good adhesion with the water based binder. The microspheres range in diameter from 100 microns to 250 microns. This size range tends to result in superior binder flow around the microspheres as the material transitions from the liquid state to the solid state. Adequate binder flow results in a well-consolidated material and superior mechanical properties [3].

The liquid component of the tooling material is a solution of water and a water-soluble refractory binder. The specific variety of binder used for this study is proprietary. While in the liquid state, the binder acts to lubricate the spheres and, when the material is in its solid state, the binder acts as the adhesive holding the tool together. Since the binder is water based and chemical alterations undergone by the material during the hardening process do not affect its water solubility, returning the water component to the solid mixture can dissolve it.

By varying the percentage of binder in the liquid component solution, the state-change material's dissolvability changes. Specifically, the material tends to dissolve and transition from the solid state to the liquid state more effectively when lower percentages of binder are used. Therefore, when the state-change material is being used in the RTS tool bed, the binder accounts for 77.7% of the solution by volume. However, when the material is used as a water-soluble tooling material, the binder accounts for only 20% of the solution by volume [4].
1.4 Literature Review

In order to develop sound methods and to learn of recent research regarding materials similar to the 2Phase state-change material, a comprehensive literature review took place. The reviewed references dealt with four major categories of material: fly ash concrete, syntactic foams, cenospheres, and the 2Phase material itself. Fly ash concrete is relevant as its behavior helps quantify the effect of the fly ash on the creep and compressive properties of concrete. A review of syntactic foam is appropriate for this study, as syntactic foams resemble the 2Phase state-change material physically. Cenospheres, or glass micropsheres, comprise the majority of the state-change material's volume. Therefore, a review of cenosphere properties is fitting. Also, several previous studies have taken place to characterize and determine the mechanical properties of the 2Phase material itself. An examination of those studies is suitable.

1.4.1 Review of Recent Research Related to Fly Ash Concrete

Fly ash particles, or glass microspheres, are often added to concrete to improve its strength and dimensional stability. When mixing concrete, the fly ash is used as a Portland cement replacement. Several studies have been completed recently to determine the exact effect of the addition of fly ash on the performance of concrete.

Alexander, et al. [5] conducted a study in which both regular concrete and concrete with the inclusion of fly ash were manufactured. The investigators evaluated the effect of the fly ash on the room temperature creep behavior of the material. It was found that after two years, the creep of the regular concrete was 50% greater than the fly ash concrete. The study also indicated that lowering the cure temperature of the concrete increases the creep behavior.
Craft, et al. [6] carried out a study in which polymer concrete was made with and without fly ash. The material was thermally cycled to temperatures ranging from -10°C to 60°C and tested at ambient temperatures to determine several mechanical properties, including compressive strength and creep. The investigators determined that the use of fly ash enhances the compressive strength and decreases the magnitude of creep. In general, the examiners found that the higher the thermal cycle temperature, the lower the compressive properties. The compressive strength decreased by as much as 50%, however the fly ash polymer concrete displayed better properties than the plain polymer concrete. Furthermore, the investigators determined that this decrease in compressive strength was due to a decrease in strength of the resin binder and, hence, the bond in the aggregate.

Jo, et al. [7] performed an investigation in which polymer concrete was made with and without fly ash. The resin binder used was polyester obtained by chemically modifying recycled PET (polyethylene terephthalate). The investigators conducted short-term creep tests in which compressive loads were applied to specimens for a 24-hour duration at elevated temperatures. It was found that the creep of the concrete without fly ash was 30-40% higher than the fly ash concrete. The investigators determined that this was due to the fly ash decreasing the amount of resin per unit volume of concrete. The primary cause of creep in the polymer concrete is due to the polyester binder, by decreasing its amount per unit volume, the creep is lowered.

Al-Manaseer, et al. [8] carried out a study in which the room temperature creep behavior of fly ash concrete was investigated. Test specimens were tested in a compressive configuration, and six different load levels were applied, corresponding to
10% to 60% of the compressive strength. It was determined that a linear relationship exists between the magnitude of the load level and the amount of resulting creep. Also, it was found that the creep of the fly ash concrete was less than that of plain concrete for all load levels.

Al-Manaseer, et al. [9] conducted another study in which the room temperature creep properties were determined for fly ash concrete that was made with and without superplasticizer. Tests specimens were tested in a compressive configuration and six different load levels were applied, corresponding to 10% to 60% of the compressive strength. It was determined that a linear relationship exists between the magnitude of the load level and the amount of resulting creep. The addition of super-plasticizer does not seem to affect the shape of the creep curve, however the magnitudes of the creep strains were slightly higher for the concrete made with the plasticizer.

Al-Manaseer, et al. [10] conducted an additional study in which the high temperature creep behavior of fly ash concrete was investigated. Test temperatures ranged from 21°C to 177°C and three different static load levels were applied. The investigators determined that for temperatures up to 177°C, there is a linear relationship between the magnitude of the applied load and the amount of encountered creep. After 28 days of testing, it was found that the regular concrete tended to creep an average of 15% more than the fly ash concrete during the lower temperature tests. However, during the tests with temperatures in excess of 121°C, the plain concrete tended to creep 27% less than the fly ash concrete. After 112 days of testing, it was found that the fly ash concrete and the plain concrete had nearly the same amount of creep at all test temperatures.
Han, et al. [11] carried out an investigation in which the compressive strength of fly ash concrete were determined and compared to the compressive behavior of plain concrete. The compressive strength was determined both experimentally and with prediction models. The investigators determined that the early stage compressive strength was lower than the long-term compressive strength for the fly ash concrete, indicating that the inclusion of fly ash in concrete tends to increase the compressive properties in the long-term.

Malhotra, et al. [12] conducted a study in which concrete was manufactured with and without fly ash and then tested for compressive strength and creep. The investigators found that the fly ash concrete had higher compressive strengths than the plain concrete, and that the rate of strength development was greater for the fly ash concrete. Also, it was determined that the creep strains for the fly ash concrete specimens were significantly lower than the plain concrete, as much as five times less.

1.4.2 Review of Recent Research Related to Syntactic Foams

Syntactic foams are a type of composite structure manufactured by filling polymer matrix materials with hollow glass microspheres. Recent research has taken place to determine the thermal and mechanical properties of certain syntactic foams.

Pleshkov, et al. [13] conducted a study in which investigators determined the suitability of syntactic foams made with glass microspheres as a sandwich panel coring material. Coring materials that display better strength and elasticity tend to promote simultaneous failure of the sandwich panel skins and core. In the study, two types of sandwich panels were constructed, one with a polyurethane foam core and one with a syntactic foam core, and tested in flexure. The investigators determined that syntactic
foams have better shear properties than conventional coring materials. The polyurethane foam core panel failed prematurely due to shear failure in the foam. Failure of the syntactic foam occurred simultaneously with the skin failure. The investigators concluded that while syntactic foams have higher densities, they allow for better strength and rigidity in sandwich structures.

Karthikeyan, et al. [14] determined the compressive properties of syntactic foams manufactured with and without the inclusion of E-glass fibers. The investigators determined that voids are more prevalent in reinforced syntactic foams. It was found that while the strength of the unreinforced foam was greater than the reinforced foam, the elastic moduli were similar. During the tests, the specimens were found to behave linearly until a point of initial failure at which point there would be a slight decrease in load, followed by crushing and material densification. Also, the investigators determined that the primary modes of compressive failure were microsphere debonding and crack initiation at material voids.

Karthikeyan [15] conducted an additional study in which the flexural properties of syntactic foams manufactured with and without the inclusion of E-glass fibers were determined. As before, the investigators found voids to be more prevalent in the reinforced foams. However, the reinforced foams tended to display better flexural properties than the plain foam. Although voids were present, it was found that the fibers increased the surface area for binder adhesion. The flexural specimens tended to behave linearly until a point of global specimen, catastrophic failure. Also, failure tended to initiate on the tensile side of the specimens due to sphere debonding and resin binder fracture.
Gupta, et al. [16] conducted a study in which the compressive properties were determined for syntactic foam made with a range of microsphere sizes. Foams were made with five different sizes of spheres. It was determined that spheres of differing sizes have differing strengths. The compression specimens behaved linearly until a point of initial failure, followed by a slight decrease in stress and material densification. The investigators observed no definite failure point. This is due to the fact the all cenospheres have slightly different radii and tend to fail at slightly different loads when their individual strengths are reached. Also, it was determined that the compressive properties tend to increase as the sphere wall thickness increases.

Gupta, et al. [17] carried out an additional investigation in which the affect of specimen aspect ratio on the compressive properties of syntactic foams was determined. Four different aspect ratios were tested. For aspect ratios greater than 0.6, the material behaved elastically followed by a decrease in stress and material densification. For aspect ratios less than 0.6, no clear yield point existed. The investigators determined that higher aspect ratios tended to result in higher compressive properties, however shear failure was more prevalent with the higher aspect ratios. It was concluded that lower aspect ratios, in the range of 0.6 to 0.67, would show no shear effects and would be more stable when loaded in compression.

Kishore, et al. [18] investigated the room temperature tensile properties of gradient density syntactic foams. When manufacturing the foams, the resin binder, which is denser than the spheres, was allowed to settle. Specimens were cut from the material at each level of varying density. It was found that the tensile properties increased as the microballoon content decreased. Specifically, the modulus was found to increase linearly
from 2.0 GPa to 2.47 GPa, with decreasing microballoon content. A similar trend was also found for the tensile strength.

1.4.3 Review of Recent Research Related to Cenosphere Characterization

Bartake, et al. [19] developed a method to easily determine the crushing strength of cenospheres. In this study, three sizes of cenospheres were tested, and the spheres underwent washing and drying prior to testing. The test apparatus consisted of a hollow stainless steel cylinder with stainless steel pistons on the top and bottom. The bottom piston was fixed, and its position could be adjusted vertically. The top piston moved vertically up and down to crush the glass spheres. By varying the height of the bottom piston, the investigators were able to determine the effect of aspect ratio, or the ratio of height to diameter, on the cenosphere crushing strength. The investigators concluded aspect ratios less than 1.0 result in non-linear crushing behavior, and aspect ratios greater than 1.0 should be used when determining the crush strength of glass microspheres. Also, the investigators determined the crushing behavior of the spheres to be linear, followed by rearranging and repacking of the spheres, followed by a loss in integrity and general crushing.

Kolay, et al. [20] characterized cenospheres to determine their thermal properties. The cenospheres were found to be thermally stable up to 280°C. The investigators determined the material's thermal degradation and mass loss at high temperatures with thermogravimetric analysis (TGA). It was concluded that after 300°C, test samples undergo a 90% weight loss. This reduction in mass is due to the expulsion of the air trapped within the cenospheres. Also, differential thermal analysis (DTA) techniques were used to determine the enthalpy changes in the material. The investigators
determined that there is an endothermic peak around 330°C. This peak is due to the removal of the water that is dissolved in the glassy material.

Bratt, et al. [21] conducted a study in which glass microspheres were characterized in isostatic and uniaxial compression. During the isostatic tests, individual microspheres were placed in a pressure chamber and pressurized hydraulically until failure occurred. During the uniaxial compression tests, individual microspheres were placed on the flat tip of a conical rod and advanced against a flat platen until failure occurred. The investigators observed that while the outer shapes of the tested microspheres were fairly consistent, wall thicknesses were not. Also, several microspheres tested contained voids in the sphere walls, which create stress concentrations and premature failures. The investigators determined from the isostatic tests that better sphere mechanical properties correlate to higher modulus values. Also, from the uniaxial tests, the investigators found the failure force to be related to the sphere wall thickness.

1.4.4 Review of Recent Research Conducted at UNLV Related to the 2Phase State-Change Material

Several previous studies have taken place at UNLV to determine the properties of the RTS material in its hardened state. The solid-state properties of the RTS material are related to the properties of the glass microspheres, the binder, and the interface between them.

Nelson, et al. [1] conducted an experimental study to determine the baseline thermal-mechanical properties of the reconfigurable tooling system material. A summary of this data is presented in Table 1-1. The material behaves in a similar manner to other porous materials. When loaded in uniaxial compression, it responds in a linear elastic manner
until it reaches an initial peak compression value and then crushes non-uniformly as the material begins to densify and fail. The material responds in a linear elastic manner up to a brittle failure point when loaded in flexure. It also has a low thermal conductivity and thermal expansion coefficient.

Table 1-1: Properties for the baseline state-change tooling material

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Compressive Strength</td>
<td>7.63 MPa</td>
</tr>
<tr>
<td>Flexural Strength</td>
<td>3.94 MPa</td>
</tr>
<tr>
<td>Compressive Modulus</td>
<td>2.85 GPa</td>
</tr>
<tr>
<td>Flexural Modulus</td>
<td>3.18 GPa</td>
</tr>
<tr>
<td>Thermal Conductivity</td>
<td>0.123 W/mK</td>
</tr>
<tr>
<td>Coefficient of Thermal Expansion</td>
<td>5.70 E-06 1/K</td>
</tr>
</tbody>
</table>

In a similar study, O'Toole, et al. [22] determined that test material created in an environment that simulates the RTS tool bed environment tends to exhibit properties similar to the properties of a full-size RTS. Material for thermal-mechanical testing was created in small batches. 2Phase material in its liquid state was poured into Teflon coated pans and cured in a furnace under heat and vacuum. The material properties determined from this study were found to be in good agreement with the thermal-mechanical properties determined by Nelson, et al.

O'Toole, et al. [23] conducted an experimental study to determine preliminary flexural and density properties for the hardened RTS material with variation in the level of binder concentration. In this study, test specimens were composed with five binder
percentages (20, 40, 50, 60, or 65 %), measured for density, and tested in four-point flexure. It was found that, generally, the flexural and density properties decrease as the percentage binder decreases. However, the three highest concentration of binder yielded the best results, but were similar to each other. This indicates that with concentrations of binder in excess of 50%, little variations in the mechanical properties should be seen. The results from this study are summarized in table 1-2. Note that “± XX%” indicates the standard deviation.

Table 1-2: Flexural properties found tooling material with various amounts of binder [4]

<table>
<thead>
<tr>
<th>Binder %</th>
<th>Flexural Strength (MPa)</th>
<th>Flexural Modulus (GPa)</th>
<th>Density (g/cc)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>1.70 ± 26%</td>
<td>2.11 ± 4%</td>
<td>0.38 ± 3%</td>
</tr>
<tr>
<td>40</td>
<td>1.71 ± 0%</td>
<td>2.05 ± 0%</td>
<td>0.39 ± 0%</td>
</tr>
<tr>
<td>50</td>
<td>2.29 ± 12%</td>
<td>2.80 ± 7%</td>
<td>0.41 ± 2%</td>
</tr>
<tr>
<td>60</td>
<td>2.01 ± 15%</td>
<td>2.71 ± 3%</td>
<td>0.41 ± 0.1%</td>
</tr>
<tr>
<td>65</td>
<td>2.32 ± 5%</td>
<td>2.66 ± 3%</td>
<td>0.46 ± 1%</td>
</tr>
</tbody>
</table>

O'Toole, et al. [24] conducted another experimental study in which the flexural properties of the hardened state-change material were determined at temperatures as high as 400°C. In this study, flexural specimens were tested in a furnace at 400°C. The specimens tended to undergo significant decreases in flexural strength compared to specimens tested at room temperature.
In an additional study related to the thermomechanical characterization of the state change material, Rahman [25] determined many different material properties, including the coefficient of linear thermal expansion (CTE) and the behavior of the state change material’s constituent components. Rahman found that the state change material’s CTE did not vary significantly with differing binder concentrations. The CTE value was also found to be consistent for all levels of specimen heat treatment resulting in an average value of 5.67 ppm per degree K. Rahman also characterized the microspheres and binder material separately. In his study of the proprietary binder, thin films of binder were observed to foam and increase several times in volume when exposed to temperatures in excess of 66°C. The binder material’s total elongation was observed to be small, and occasionally negative, when exposed to temperatures up to 145°C. However, when exposed to temperatures in the range of 200°C, elongations as high as 850% were often encountered. Furthermore, it was found that the binder’s rate of elongation increased significantly with increasing heating rates and binder film thickness. Rahman concluded that the average CTE value associated with the state change material is a function of the binder material’s elongation behavior.

The references reviewed in this survey demonstrate the challenges present in characterizing materials similar to the state change material. Standard methods for testing the 2Phase material are required to ensure confidence in the resulting data. However, current ASTM standards do not account for the uniqueness of the newly developed 2Phase state-change material.
1.5 Objectives

As mentioned, alternatives to metallic tooling materials are becoming a necessity. However, an alternative material needs to be strong enough to withstand the forces and pressures associated with various composite manufacturing techniques. The material should also be dimensionally stable to preserve part tolerances. In the manufacturing of modern composites, pressures in the range of 0.101 MPa are commonly encountered. Also, depending on the matrix material, curing temperatures in the range of 177°C to 400°C are often encountered. The term “curing” when used in regards to composites manufacturing, refers to the chemical reactions that take place to harden a resin system from a liquid state to a cross-linked solid structure. Very few non-metallic tooling materials are capable of withstanding exposure to elevated temperatures without deterioration and significant dimensional changes. The 2Phase state change material has been developed as an alternative to metallic tooling materials. The constituent components of the state change material are inexpensive to acquire and relatively easy to assemble into a usable composite tool. However, the material must be investigated to determine its suitability to tooling applications in terms of its strength and dimensional stability. Therefore, the main objectives of this thesis are:

- Determine the properties and behavior of the 2Phase state change material when loaded in uniaxial compressions at ambient and elevated (177°C) temperatures.

- Determine the creep behavior of the 2Phase state change material when subjected to prolonged uniaxial compressive loads at ambient and elevated (177°C) temperature.
• Determine the 2Phase state change material's suitability as a tooling material by conducting the above mentioned tests for material compositions corresponding to RTS tool bed use and water soluble tooling use.
CHAPTER 2

EXPERIMENTAL

2.1 Overview of Experimental Work

The comprehensive literature review presented in the previous chapter provided an overview of recent research related to the characterization of fly ash concretes, syntactic foams, cenospheres or glass microspheres, and the 2Phase state change material itself. The presented studies indicate two major characterization test types as potentially relevant in the determination of the 2Phase state change material’s appropriateness to reformable and water soluble tooling applications.

First, prevalent in the reviewed investigations was uniaxial compressive testing at both ambient and elevated temperatures. Characterization tests of this type will yield such useful information as the state change material’s compressive strength and compressive modulus. As previously mentioned, a tooling material, as an alternative to metallic molding materials, must have good mechanical properties. Through the determination of compressive strength and compressive modulus, comparisons can be drawn between the state change material’s mechanical properties and the properties of metallic materials commonly used for tooling application (Invar and Aluminum).

Next, as mentioned in the previous chapter, tooling materials for composite manufacturing applications should be dimensionally stable at both ambient and elevated
temperatures. A type of characterization test that provides good measure of a material’s dimensional stability is creep testing. Several of the referenced investigations conducted both long and short-term creep tests at both ambient and elevated temperatures, providing sound methods for the determination of the 2Phase state change material’s creep behavior. Creep can be defined as deformation undergone by a material due to the influence of statically applied stresses and, when testing to determine a material’s creep behavior, several useful properties can be found. In this study, the properties of greatest interest are the magnitude and rate of creep encountered due to prolonged exposure to statically compressive loads.

2.2 Test Specimen Preparation

2.2.1 Overview of Test Specimen Manufacturing

In an experimental study conducted by O’Toole, et al. [19], the investigators determined that state change material that is manufactured under a set of circumstances mimicking the 2Phase Reformable Tooling System’s environment will behave similarly, and exhibit mechanical properties comparable to, material taken from the RTS tool bed itself. The researchers from that study also determined that state change material taken from various regions of an RTS tool bed tends to not vary significantly in mechanical characteristics. Therefore, when preparing uniaxial compression and creep test specimens for this thesis study, two assumptions were made. First, since state change material that is manufactured outside of the RTS in an atmosphere imitating the reformable tooling system environment behaves similarly to material taken from an actual tool bed, it was deemed unnecessary to manufacture test specimens in an actual RTS tool bed. Second,
since O'Toole, et al. also showed that mechanical properties do not vary significantly by tool bed region, it was concluded that molding specimens into net shapes would not have a significant effect on the uniaxial compressive and creep behavior.

Specimens manufactured for both the uniaxial compression and creep tests were cylindrical in shape. For both test types, the cylinders measured approximately 50.8 mm in height and approximately 25.4 mm in diameter. These dimensions were chosen such that during compressive loading of the test specimens, a height-to-diameter (h/D) ratio of approximately 2:1 would be attained. This h/D ratio was chosen for two primary reasons. First, height-to-diameter ratios much greater than 2:1 tend to result in specimen failures due to buckling. When a failure due to buckling occurs, the failure load is related to the bending moment experienced by the test specimen and will, therefore, generally be of a lower magnitude than that which would have been encountered if the specimen failure was due to the uniaxial compressive loading alone. Next, compression test specimens with height-to-diameter ratios much less than 2:1 often do not clearly show a yield point. Rather, when loaded in uniaxial compression, tests specimens with low aspect ratios often allow for a constant increase in stress with no clear failure point.

2.2.2 Test Specimen Manufacturing

The 2:1 h/D cylindrical test specimens for the uniaxial compression and creep tests were created in a Teflon mold. Teflon, or polytetrafluoroethylene (PTFE), is a synthetic fluoropolymer that lends itself well to non-stick applications. Water and water containing substances do not affect PTFE and, for this reason, adhesion to Teflon is difficult and generally prevented. PTFE is also very non-reactive and tends to reduce wear. For these reasons Teflon was deemed ideal for molding the 2Phase state change material. A PTFE
mold would not react with water based binder solution, would resist significant wear after multiple uses, and would allow for easy removal of specimens from the mold.

The mold was created from a 50.8 mm thick sheet of Teflon having both a length and width of 152.4 mm. A pattern of 25.4 mm diameter through holes was created in the thickness direction of the sheet using a VF5 Haas CNC mill. A 6.4 mm tolerance, or spacing, was accorded between each hole and between any outer hole and the edge of the sheet. Due to the outer dimension of the sheet and the allotted tolerances, it was determined that a maximum of 23 through holes could be made to fit in the purchased piece of Teflon. The pattern was modeled into the Teflon sheet using the parametric solid modeling software ProEngineer Wildfire v3.0 and then transferred into MasterCAM v9.0 in an IGES format to develop the G code prior to machining. Figure 2-1 shows the ProEngineer solid model of the mold and the finished mold upon completion of machining in the CNC mill.

Figure 2-1: Solid model rendering of Teflon mold (left) and actual machined part (right)
A standard method was developed for manufacturing the cylindrical test specimens with the Teflon mold upon completion of its machining. To ensure that the bottom surfaces of the molded test specimens would be approximately horizontal and level, a flat and even piece of low carbon, 1020 steel sheet was affixed to the bottom of the mold prior to molding test specimens. The steel sheet had a thickness of 3.2 mm and the length and width both measured 152.4 mm. The 3.2 mm thickness measurement was chosen to ensure the steel plate remained rigid once subjected to heat and vacuum forces. Also, the length and width measurements of the steel plate were chosen such that they would match the length and width dimensions of the Teflon mold. A high temperature polyester masking tape with a 50.8 mm width was used to rigidly affix the steel plate to the bottom of the Teflon mold. The polyester tape also helped to create an air and liquid tight seal between the 1020 steel plate and the bottom of the Teflon mold. Figure 2-2 shows the Teflon mold and steel plate both before and after their joining.

![Figure 2-2: Teflon mold and steel plate before joining (left) and after (right)](image-url)
Immediately after attaching the 1020 steel plate to the Teflon mold, all of the mold’s surfaces were sprayed with a dry film PTFE mold release. The use of a PTFE mold release was found to aid in the removal of the test specimens from the mold and, since PTFE tends to reduce wear, help prolong the life of the mold. Then, a liquid slurry of the state change material was prepared. Much experimentation occurred to determine the proper ratio of glass microspheres-to-binder solution to achieve a consistency similar to that of the material within the reformable tooling system tool bed. However, it was eventually determined that an approximate microsphere-to-binder solution ratio of 3:2 was appropriate for the test specimen molding process. Given the size and volume of the Teflon mold, a suitable amount of state change material could be achieved by mixing 600 ml of glass microspheres with 400 ml of binder solution. Table 2-1 shows the state change material recipes used in creating the liquid slurries for the both the 20% and 77.7% binder formulae for the water soluble tooling and RTS tool bed applications, respectively.

Table 2-1: Test material recipes for the water soluble tooling and RTS applications

<table>
<thead>
<tr>
<th></th>
<th>20% Binder Formula</th>
<th>77.7% Binder Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>Binder Volume in Solution</td>
<td>80 ml</td>
<td>311 ml</td>
</tr>
<tr>
<td>Water Volume in Solution</td>
<td>320 ml</td>
<td>89 ml</td>
</tr>
<tr>
<td>Volume of Glass Spheres</td>
<td>600 ml</td>
<td>600 ml</td>
</tr>
</tbody>
</table>
When creating the liquid slurry of 2Phase state change material, all components (binder, water, and glass microspheres) were measured with a graduated cylinder to ensure accuracy. The three components of the slurry were mixed by hand in a container until consistent, and then the container was vibrated to remove excess air. Once adequately mixed, the state change material was poured into the Teflon mold. An offset spatula was used to ensure that each of the mold’s individual 25.4 mm holes were filled with slurry to the top and that the liquid level was even with the upper surface of the Teflon mold. Once filled, the Teflon mold was vibrated by hand to help the material to settle into the mold and to remove voids of excess air. Additional slurry material was added to any of the mold’s holes in which the material level decreased due to settling. Figure 2-3 shows the mold filled with the liquid slurry of state change material.

Figure 2-3: Teflon mold filled with a slurry of the liquid state change material
After filling the Teflon mold with state change material, a layer of peel ply was affixed to the mold's top. Peel ply is a type of tightly woven fabric, often made of nylon, which allows the binder to flow through and escape when vacuum is applied. However, because of the fabric's tight weave, it is impermeable to the glass microspheres. The use of peel ply was essential as vacuum pressure would have been lost and the vacuum pump would have sustained damage if glass microspheres were pulled through the vacuum lines into the vacuum pump. The peel ply was affixed to the mold's top surface with the same high temperature polyester tape used to join the 1020 steel plate with the bottom of the mold. Figure 2-4 shows the Teflon mold with the attached peel ply.

Figure 2-4: Teflon mold covered with a layer of peel ply fabric
After affixing the peel ply to the upper surface of the mold, the Teflon mold was placed inside of a vacuum bag. A vacuum bag is composed of vacuum bagging material and bag sealant tape. Vacuum bag material is a relatively thick, but flexible plastic that is generally capable of withstanding elevated temperatures. The vacuum bagging material used in this study was rated to 204.4°C. It was necessary that the bag be somewhat thick, and also flexible, so that it did not suffer puncturing as it conformed to the sharp corners of the Teflon mold. After being placed inside the bag, the Teflon mold was covered with two layers of bleeder/breather cloth. Bleeder/breather cloth is a thick, felt like material that serves two major purposes. Its first function is to “bleed”, or absorb, the binder solution as it flows through the peel ply and out of the mold when undergoing the state change material hardening process. As it “bleeds” off the excess solution, it provides a path for the liquid to follow to the vacuum lines. The cloth’s second purpose is to “breath” or provide a continuous air path when pulling a vacuum. In the absence of the bleeder/breather cloth, it is possible for the vacuum bag to seal itself to the mold surface at the ports in the vacuum lines, preventing the flow of excess solution from the mold. Figure 2-5 shows that mold within the vacuum bag both before and after the addition of the bleeder/breather cloth layers.
Figure 2-5: Vacuum bagged Teflon mold before (left) and after (right) the addition of bleeder/breather cloth

After the mold was placed inside the vacuum bag and the bleeder/breather cloth was in place, two vacuum lines were placed on top of the bleeder/breather cloth layers. These lines provided the pressure necessary to consolidate the test material into a force resisting state in addition to a path for the excess binder solution to travel through as it exited the mold. The vacuum lines were made of temperature resistant, flexible (yet non-collapsing) plastic tubing. The plastic tubing was prepared by drilling holes in it at regular intervals to create vacuum ports. Then, the prepared vacuum lines were wrapped in bleeder/breather cloth to prevent the vacuum bag from sealing to the ports and stopping the flow of the binder solution. Once the vacuum lines were in place, the bag was sealed and checked for leaks. Figure 2-6 shows the completed specimen molding vacuum bag setup and shows the vacuum lines in detail.
The completed vacuum bag for the test specimen molding process was then placed inside a furnace and subjected to vacuum. To thermally harden the material, the mold was subjected to a temperature of 177°C for 24 hours while vacuum was maintained. To prevent damage to the vacuum pump during the material hardening process, two precautions were put in place. First, the introduction of fluids into the vacuum pump would cause its eventual decomposition. To prevent this, a vacuum trap was placed between the specimen vacuum bag and the vacuum pump. The vacuum trap effectively "traps" all of the liquid binder solution flowing from the mold before it can reach the vacuum pump. Secondly, because the cure temperature of the state change material is well above the boiling point of water, some amount of the solution travels through the vacuum lines as vapor. Since the vacuum trap is only equipped to stop liquids from entering the vacuum pump, a heat exchanger was put in place between the specimen vacuum bag and the vacuum trap. The heat exchanger is of the tube-fin, liquid-air variety and, when equipped with an appropriately sized fan, is capable of removing 3.3 MJ per
hour. This heat exchanger was found to remove the necessary amount of heat energy from the heated solution. Figure 2-7 is a flow chart describing the vacuum lines through which the solution flowed from the specimen-molding vacuum bag to the vacuum pump. Figure 2-8 shows the laboratory set up used in preparing the test specimens.

Figure 2-7: Flow chart describing test specimen manufacturing setup
Upon completion of the test specimen cure cycle, the vacuum pump and furnace were turned off, and the test specimen vacuum bag was allowed to cool inside of the oven. The term “curing,” when used in regards to the 2Phase state change material, refers to the thermal hardening process employed to transform the material from its liquid state to a solid state. Once the test specimen assembly was cool enough to handle without
protective equipment, it was removed from the furnace and the Teflon mold was removed from the vacuum bag. Then, the layers of peel ply, bleeder/breather cloth, and the vacuum lines were separated from the top of the mold. The 1020 steel plate was also removed from the bottom of the Teflon mold at that time. Figure 2-9 shows the Teflon mold prior to the removal of the hardened state change material.

![Image](image.png)

**Figure 2-9: Teflon mold with hardened state change material**

The finished test specimens were then removed from the mold. Although the mold was made of Teflon, and a layer of dry film PTFE mold release was applied prior to the specimens' manufacturing, a large amount of force was required to separate the test cylinders from the mold. Since the test specimens were molded with outer diameters of exactly 25.4 mm, a press fit was created with the holes in the mold. To break the fit and remove the material from the mold, a hand press was utilized. A hand press is a common machine shop device that increases the amount of downward pressure a person can exert.
with mechanical advantage. Figure 2-10 shows the finished test specimens being removed from the Teflon mold with the UNLV Engineering machine shop hand press.

Figure 2-10: Test specimen removal from the Teflon mold with a hand press

Upon removal from the mold, only those test specimens recognized as standard were kept for eventual testing. All others were discarded. To be considered standard, the state change material must not have lost vacuum pressure at any time during the 24-hour cure cycle and the cure temperature must have been 177°C. Once removed from the Teflon mold, the presence of large visible voids at the surface of any test specimen would cause it to be deemed substandard. Additionally, if after a compression or creep test was completed voids were found to be present in the interior of the tests specimen, that specimen would be deemed substandard and another test specimen would be tested in its place. Figure 2-11 shows a set of standard test specimens immediately after removal from the Teflon mold.
2.2.3 Test Specimen Preparation for Testing

The same preparation technique was used for both the specimens prepared for the uniaxial compression tests and the uniaxial creep tests. First, it was required that the top and bottom surfaces of the test cylinders be checked for parallelism. This step was essential, as nonparallel surfaces would result in eccentric loading and premature specimen failures during the compression and creep tests. As seen in figure 2-11 (above), the test cylinders, immediately after removal from the Teflon mold, did not have parallel faces. To remedy this, the top and bottom surfaces of the test cylinders were lightly sanded with a standard bench top disc sander. During this process, approximately 1.6 mm of material was removed from the overall height of the cylindrical test specimens. Figure 2-12 shows a specimen for testing, as it was prepared with the disc sander.
Figure 2-12: A cylindrical test specimen being sanded during the specimen preparation process

Also, if, during the process of sanding the top and bottom surfaces of the test specimens, interior voids were revealed near the outer faces, those specimens would be deemed substandard and would be discarded. Figure 2-13 shows a group of standard specimens after sanding the top and bottom surfaces to be approximately parallel.
Once the sanding of the top and bottom surfaces of the cylindrical specimens was completed, a strain gage was bonded to all test specimens scheduled for room temperature testing. The measurement of strain in the direction of the applied load was required for the determination of the compressive modulus and the creep characteristics. Strain gages were not bonded to the elevated temperature test specimens as a laser extensometer was used to measure strain during the elevated temperature compression and creep tests. The gages used during the room temperature tests were Vishay type EA-06-250BF-350 and were capable of measuring strain at 350 ohms of resistance with a gage factor of 2.10.

To attach the gages, a method similar to the standard strain gaging process was applied with a few adjustments made for the 2Phase state change material. The test specimens were first lightly brushed to remove any loose microspheres at the surface of the material. Then the gages were bonded to the surfaces of the specimen, in the specimens' gage lengths and in the axial direction. Once the bonding process was complete, lead wires were soldered to the terminals of the strain gages. Prior to the
compression and creep testing, the gages were checked for correct operation with a portable strain conditioning box. Figure 2-14 shows a group of test specimens with strain gages attached, prior to testing.

![Strain gaged test specimens ready for testing](image)

**Figure 2-14: Strain gaged test specimens ready for testing**

2.3 Uniaxial Compression Characterization of the Tooling Material

2.3.1 Material Testing System (MTS)

All testing to determine the uniaxial compression characteristics of the 2Phase state change material took place using the servo-hydraulic MTS Axial/Torsional Material Testing System. The MTS machine consists of several primary components. Figure 2-15 shows a schematic describing the layout of the MTS machine’s primary components. Of specific importance to this study are the machine’s load frame, the control computer and pod, the hydraulic grip supply, the strain conditioning box, and the temperature controller.
The load frame (component #1 in figure 2-15) is the structure that manages the application of loads and the resulting displacements during a test. There are several physical specifications that limit the load frame and they are given in table 2-2. Also, figure 2-16 shows a schematic of the MTS machine’s load frame.

Table 2-2: Working specification of the MTS machine

<table>
<thead>
<tr>
<th>Specification</th>
<th>Limit/Measurement</th>
</tr>
</thead>
<tbody>
<tr>
<td>Working Dimensions</td>
<td>635 mm wide by 1463 mm high</td>
</tr>
<tr>
<td>Axial Load Transducer Limit</td>
<td>250 KN</td>
</tr>
<tr>
<td>Torsional Load Transducer Limit</td>
<td>2200 N-m</td>
</tr>
<tr>
<td>Linear Actuator Limit</td>
<td>± 76 mm, measured with an LVDT</td>
</tr>
<tr>
<td>Rotary Actuator Limit</td>
<td>100° static rotation (± 50°), measured with ADT</td>
</tr>
<tr>
<td></td>
<td>90° dynamic rotation (± 45°)</td>
</tr>
</tbody>
</table>
The MTS machine’s computer (component #2 in figure 2-15) manages the movement of the load frame’s actuators as static loads are applied and acquires useful data (displacement, force, strain, etc.) during tests. This computer is a PC system operating on Windows 2000. The main software program used to operate the MTS, including the direction of tests and the creation of testing templates, is TestStar.

The pod (component #3 in figure 2-15) is the main interface with the system for manually controlling the MTS. Although much of the MTS machine’s control takes place with the computer, use of the pod is essential when fixturing test specimens and when preparing the machine immediately prior to running a test. Figure 2-17 shows a schematic of the pod and describes its functions in detail.
The hydraulic grip supply (component #4 in figure 2-15) controls the gripping pressure used to hold testing fixtures in place throughout the duration of tests. The supply of grip pressure is controlled with a combination of hydraulic fluid and compressed air, and the maximum gripping pressure the supply is capable of is 45 MPa. Figure 2-18 shows the MTS machine’s grips as they are used to hold compression platens in place for a test.
The strain gage conditioning box (component #5 in figure 2-15) is used for the interpretation and conversion of resistance changes across strain gages into microstrain. The strain data, as determined by the strain gage conditioning box, is recorded and displayed on the MTS computer during testing.

The final MTS component of specific interest to this study is the MTS machine’s temperature controller (component #6 in figure 2-15). The temperature controller operates a furnace that is affixed to the load frame of the MTS machine for the purpose of
elevated temperature tests. The maximum temperature that the furnace is capable of is 1000°C. The internal volume of the furnace is small, as it measures only approximately 203.2 mm in height by 127.0 mm in width by 101.6 mm in depth. Therefore, the dimensions of the furnace only allow elevated temperature testing for smaller sized specimens. Figure 2-19 shows the MTS machine’s attached furnace.

Figure 2-19: The MTS machine with the attached furnace
2.3.1.1 Strain Measurement Devices

The MTS machine is capable of measuring strain in four ways. The first method utilizes strain gages. The strain data recorded from the gages is transferred to the MTS computer through a Vishay 2100 strain conditioning box. The second method utilizes a high temperature extensometer. The high temperature extensometer, MTS model number 632.54E-11, consists of two ceramic arms than come into physical contact with the surface of test specimens. As the specimens deform, the arms register the length changes and the deflection data is transferred to the MTS computer through the strain conditioning box. The third method uses an EIR laser extensometer in conjunction with laser reflective tape. When applying the laser tape to the test specimens, the required gage length was approximately 25.4 mm. The fourth method of strain measurement utilizes the LVDT data. Assuming that the deflections recorded from the LVDT accurately represent the axial deformations of the test specimens, its data can be manipulated to determine engineering strain.

Testing was completed at both room temperature and elevated temperatures to determine which strain measurement method best suited the 2Phase state change material. It was established that, for the room temperature compression and creep characterizations, that strain gages would yield the best results. Also, it was found that the laser extensometer would yield the best results for the elevated temperature tests. In both temperature cases, the LVDT was found to be the most inaccurate method. It is likely that the testing fixtures tended to settle once load was applied, resulting in distorted LVDT data. Also, the high temperature extensometer was unable to register the relatively small deformations of the state change material specimens.
Since two different strain measurement techniques were found to be appropriate for testing the material, testing was done to verify that strain data from the two devices would agree. Specifically, several of the cylindrical test specimens manufactured with the 77.7% binder formulation had both strain gages and laser tape attached. The specimens were then tested in compression with the MTS while strain was recorded from the laser extensometer and the strain conditioning box at regular intervals of one second. Upon completion of testing, the data was compared by plotting the strain recorded from the gages versus the strain recorded from the laser extensometer. A representative plot is shown below.

![Comparison of Strain Measuring Devices](image)

Figure 2-20: Comparison of strain measurement devices
As shown in the above plot, the resulting curve in linear in shape with a slope of approximately one and a y-intercept value of approximately zero. This indicates excellent agreement and verifies that the two strain measurement devices agree.

2.3.2 Uniaxial Compression Test Procedures

Uniaxial compression testing was conducted with a method based upon aspects from two ASTM test standards. As the 2Phase state change material is unique in its formulation, no single ASTM test standard was found to be appropriate in defining a compression test method. The resulting “composite standard” combined appropriate preparations, procedures, and analyses from ASTM standards for polymer concretes (C579-01) [26] and rigid cellular plastics (D1621-94) [27].

The test specimens, as described in preceding sections, were approximately 50.8 mm in height and 25.4 mm in diameter. During the preparation of the specimens for testing, although much care was taken to ensure that the loaded ends of the specimens were parallel to each other, special fixturing was required to eliminate any possibility of eccentric loads. As specified by ASTM C579-01, the testing machine was equipped with compression platens with hardened faces. The uppermost platen was spherically seated; so as to conform to minor misalignments between the top and bottom faces of each test specimen. The UNLV Engineering machine shop Haas SL20 CNC lathe was used to manufacture the test platens. Prior to machining, the test fixtures were modeled in the parametric solid modeling software ProEngineer Wildfire v3.0 and then transferred into MasterCAM v9.0 in an IGES format to develop the G code prior to machining. Figure 2-20 shows the ProEngineer solid model of the uniaxial compression test configuration. Also, figure 2-21 shows the machined platens in the experimental set up.
Figure 2-21: Solid model depiction of the compression test fixtures

Figure 2-22: Compression fixtures, after machining, in the experimental setup
As discussed in the previous section, all of the quasi-static uniaxial compression tests were conducted with the MTS machine. A template defining the parameters for testing was created with the MTS software, TestWare/SX, prior to testing. The test template, named High Temp Tensile Test With Controlled Heat Cycle Default Procedure, operated in three stages. First, the test specimens were preloaded to 22.2 N with a crosshead descent rate of 12.7 mm/min. After the preloading segment, the specimens were loaded to the point of failure with a constant crosshead descent rate of 5.08 mm/min, or 2.5 mm/min for every 25.4 mm of specimen height. Upon failure, the crosshead returned to its initial position at a rapid rate, unloading the specimen for removal from the MTS machine. During the template’s second segment, in which the test specimens were loaded to failure, instantaneous force and strain data were recorded at regular intervals of 0.1 seconds. As previously mentioned, strain data was recorded from strain gages during the room temperature tests and from a laser extensometer during the elevated temperature tests.

With the above-described procedure, compression tests were carried out at both ambient and elevate temperatures. Table 2-3 is a matrix that describes the compression tests completed and separates the tests into four types. The room temperature tests utilized the compression platens alone, as shown in figure 2-21, without the use of the MTS machine’s furnace. However, the elevated temperature tests utilized both the platens and the MTS furnace. Figure 2-22 shows the experimental setup used for the elevated temperature tests.

The temperature applied to all of the elevated temperature tests was 177°C. This temperature was used as it corresponds to the curing temperature required to solidify the
Bismaleimide (BMI) resin system expected for use with the state change material in the manufacturing of filament wound pressure vessels. The compression test specimens were conditioned within the MTS machine’s furnace for 40 minutes once the furnace reached a temperature of 177°C to ensure an even temperature distribution throughout the test material. The 40-minute time duration was determined with a set of experiments, in which thermocouples were embedded into the centers of several test specimens.

Table 2-3: Test matrix describing the completed compression tests

<table>
<thead>
<tr>
<th>Uniaxial Compression Test Matrix</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature for Testing</td>
</tr>
<tr>
<td>Binder Volume in Solution</td>
</tr>
<tr>
<td>20%</td>
</tr>
<tr>
<td>77.7%</td>
</tr>
</tbody>
</table>

Figure 2-23: Elevated temperature Experimental setup
Upon completion of the compression testing, the uniaxial compressive strength and the uniaxial compressive modulus were determined. Each test group was composed of five to nine cylindrical test specimens. The compressive properties were determined for each individual specimen within a specific group and then averaged to determine the compressive properties for the group. The uniaxial compressive strength was taken as the largest occurring engineering stress. To determine the engineering stress, the instantaneous load data, as recorded by the MTS machine’s computer, was divided by the test specimen cross-sectional area, as shown in the below equation.

\[
\sigma = \frac{P}{A} = \frac{P}{\pi r^2}
\]

Where:
\( \sigma \) = Instantaneous Engineering Stress
\( P \) = Instantaneous Force
\( r \) = Specimen Radius (~12.7 mm)

To determine the compressive modulus for each of the tested specimens, the engineering stress values, calculated with the above equation, were plotted versus the axial strain values recorded by the MTS computer during the compression testing. The compressive modulus was taken as the slope of the initial linear portion of the resulting engineering stress versus axial strain curve. This principle is demonstrated visually in figure 2-23.
2.4 Uniaxial Compressive Creep Characterization of the Tooling Material

Creep testing was completed to determine the dimensional stability of the 2Phase state change material when prolonged uniaxial compressive loading was applied at ambient and elevated temperatures. The test method used to determine the creep behavior was inspired by, and loosely based upon, the ASTM standard C1181-00 [28], which specifies the standard test method for compressive creep of chemical-resistant polymer machinery grouts. The magnitude of the applied compressive load corresponded to certain percentages of the material’s uniaxial compressive strength. The creep
characterization took place upon the completion of the compressive characterization, as the uniaxial compressive properties were required.

The test specimens used for the uniaxial compressive creep characterization were identical to the test specimens used for the quasi-static uniaxial compression tests. As mentioned in the preceding sections, the test specimens were cylindrical in shape and measured approximately 25.4 mm in diameter by 50.8 mm in height. Also, each specimen had a strain gage bonded in its gage length in the axial direction.

During testing, test specimens were loaded in compression to levels corresponding to either 15% or 30% of the compressive yield strength. These load levels were chosen for two primary reasons. Neville, et al. [29] conducted an extensive review of research related to the influences and factors affecting the creep of concrete. The first conclusion made by the investigators was in regards to the level of applied stress and its proportionality to the magnitude of the resulting creep. Specifically, when creep specimens are loaded in compression, the resulting creep is generally proportional to the stress-strength ratio when the applied load corresponds to 30% of the compressive yield strength or less. Another important conclusion made by the investigators related the applied load level to the likelihood of test specimen fracture. Neville, et al. found that internal cracking of the specimen tends to take place with stress levels greater than 40% of the yield strength. As internal cracking has a significant effect on the resulting creep behavior, specimen fracture should be avoided.

The temperatures and durations chosen for the creep characterization tests corresponded to the required cure cycles of resin systems expected for use with the 2Phase state change material. The ambient temperature creep tests had a total duration of
24 hours. This cycle was meant to mimic a commonly used epoxy resin system that cures at room temperature in 24 to 48 hours. The elevated temperature creep tests took place at 177°C and had a total duration of four hours. This cycle was meant to mimic the requirements for curing the BMI resin, mentioned in the preceding section, expected for use in the manufacturing of filament wound pressure vessels. Table 2-4 is a matrix summarizing the creep tests that took place.

Table 2-4: Test matrix describing the completed creep tests

<table>
<thead>
<tr>
<th>Room Temperature for 24 Hours</th>
<th>Binder %</th>
<th>% of Strength Applied</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>15</td>
<td>30</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>Group 1</td>
</tr>
<tr>
<td></td>
<td>77.7</td>
<td>Group 2</td>
</tr>
</tbody>
</table>

177°C for 4 Hours

<table>
<thead>
<tr>
<th>Binder %</th>
<th>% of Strength Applied</th>
</tr>
</thead>
<tbody>
<tr>
<td>15</td>
<td>30</td>
</tr>
<tr>
<td>20</td>
<td>Group 5</td>
</tr>
<tr>
<td>77.7</td>
<td>Group 6</td>
</tr>
</tbody>
</table>

The test fixtures used to conduct the room temperature creep tests were based upon the recommendations of the ASTM standard C1181-00. A single creep fixture consisted of a compression spring and several loading plates inside of a cylindrical aluminum tube. Individual creep fixtures were assembled in the following way:
• At the base of the cylindrical tube, an aluminum plate, with a cross-section to match the inner diameter of the tube, was rigidly attached.

• A test specimen was placed on top of the rigid plate at its center.

• A loading plate was placed on top of the specimen. The loading plate was free to move vertically up and down inside of the aluminum tube. This allowed for test specimens of varying heights.

• The compression spring was placed on top of the loading plate.

• A plate to control the height of the compression spring was placed on top of the compression spring. The upper half of the aluminum tube was threaded with a pitch of 10 threads per 25.4 mm. The circumferential outer surface of the control plate was machined with a matching thread pattern. By threading the control plate vertically up and down within the tube, the height of the compression spring could be controlled.

Again, the above described fixtures were used only for the room temperature creep tests. Figure 2-24 shows a solid model representation of the creep fixtures in the testing configuration.
The creep inducing compressive load was applied to the test specimens through the compression spring. As shown in the below equation, the force exerted by a spring is proportional to its stiffness and sustained deflection.

\[ P = k\delta \]

Where:

P = Force exerted by spring  
k = Spring stiffness  
\( \delta \) = Spring deflection

Therefore, if the stiffness of a spring is known, by varying the spring's deflection, the force it exerts can be controlled.

A total of eight fixtures were manufactured, so as to allow for the simultaneous testing of multiple creep specimens. The stiffness of the eight compression springs purchased for this study was reported as 20.47 N/mm by the manufacturer. However, confirmation of the reported stiffness value was required and each spring was
experimentally tested to determine its actual stiffness. Table 2-5 summarizes the experimentally determined spring stiffness values. Appendix A gives a detailed description of the experimental processes and equipment used to determine the stiffness values of the compression springs.

Table 2-5: Actual stiffness values of the eight springs used for testing

<table>
<thead>
<tr>
<th>Compression Spring #</th>
<th>Room Temperature Stiffness Value (N/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>21.12</td>
</tr>
<tr>
<td>2</td>
<td>21.52</td>
</tr>
<tr>
<td>3</td>
<td>21.42</td>
</tr>
<tr>
<td>4</td>
<td>21.31</td>
</tr>
<tr>
<td>5</td>
<td>21.27</td>
</tr>
<tr>
<td>6</td>
<td>21.39</td>
</tr>
<tr>
<td>7</td>
<td>21.50</td>
</tr>
<tr>
<td>8</td>
<td>20.93</td>
</tr>
</tbody>
</table>

Upon completion of the spring stiffness verification tests, the creep fixtures were assembled and the ambient temperature creep tests took place. Test specimens were loaded into the fixtures and the compression springs were compressed to load levels corresponding to either 15% or 30% of the compressive yield strength. As previously mentioned, as the thread pitch of the aluminum tube was known, by controlling the
number of revolutions made by the loading plate when threading it into the aluminum tube, the desired compressive load level could be achieved. Once loaded to the correct load level, the apparatuses were left in the testing configuration for 24 hours. Throughout the durations of the tests, strain data was recorded at regular intervals. The data acquisition system was composed of a portable Vishay P3 strain conditioning box connected to a computer via USB. The Vishay P3 conditioning box software allowed for strain readings to be recorded at 10-second intervals and saved the recorded data to text files.

While the compression spring assemblies were used to determine the room temperature creep behavior of the state change material, the MTS machine was again made use of in determining the material’s elevated temperature creep behavior. A template defining the parameters for the creep testing was created with the MTS software, TestWare/SX. The test template operated in two stages. First, the test specimens were loaded in compression to levels corresponding to either 15% or 30% of the compressive strength with a crosshead descent rate of 12.7 mm/min. Then, the applied load was maintained for a total duration of four hours. During the template’s second segment, in which the test specimens were held under the continuous load, instantaneous force and strain data were recorded at regular intervals of 10 seconds. The strain data was recorded from the laser extensometer. Also, these tests took place within the MTS machine’s furnace with a test temperature of 177°C.

Upon completion of the characterization tests, the recorded data was reduced to determine the creep behavior. The first property of interest was the maximum specimen deformation. To determine this property from the recorded strain data for an individual
test specimen, the largest occurring strain value was multiplied by the test specimen's original height. The resulting value would be the total deformation of the specimen in the axial direction. The below equation demonstrates this.

\[ \Delta L = \varepsilon L \]

Where:

\[ \Delta L = \text{Specimen Deformation} \]

\[ \varepsilon = \text{Maximum Strain Reading} \]

\[ L = \text{Original Specimen Height} \]

The second creep property of interest was the rate of creep. To determine this property for an individual test specimen, an instantaneous strain versus time plot would be created. As shown by Al-Manaseer, et al. [8-10], the shape of the resulting curve should be logarithmic in shape. Therefore, by fitting a logarithmic regression to the resulting curve, an equation of the form below would result.

\[ y = A \cdot \ln(x) + B \]

In the above equation, A and B are constants. A is taken as the rate of creep and is used for qualitative comparisons; as creep rates with larger values indicate more creep deformation in a given duration of time.

As shown in table 2-4, eight types of creep testing took place. Each test group was composed of three to four test specimens. The above mentioned creep properties were determined individually for each of a group's three to four specimens. The individual specimen properties were then averaged to determine the uniaxial compressive creep behavior for the group.
CHAPTER 3

RESULTS AND DISCUSSION

3.1 Uniaxial Compression Results

3.1.1 Room Temperature Compression Results

Quasi-static uniaxial compression testing was conducted using the methods described in the previous chapter. Room temperature compression testing was completed for two formulations of the state change material. The first type was manufactured with a solution composed of 20% binder and the second type consisted of 77.7% binder. Both of the room temperature compression test groups were composed of nine specimens. Strain gages were attached to five of the nine specimens in each test group for the measurement of strain and the determination of modulus.

As determined from the uniaxial compression testing, the primary properties of interest were the compressive strength and the compressive modulus. The compressive strength was found by dividing the largest occurring instantaneous load by the specimen cross-sectional area. This can be demonstrated with an example. Compression test specimen #3 of the 77.7% binder test group experienced a maximum load prior to failure of 2438 N and had a diameter of 24.9 mm. With the diameter, the cross-sectional area was determined to be 474 mm². Then, the maximum load was divided by the specimen area and the resulting compressive strength was found to be 5.14 MPa (figure 3-3 is the
valid portion of the engineering stress versus axial strain curve for specimen #3). To determine the materials' compressive moduli, engineering stress values were determined and then plotted versus the axial strain data measured from the strain gages. The compressive modulus was taken as the slope of the initial linear portion of the resulting engineering stress versus axial strain curve.

With the above described methods, the individual compressive properties were found for each of the nine specimens in both test groups. Then, the individual specimen values were averaged to determine group properties. Table 3-1 gives the average compressive strengths and modulus values with standard deviations for the two material formulations. Figures 3-1 and 3-2 show the linear portions of stress versus strain curves for the 20% binder material and 77.7% binder material respectively. Figure 3-3 shows an entire stress versus strain curve typical of the state change material tested at room temperature.

Table 3-1: Room temperature compressive properties for two formulations of the 2Phase state change material

<table>
<thead>
<tr>
<th>Material Type</th>
<th>Compressive Strength</th>
<th>Compressive Modulus</th>
</tr>
</thead>
<tbody>
<tr>
<td>20% Binder</td>
<td>2.26 MPa ± 7%</td>
<td>2.32 GPa ± 18%</td>
</tr>
<tr>
<td>77.7% Binder</td>
<td>6.15 MPa ± 14%</td>
<td>19.50 GPa ± 22%</td>
</tr>
</tbody>
</table>
Figure 3-1: Linear portion of an engineering stress versus axial curve for the 20% binder material test group

Figure 3-2: Linear portion of an engineering stress versus axial curve for the 77.7% binder material test group
Figure 3-3: Typical engineering stress versus axial curve for the material tested at room temperature

As demonstrated in the above figure, all compression test specimens showed a linear stress-strain relationship up to an initial failure point followed by a significant drop in carried load. A sharp snap was usually heard at this initial failure point and visible vertical cracks would appear over the outer circumferences of the test cylinders. After the initial failure, the load would usually increase to a level just below or close to the failure point and then stay somewhat constant or decrease gradually as the specimen continued to crush and the material densified. Figure 3-4 shows two specimens, upon completion of testing, that are demonstrative of the common deformation mode seen in a majority of the room temperature compression test specimens.
The mode of deformation shown in the above figure is similar to barreling. Specimen barreling is often seen during the compression testing of cylindrical specimens with height-to-diameter ratios of 2.0 or less, when friction is present at the interface between the specimen surfaces and the load platens.

3.1.2 Elevated Temperature (177°C) Compression Results

Elevated temperature compression testing was also completed for two formulations of the state change material. The first type was manufactured with a solution composed of 20% binder and the second type consisted of 77.7% binder. Each of the elevated temperature compression test groups was composed of five specimens. As described in the previous chapter, strain gages were not used to determine the elevated temperature modulus, rather, a laser extensometer was applied to measure changes in specimen axial length.
Again, the primary properties of interest were the compressive strength and the compressive modulus. As before, the compressive strength was found by dividing the largest occurring instantaneous load value by the specimen cross-sectional area. As with the room temperature testing, to determine the materials' elevated temperature compressive moduli, engineering stress values were determined and then plotted versus the axial strain data measured from the laser extensometer. The compressive modulus was taken as the slope of the initial linear portion of the resulting engineering stress versus axial strain curve. The individual compressive properties were found for each of the five specimens in both test groups. Then, the individual specimen values were averaged to determine elevated temperature properties for the two test groups. Table 3-2 gives the average compressive strengths and modulus values with standard deviations for the two material formulations. Figures 3-5 and 3-6 show the linear portions of stress versus strain curves for the 20% binder material and 77.7% binder material respectively. Figure 3-7 shows an entire stress versus strain curve typical of the state change material tested at 177°C.

Table 3-2: Elevated temperature (177°C) compressive properties for two formulations of the 2Phase state change material

<table>
<thead>
<tr>
<th>Material Type</th>
<th>Compressive Strength</th>
<th>Compressive Modulus</th>
</tr>
</thead>
<tbody>
<tr>
<td>20% Binder</td>
<td>2.37 MPa ± 10%</td>
<td>0.88 GPa ± 18%</td>
</tr>
<tr>
<td>77.7% Binder</td>
<td>5.56 MPa ± 11%</td>
<td>1.74 GPa ± 23%</td>
</tr>
</tbody>
</table>
Figure 3-5: Linear portion of an engineering stress versus axial strain curve for the 20% binder material tested at 177°C

Figure 3-6: Linear portion of an engineering stress versus axial strain curve for the 77.7% binder material tested at 177°C
As shown in figure 3-7, and similar to the room temperature results, all elevated temperature compression test specimens showed a linear stress-strain relationship up to an initial failure point. A significant drop in carried load would follow, along with the appearance of vertical cracks over the outer circumferences of the test cylinders. After the initial failure, the load would usually increase to a level just below or close to the failure point and then stay somewhat constant or decrease gradually as the specimen continued to crush. Figure 3-8 show the common mode of deformation observed during the elevated temperature compression testing.
Figure 3-8: Common deformation mode observed during two of the elevated temperature (177°C) compression tests

As with the room temperature testing, the mode of deformation observed during the elevated temperature tests was similar to barreling. Again, specimen barreling is often seen during the compression testing of cylindrical specimens with height-to-diameter ratios of 2.0 or less, when friction is present at the interface between the specimen surfaces and the load platens.

3.1.3 Discussion of Uniaxial Compression Results

Both the room temperature and the elevated temperature compression test results show that the material composed with the 20% binder solution has significantly lower properties than the material prepared with the 77.7% binder solution. As mentioned in Chapter 1, the binder is the adhesive that holds the material together and gives it strength when in the hardened state. Therefore, the lack of binder present in the material made
with the 20% solution is most likely why its compressive properties are lower than the material composed with the 77.7% solution. To verify that the percentage of binder present in the solution was the potential cause of the difference in the compressive properties, a scanning electron microscope (SEM) was used to photograph the failure surfaces of the material. Figures 3-9 and 3-10 depict the 20% binder solution material and the 77.7% binder solution material, respectively.

Figure 3-9: SEM photograph of the 20% binder solution state change material
In the above figures, when the 77.7% binder material is compared with the 20% binder material, the amount of visible binder increases. As seen in figure 3-9, the 20% binder material is characterized with the presence of many whole spheres with little visible binder. However, in figure 3-10, the exact opposite trend is seen for the 77.7% binder material. This increase in the number of broken spheres in the higher percentage binder material can be explained in the following way: as an excess of binder is present, the bond holding the spheres together is greater. Therefore, when load is applied, the test material tends to fail at the spheres, rather than at the interfaces between them. This may help explain the lower compressive strength and modulus seen in the 20% binder material. The lack of broken spheres present in the lower percentage binder material, as seen in figure 3-9, indicates that the amount of binder present in capillarity between the
spheres is at a low level. This lack in binder between the spheres causes the material, when loaded, to fail at the interface between the spheres at locations of inadequate adhesion.

Next, the room temperature compressive properties, when compared to the elevated temperature properties, were, in the case of compressive strength, similar and, in the case of compressive modulus, significantly higher. Table 3-3 summarizes the room temperature and elevated temperature (177°C) compressive properties.

Table 3-3: Summary of compressive properties

<table>
<thead>
<tr>
<th>Binder Concentration</th>
<th>Ambient Strength</th>
<th>Ambient Modulus</th>
<th>177°C Strength</th>
<th>177°C Modulus</th>
</tr>
</thead>
<tbody>
<tr>
<td>20%</td>
<td>2.26 MPa</td>
<td>2.32 GPa</td>
<td>2.37 MPa</td>
<td>0.88 GPa</td>
</tr>
<tr>
<td>77.7%</td>
<td>6.15 MPa</td>
<td>19.50 GPa</td>
<td>5.56 MPa</td>
<td>1.74 GPa</td>
</tr>
</tbody>
</table>

As shown in the above table, there is no appreciable difference in the compressive strengths. However, two observations can be made from the compressive modulus values:

- The room temperature compressive modulus values are greater than the elevated temperature modulus values.
- There is a greater difference between the room temperature and elevated temperature values with the 77.7% binder material.

In previous studies, dealing with high temperature effects on the 2Phase state change material, it was concluded that when the material is tested at 400°C, the spheres become brittle and the binder deteriorates [24, 30]. In those studies, the material tested at 400°C
experienced significant decreases in both the strength and modulus. As previously discussed, when tested at room temperature, the material composed with lower percentages of binder (20% for example) fail at the interfaces between the microspheres, as little binder remains in capillarity. Therefore, deterioration of the binder due to exposure to a temperature of 400°C would cause premature failure of the 20% binder test material. Also, as previously discussed, when tested at room temperature, the material composed with higher percentages (77.7% for example) of binder experience failure in the sphere walls, rather than the interfaces between them. Therefore, embrittlement of the microspheres would cause premature failure of the 77.7% binder material. However, in this thesis investigation, and unlike those previous studies, testing was performed at 177°C and only the compressive modulus was found to be affected. It is likely that the binder gluing the material together begins to soften, at temperatures in the range of 177°C, before its deterioration at the higher temperatures. Also likely, is the possibility that the binder, although it may soften, does not lose its structural integrity, resulting in a material that displays some ductility at higher temperatures, but no significant loss in strength. Rahman [25] observed that thin films of the proprietary binder experience significant volume increases when exposed to temperatures in excess of 145°C especially with fast heating rates. The binder's foaming would contribute to a decrease in the state change material's overall modulus. Rahman also determined that thicker films of binder experience volume increases greater than thinner films when exposed to elevated temperatures. The binder layer between the spheres of the 77.7% binder material is thicker than in the 20% binder material. Therefore, it is expected that the binder layer increases in volume more in the 77.7% material than the in the 20% binder material.
resulting in more room for deformation and stiffness loss. This additional binder present in the state change material composed with greater amounts of binder may help explain its propensity to lose stiffness faster than the material made with less binder. There is more binder between the spheres in the 77.7% binder material, as the binder softens, the spheres are permitted more deformation before encountering another sphere; while in the 20% binder material, little binder remains between the spheres, which are more tightly packed than the 77.7% binder material, resulting in only a slight space between spheres for translation.

The relatively high standard deviations present in the 2Phase state change material’s compressive properties require discussion of the material’s homogeneity. Deviations of the presented magnitudes are not uncommon or considered unacceptable. Several factors contribute to the material’s non-homogeneous behavior. The glass microspheres range from 100 to 250 microns in diameter and there are also variations in the spheres’ wall thickness measurements. These variations result in slightly different compressive failure strengths for each individual microspheres. Also, there is known to be a binder density gradient throughout the 2Phase material in the thickness direction. As vacuum pressure in applied to consolidate the material from the topside of the mold only, the binder density tends to increase towards the lower portions of the test specimens. Aside from the binder density gradient, the binder distribution throughout the material is somewhat variable and cannot be controlled.
3.2 Creep Results

3.2.1 Room Temperature Creep Results

With the methods described in the previous chapter, room temperature creep testing was completed for two formulations of the 2Phase state change material. The first formulation was manufactured with a solution composed of 20% binder and the second was composed of 77.7% binder. The room temperature creep testing was conducted with two different load levels corresponding to 15% and 30% of the materials’ uniaxial compressive strength. As there were two varieties of test material and two load levels, test specimens were divided into four test groups. The first group was composed of specimens manufactured with the 20% binder solution and was tested with the 15% load level. The second test group was composed of specimens manufactured with the 20% binder solution and was tested with the 30% load level. The third test group was composed of the specimens manufactured with the 77.7% binder solution and was tested with the 15% load level and the fourth group was composed of specimens manufactured with the 77.7% binder solution and was tested with the 30% load level. Also, each test group was composed of three to four specimens and a strain gage was attached to each specimen to measure long term creep strain.

The primary creep properties of interest were the overall specimen deformations and the rates at which the specimens crept. To determine the overall deformation for an individual test specimen, the definition of strain was applied and the largest occurring strain value was multiplied by the test specimen’s original height. The resulting value was the total deformation of the specimen in the axial direction. To determine the creep rate of an individual test specimen, an instantaneous strain versus time plot was created.
When creep behavior was present, the shape of the resulting curve was approximately logarithmic in shape. By fitting a logarithmic regression to the curve, an equation would result from which the creep rate could be obtained.

With the methods described above, the individual creep deformations and rates were found for each of the specimens in all four test groups. Then, the individual specimen values were averaged to determine group properties.

Upon completion of the room temperature creep testing, creep behavior was found present in the material composed with 20% binder. However, no clear trend of creep was found in the test specimens made with 77.7% binder. Table 3-4 summarizes the average creep properties for the two material formulations. A representative creep strain versus time curve for the 20% binder formulation is shown in figure 3-11. Figure 3-12 shows a representative creep strain versus time curve demonstrating the non-existent creep behavior of test specimens composed with 77.7% binder as a function of time.
Table 3-4: Room temperature creep properties for two formulations of the 2Phase state change material

<table>
<thead>
<tr>
<th></th>
<th>20% Binder Formulation</th>
<th>77.7% Binder Formulation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stress/Strength=15%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Deformation after 24</td>
<td>1.49 μm ± 43%</td>
<td>0.00 μm ± 0%</td>
</tr>
<tr>
<td>hrs</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Rate over 24 hrs</td>
<td>8.64 με/log(s) ± 52%</td>
<td>0.00 με/log(s) ± 0%</td>
</tr>
<tr>
<td>Stress/Strength=30%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Deformation after 24</td>
<td>7.55 μm ± 18%</td>
<td>0.00 μm ± 0%</td>
</tr>
<tr>
<td>hrs</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Rate over 24 hrs</td>
<td>32.37 με/log(s) ± 33%</td>
<td>0.00 με/log(s) ± 0%</td>
</tr>
</tbody>
</table>

Figure 3-11: Representative strain-time curve for the 20% binder formulation
77.7% Binder Formulation
Room Temperature Creep Test

3.2.2 Elevated Temperature (177°C) Creep Results

Elevated temperature creep testing was also completed for two formulations of the state change material. The first type was manufactured with a solution composed of 20% binder and the second type consisted of 77.7% binder. Each of the elevated temperature creep test groups was composed of three specimens. As described in the previous chapter, strain gages were not used to determine the elevated temperature creep properties; rather, a laser extensometer was applied to measure axial length changes due to temperature and load.

As with the room temperature creep testing, the elevated temperature creep characterization was conducted with two different load levels corresponding to 15% and 30% of the materials' uniaxial compressive strength. Again, as there were two varieties of test material and two load levels, test specimens were divided into four test groups. The first group was composed of specimens manufactured with the 20% binder solution
and was tested with the 15% load level, the second test group was composed of specimens manufactured with the 20% binder solution and was tested with the 30% load level, the third test group was composed of the specimens manufactured with the 77.7% binder solution and was tested with the 15% load level and the fourth group was composed of specimens manufactured with the 77.7% binder solution and was tested with the 30% load level.

Similarly to the room temperature testing, the primary creep properties of interest in the elevated temperature creep characterization were the overall specimen deformations and the rates at which the specimens crept. To determine the overall deformation for an individual test specimen, the largest occurring strain value was multiplied by the test specimen's original height, resulting in the total deformation of the specimen in the axial direction. The creep rates of individual test specimens were determined with a method similar to the one used for the room temperature testing. Instantaneous strain versus time plots were created and logarithmic regressions were applied to the curves. With these methods, the individual creep properties were found for each of the specimens in all four test groups. Those values were then averaged to determine group creep properties.

Creep behavior was observed to be present in all of the high temperature creep test groups. Table 3-5 gives the average creep properties and standard deviations for the two material formulations. Representative creep strain versus time curves for the 20% and 77.7% binder formulations are shown in figures 3-13 and 3-14 respectively.
Table 3-5: Elevated temperature (177°C) creep deformation for two formulations of the 2Phase state change material after 4 hours

<table>
<thead>
<tr>
<th></th>
<th>20% Binder Formulation</th>
<th>77.7% Binder Formulation</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Stress/Strength=15%</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Deformation after 24 hrs</td>
<td>10.49 μm ± 2%</td>
<td>74.85 μm ± 28%</td>
</tr>
<tr>
<td>Rate over 24 hrs</td>
<td>25.28 με /log(s) ± 30%</td>
<td>332.91 με /log(s) ± 36%</td>
</tr>
<tr>
<td><strong>Stress/Strength=30%</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Deformation after 24 hrs</td>
<td>19.55 μm ± 23%</td>
<td>107.11 μm ± 3%</td>
</tr>
<tr>
<td>Rate over 24 hrs</td>
<td>48.32 με /log(s) ± 19%</td>
<td>520.35 με /log(s) ± 4%</td>
</tr>
</tbody>
</table>

Figure 3-13: Representative strain-time curve for the 20% binder formulation tested at 177°C for 4 hours
3.2.3 Discussion of Creep Results

From the room temperature and elevated temperature creep properties presented in the previous sections, four observations can be made:

- At room temperature, the 20% binder material experiences creep, but at low magnitudes.
- The 77.7% binder material does not experience creep at room temperature for the compressive loads applied.
- Both material formulations experiences significant increases in creep behavior at elevated temperatures.
- A greater difference is displayed in the room temperature and elevated creep properties for the 77.7% binder material.
Tables 3-6 and 3-7 summarize the room temperature and elevated temperature (177°C) creep deformations for the two material formulations.

Table 3-6: Summary of room temperature creep deformations

<table>
<thead>
<tr>
<th>Binder Concentration</th>
<th>Deformation 15% Load</th>
<th>Deformation 30% Load</th>
</tr>
</thead>
<tbody>
<tr>
<td>20%</td>
<td>1.49 μm</td>
<td>7.55 μm</td>
</tr>
<tr>
<td>77.7%</td>
<td>0.00 μm</td>
<td>0.00 μm</td>
</tr>
</tbody>
</table>

Table 3-7: Summary of elevated temperature (177°C) creep deformation

<table>
<thead>
<tr>
<th>Binder Concentration</th>
<th>Deformation 15% Load</th>
<th>Deformation 30% Load</th>
</tr>
</thead>
<tbody>
<tr>
<td>20%</td>
<td>10.49 μm</td>
<td>19.55 μm</td>
</tr>
<tr>
<td>77.7%</td>
<td>74.85 μm</td>
<td>107.11 μm</td>
</tr>
</tbody>
</table>

From the observations made of the room temperature creep properties, it is possible that the ambient temperature creep is a function of compressive strength. As shown by the compression testing results, the 77.7% binder material displays a superior compressive strength when compared to the 20% binder material. Also, during the room temperature creep testing, global specimen fracture was encountered on three occasions with the 20% binder material with failures occurring at both load levels. As the material composed with the 20% binder formulation has a lower compressive strength, it is likely that internal specimen fracture occurs over time when under a sustained load. Internal fractures would contribute to the permanent plastic deformation of test specimens.
Furthermore, global fracture was not encountered at either load level with the 77.7% binder material, indicating its superior strength and resistivity to room temperature creep.

The observations made of the elevated temperature creep behavior indicate that creep at higher temperatures is related to the binder material. As mentioned during the discussion of the compression results, it is possible that the binder softens when exposed to temperatures in the range of 177°C as a result of the binder material's tendency to foam, and increase in volume, when exposed to elevated temperatures. This softening provides some ductility to the material allowing the spheres to translate and move when loaded in compression. Also, the material composed with the 77.7% binder formulation has an excess of binder in capillarity between the spheres. This allows more deformation among the spheres in the material as the binder softens. The diminishing rigidity of the binder would explain the increase in creep behavior in both material formulations. Furthermore, the abundance of binder in the 77.7% binder material helps explain its propensity to experience significant increases in creep deformation when compared to the 20% binder material.

As with the compressive characterization, the standard deviations present in the creep results require discussion of the material's homogeneity. Again, deviations of these magnitudes are not uncommon for the state change material and are considered unacceptable. As mentioned in the discussion of the compression results, the non-homogeneous behavior of the material is most likely due to variation in microsphere dimension as well as in the binder distribution throughout the material.
CHAPTER 4

CONCLUSIONS

4.1 Experimental Observations and Conclusions

4.1.1 Observations and Conclusions of the Uniaxial Compression Test Results

The various outcomes from the uniaxial compression testing presented in the previous chapter result in a number of experimental observations and conclusions. Primarily, it was found that the proprietary binder material plays the most important role in determining the compressive behavior of the material. Again, the binder is the adhesive that holds the state change material together in its hardened state and gives it strength and force resistance. It was found that by varying the percentage of binder in the liquid component of the material, the failure mode of the material when, loaded in compression, changes. Specifically, it was found that the 20% binder material, which is representative of the material's water soluble tooling application, tends to have very little binder remaining in capillarity between the microspheres. As a result, the bond holding the spheres together is weak. When the material is loaded in compression, failure occurs as whole spheres separate from each other in areas of poor binder adhesion. Subsequently, this scant amount of binder and the resulting lack in adhesion between spheres results in relatively low failure loads. However, state change material formulations composed of
higher concentrations of binder do not lend themselves well to application requiring water solubility in short amounts of time.

However, a different failure mode was observed for the material manufactured with the 77.7% binder formulation. The 77.7% binder material, which is representative of the material's tool bed applications, tends to have an excess of binder remaining between the spheres in capillarity. Subsequently, the bond holding the spheres together is much stronger in the higher concentration binder material than the material composed with lower concentrations of binder. As the bond between the spheres is greater, when this material is loaded in compression, failure tends to occur in the spheres themselves. Specifically, it was found with SEM microscopy that the abundance of binder remaining in capillarity prevents failure at the interface between the spheres, unlike the 20% binder material. Rather, failure was observed to occur in the sphere walls themselves. Also, this increased amount of binder holding the material together results in higher failure loads and, therefore, a higher average compressive strength.

The binder was also observed to heavily influence the state change material's stiffness when tested at elevated temperatures. It is known from previous studies that the binder, when in the material's hardened state, deteriorates and the material's strength and stiffness values are both significantly decreased when tested at 400°C. The tests in this study took place at 177°C and only the compressive modulus was found to be affected. It was concluded that the binder material begins to soften at temperatures in the range of 177°C, prior to its complete deterioration at temperatures in excess of 400°C. Furthermore, it was concluded that, although the binder material does begin to soften, it does lose its structural integrity at 177°C. The result is a material with lower stiffness at
test temperatures of 177°C, but with no significant loss in strength. These conclusions, of the binder material’s elevated temperature behavior, explain the observed trend in which, for both tested material formulations, the elevated temperature strength was approximately identical to that of the room temperature testing, but the elevated temperature modulus was significantly less than the room temperature modulus.

It was also observed that the modulus of the 77.7% binder material was affected more due to the elevated temperatures than the 20% binder material. Specifically, the 77.7% binder material demonstrated a room temperature modulus of 19.50 GPa and an elevated temperature modulus of 1.74 GPa, while the 20% binder material’s room temperature modulus dropped from 2.32 GPa to 0.88 GPa as a result of elevated temperate exposure. It was concluded that there is significantly more binder remaining between the spheres in the material composed with a higher concentration of binder. Therefore, as the binder begins to soften and spheres begin to translate, more movement is permitted in the 77.7% binder material, as there is more binder present in the “cushion” between microspheres. In the 20% binder material, minimal binder remains between the spheres, and practically no binder “cushion” exists. As the binder softens and the spheres move, there is less space for translation than in the material composed with higher concentrations binder material.

In general, it can be said that state change material composed with higher concentrations of binder are more sensitive to temperature change than the lower concentration binder material.
4.1.2 Observations and Conclusions of the Creep Test Results

A number of experimental observations and conclusions can be drawn from the results of the uniaxial compression creep testing. Similarly to the conclusions of the compression testing, the proprietary binder was found to have a major influence in the creep behavior of the 2Phase state change material. It was concluded that the room temperature creep behavior is related to the material’s ultimate compressive strength. It was observed upon completion of the room temperature creep tests that the higher concentration binder material does not creep any appreciable amount. However, the test specimens composed with 20% binder did creep, although minimally. It was also observed that several of the 20% binder specimens cracked well into the 24 hour test duration, in all cases after the eighth hour of testing, which could indicate the formation of cracks within the specimens as a result of sustained exposure to the compressive loads. It is suspected that crack formation is related to the material’s compressive strength, as it was found that the binder concentration has a direct effect on the material’s rigidity. Small cracks within the interior of a specimen would lessen its structural integrity and lead to deformation, helping to explain the 20% material’s propensity to creep at room temperature and the 77.7% material’s resistance to temporal deformation.

The proprietary binder was also found to affect the elevated temperature creep. It was concluded that the elevated temperature creep behavior is related to the material’s stiffness. The amount of creep deformation for both material formulations was observed to increase significantly when exposed to temperatures of 177°C. As previously discussed, the state change material’s compressive modulus decreased when tested at elevated temperatures. This was concluded to be a direct result of the amount of binder
present between the spheres in capillarity. Specifically, more binder was observed between the spheres in the 77.7% binder material. Therefore, as the binder softened due to high temperature exposure, the spheres in the 77.7% binder material were able to move more before encountering another sphere. Similarly, the 20% binder material was found to have significantly less binder between the spheres. Therefore, when the binder material softened due to elevated temperature exposure, the spheres had less space to change position. These same principles can be applied to explain the creep behavior of the 2Phase state change material at elevated temperatures. As there is more binder in capillarity between the spheres of the 77.7% binder material, as the binder softens, the spheres translate more and more creep deformations are observed and recorded. Conversely, there is less binder between the spheres in the 20% binder material. Therefore, the spheres translate less and less creep deformation if seen.

In general, as previously found, the state change material composed with a higher concentration of binder is significantly more sensitive to elevated temperature exposure, than are material formulations consisting of lower concentrations of binder.

4.2 Comparison of the State Change Material to Other Common Tooling Materials and the State Change Material’s Suitability to Tooling Applications

To quantify the usefulness of the 2Phase state change material as an alternative to metallic tooling, it should be compared to other common molding materials. As mentioned in Chapter 1, metals are often used for composite tooling. These materials are often costly to acquire and complicated machining processes are usually required. Therefore, the 2Phase state change material lends itself to the application of composite
tooling, as it is both inexpensive and relatively easy to use. However, a good molding material should be both strong and dimensionally stable. Strength is important as molds must be able to withstand the forces and pressures involved in various composite manufacturing processes and dimensional stability is important as molds must not creep by any appreciable amount to preserve part tolerances. Aluminum and Invar are common metallic tooling materials. A review of recent research related to the strength and dimensional stability of these metals was conducted to determine appropriate values with which to compare the 2Phase state change material.

The reviewed research related to the mechanical and thermal properties of various aluminum alloys yielded much information. In one study, the high temperature creep behavior of aluminum alloys under the application of low stress-strength ratios was determined. For a test duration of approximately 24 hours, the maximum occurring percentages of strain were determined as 0.15% to 0.20%. In an additional study in which the creep resistance in aluminum was studied, the investigators concluded that creep in aluminum alloys is generally caused by gliding dislocations limited by obstacles. Furthermore, hardening the material can actually decrease the apparent creep deformation. Also, the yield strength of aluminum alloys commonly used for tooling is approximately 250 MPa. [31-32]

Similarly, much information was also yielded from the review of research related to the thermo-mechanical characterization of Invar. First, the yield strength of Invar was determined to be approximately 480 MPa. Next, in a study to determine the temporal length change of Invar bars, the test material was monitored at room temperature for 170 days with no applied load. The material was observed to experience a change of length of
5.64 parts per billion a day. In an additional study, the effects of elevated temperature and heat treatment on the creep behavior of Invar were determined. It was concluded that, depending on the composition of the Invar, the deformation could be as great at 11.0 parts per million per day. However, it was also observed that, by limiting the carbon content and amount of impurities in the material, the deformations could be decreased to less than 1.0 parts per million per year. It was also found that heat treatment could increase the material's dimensional stability. [33-34]

The results from this thesis study can be compared with the data concluded from the aluminum and Invar literature. Tables 4-1 and 4-2 summarize the mechanical characterization and creep deformations of the 2Phase state change material.

Table 4-1: Compressive properties of the 2Phase state change material

<table>
<thead>
<tr>
<th>Binder Concentration</th>
<th>Room Temperature Strength</th>
<th>177°C Strength</th>
<th>Room Temperature Modulus</th>
<th>177°C Modulus</th>
</tr>
</thead>
<tbody>
<tr>
<td>20%</td>
<td>2.26 MPa</td>
<td>2.37 MPa</td>
<td>2.32 GPa</td>
<td>0.88 GPa</td>
</tr>
<tr>
<td>77.7%</td>
<td>6.12 MPa</td>
<td>5.56 MPa</td>
<td>19.50 GPa</td>
<td>1.74 GPa</td>
</tr>
</tbody>
</table>
From the data given in table 4-1, it is seen that the state change material's strengths for the various test temperatures and binder formulations are significantly lower than the yield strengths reported for the metallic tooling. However, the maximum vacuum pressure generally applied during composite manufacturing is 0.101 MPa. Therefore, despite the 2Phase material’s relatively low strength, it should be strong enough to withstand the pressure applied during composite manufacturing.

The creep deformations observed of the 2Phase state change material are given in table 4-2. The deformation are in terms of parts per million per hour and, although the rate of creep of the state change material was not perfectly linear, this is a convenient way of reporting changes in length for comparison with the aluminum and Invar data. When compared, the creep behavior of the state change material is somewhat similar to the temporal length changes of the aluminum alloys. However both the aluminum and the 2Phase material are significantly more dimensionally unstable when compared to Invar. It is difficult to qualify the 2Phase material for tooling applications in terms of its creep behavior. The state change material’s appropriateness depends upon allowable tolerances
as specified by the potential users of the 2Phase Reformable Tooling System (RTS). However, given the data of table 4-2 room temperature applications for the state change material should utilize a binder concentration of 77.7%. The 77.7% binder formulation tends to creep little, or not at all, at room temperature. However, for high temperature application, state change material formulation composed with lesser amounts of binder should be applied. It is observed, from both the compressive and creep characterizations, the material composed with greater amounts of binder are much more sensitive, and more negatively affected, by exposure to elevated temperatures.

The coefficient of thermal expansion is also important when discussing a material’s dimensional stability and suitability to tooling applications. Rahman [25] determined the average CTE for the 2Phase state change material to be 5.67 ppm per degree K for all temperature exposures and binder formulations. This value is small and, in addition to the CTE value’s consistency for different environments, is considered excellent for a tooling material.

In conclusion, the 2Phase state change material, in terms of compressive strength, is strong enough to withstand the expected forces and pressures generally encountered during composite manufacturing. Also, by varying the percentage of binder present in the material formulation, the state change material can be made relatively dimensionally stable for various environmental situations and temperatures.

4.3 Recommendations for Future Work

Aside from the findings presented in this thesis study, there is scope for much future work. As a result of the presented observations, many research questions have been
raised indicating the possible need to continue the mechanical and thermal characterizations of the 2Phase state change material. Primarily, the proprietary binder material was found to have the most profound effect on the material's behavior. Therefore, it is of interest to characterize formulations of the 2Phase material composed with various levels of binder, other than 20% and 77.7%. At least one formulation should consist of a percentage between 20% and 77.7%, possibly 50%, and be investigated to determine if there is a formulation at which a trade off between the thermo-mechanical properties and ease of water solubility exists. Additionally, it has been found that lower levels of binder concentration facilitate easier material dissolvability in water. Therefore, when considering the 2Phase material for water soluble tooling, additional material formulations, composed of binder concentrations less than 20%, should be investigated for thermal and mechanical properties. Formulations composed with higher concentrations of binder, in excess of 80%, could also be of interest for applications requiring higher strengths and stiffnesses.

Aside from the proprietary binder, the test temperature also profoundly affected the thermal and mechanical properties of the state change material. The results of this study and the results of previous studies indicate that the binder, when in the hardened state of the material, softens at temperatures in the range of 177°C and deteriorates at temperatures of approximately 400°C. Further investigations should be conducted in which mechanical and thermal testing is conducted at temperatures other than those previously researched. At least one test temperature should be between 177°C and 400°C to determine if there exists a temperature at which the binder transitions from softening to deterioration. Additionally, testing could take place at temperatures in excess of 400°C to
find the point at which the material loses all thermal and mechanical integrity. Testing could also take place with temperatures in between ambient and 177°C to determine at what point the material begins to soften. Testing of various binder formulations could take place concurrently with the test temperature experimentations to determine if the state change material can be tailored to specific application.

It may be of interest to experiment with alternate binder materials. It was determined from this thesis study that the proprietary binder material is the primary component causing stiffness loss and dimensional instability at elevated temperatures.

Also, it is known that the tooling material undergoes loads other than compression, such as flexural and tensile. It may be of interest to determine the thermomechanical properties of the state change material when tested in tension and flexure.
VERIFICATION OF COMPRESSION SPRING STIFFNESS

A.1 Overview of Verification Tests

As mentioned in Chapter 2, the eight compression springs were tested to verify the stiffness values reported by the manufacturer. The stiffness value given by the distributor for all eight springs was 20.47 N/mm. However, to ensure accuracy during the application of the creep inducing loads, the reported stiffness value had to be validated.

A compression spring's stiffness is directly proportional to the applied axial load and inversely proportional to the resulting deflection. These principles are demonstrated in the below equation.

\[ k = \frac{P}{\delta} \]

Where:

k = Spring Stiffness

P = Force exerted by spring

\( \delta \) = Spring deflection

To experimentally determine a compression spring's stiffness value, testing to find the ratio of applied load-to-sustained deflection must take place. Uniaxial compression testing is a standard method of determining the aforementioned ratio.
A.2 Room Temperature Verification Tests

A.2.1 United Testing System

All of the room temperature uniaxial compression tests done to verify the spring stiffness values were conducted with a United SSTM-1 United Testing System. The United SSTM-1 is a mechanical type tension/compression testing system, which, unlike the MTS, which is comprised of several primary components, consists of only two interconnected components. The load frame is the first component. Comparable to the MTS machine's load frame, the United machine’s load frame manages the application of loads and the resulting displacements. The second component is the United machine’s computer. The computer manages the movement of the load frame’s actuators as static loads are applied and acquires useful data (displacement, force, strain, etc.) during tests. This computer is a PC system operating on Windows XP. The main software program used to operate the United machine, including the direction of tests and the creation of testing templates, is DATUM 4.0. Figure A-1 shows the United machine's laboratory setup.

Figure A-1: The UNITED SSTM-1 laboratory setup
A.2.2 Room Temperature Verification Test Procedures

Stiffness verification testing was conducted based on a method derived from the principles given in the preceding section. Specifically, the United SSTM-1 system software, DATUM 4.0, was used to create a testing template in which the springs would be tested in compression to a maximum deflection of 25.4 mm at a constant crosshead descent rate of 5.08 mm/min. During the tests, instantaneous load and crosshead deflection data were recorded.

To conduct the tests, the United machine was outfitted with a 22.2 kN load cell. The test specimens were then loaded with a set of compression platens. Although the ends of the compression springs were closed and ground, the top and bottom faces were not perfectly parallel. Therefore, the uppermost platen was spherically seated. Figure A-2 shows a compression spring in the experimental set-up. Upon completion of the verification testing of the eight compression springs with the aforementioned template, load versus crosshead deflection curves were created. The slopes of the resulting linear curves were taken to be the compression springs’ actual stiffness values.

Figure A-2: Compression spring in testing configuration
A.3 Results

The compression springs were numbered one through eight and then tested sequentially at ambient temperatures. In the following section, the load versus crosshead deflection curves for the room temperature tests are given.

A.3.1 Room Temperature Results

Compression Spring #1: $k=21.12 \text{ N/mm}$
Compression Spring #2: $k=21.52 \text{ N/mm}$

\[
y = 21.517x - 0.326
\]
\[R^2 = 1\]

Compression Spring #3: $k=21.42 \text{ N/mm}$

\[
y = 21.42x + 0.069
\]
\[R^2 = 0.9999\]
Compression Spring #4: $k=21.31 \text{ N/mm}$

Load vs Deflection

$y = 21.307x - 0.3892$
$R^2 = 0.9999$

Compression Spring #5: $k=21.27 \text{ N/mm}$

Load vs Deflection

$y = 21.27x - 1.6625$
$R^2 = 0.9999$
Compression Spring #6: k=21.39 N/mm

Compression Spring #7: k=21.50 N/mm
Compression Spring #8: k=21.50 N/mm

Load vs Deflection
Spring #8

\[ y = 20.933x - 1.8129 \]

\[ R^2 = 0.9998 \]
APPENDIX B

TESTING EQUIPMENT SPECIFICATIONS

MTS Testing Systems
Working dimensions: 635 mm wide by 1463 mm high
Axial load transducer limit: 250 KN
Torsional load transducer limit: 2200 N-m
Linear actuator limit: ±76 mm, measured with an LVDT
Rotary actuator limit: 100° static rotation (± 50°), measures with ADT; 90° dynamic rotation (± 45°)

MTS Load Cell
Model 662.10A-08
Range: 0-250 kN (0-55 kips)
Non-Linearity: ±0.15% Full Scale
Hysteresis: ±0.10% Full Scale
Accuracy: ±1.00%

MTS Laser Extensometer
Electronic Instrument Research
Model LE-01
Measurement range: 8-80 mm (0.3-3.2 in)
Non-Linearity: ±0.005 mm. (±0.0002 in)
Resolution: 0.001 mm (0.0001 in)
Repeatability: ±0.001 mm (±0.0001 in)
Target distance: 250 mm (10 in) nominal, 500 mm (20in) maximum
Scan rate: 100 scans/second

MTS Strain Conditioning Box
Vishay Measurements Group
2100 System
Built-in bridge completion
Accuracy: ±0.1% of readings ±3 counts
Gage factor settings: Range 0.0 to 10.0
Portable Strain Conditioning Box
(Room temperature creep testing)
Vishay Micro-Instrument
Model P3
Built-in bridge completion
Accuracy: ±0.1% of readings ±3 counts
Gage factor settings: Range 0.500 to 9.900

United SSTM-1 Testing Systems
Working dimensions: 635 mm wide by 1600mm high
Axial load transducer limit: 22.2 kN
Linear actuator limit: ±1067 mm, measured with an LVDT

United SSTM-1 Load Cell
Model TV1104495
Range: 0-22.2 kN (0-5 kips)
Non-Linearity: ±0.04% Full Scale
Hysteresis: ±0.04% Full Scale
Accuracy: ±0.10%
REFERENCES


[4] Interview by Author: George Calvert, Engineer, 2Phase Technologies, Inc.


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• “Characterization of State Change Reconfigurable Tooling Material.” Proceedings of 2005 SAMPE Fall Technical Conference

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