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## **AC 2011-2729: UNDERGRADUATE RESEARCH ON HIGH TEMPERATURE CREEP BEHAVIOR OF POLYMERS**

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# **Undergraduate Research on High Temperature Creep Behavior of Polymers**

## Abstract

Creep is very slow deformation of materials at constant temperature and under steady stress. It is a diffusion phenomenon accelerated by higher temperatures at stress level below yield strengths. Although the result of creep in all materials are similar but physical mechanisms of creep in metals and polymers are very different because of the difference in the atomic and crystal structures. Specific knowledge of creep deformation is necessary for design and application of polymer based products at elevated temperatures. A senior design group was assigned a project to design and build a high temperature tester for polymers. The tester was designed to utilize small standard dog bone specimens under tensile load inside a heated chamber. It was equipped with automated temperature control and instrumented for computerized monitoring and logging of both temperature and elongation over time. Successful tests on a limited set of industrial polymers demonstrated the functionality of the tester. The results provided good estimates of the creep behavior of the experimental polymers for design purposes that were not easily available in the literature. Two minority and two honors research students were also engaged to fine tune the hardware, install and test the data acquisition system, and finally perform a range of creep experiments on the tester. The data on creep rates and times to failure conform to the hypothesis that these would be affected proportionately as stress and temperature are changed. Not only the design and operation of the tester gave the students high level of creep awareness and knowledge but also the tester and the experimental process now provide opportunities for generating experimental creep data for design and research purposes. The design group and the research students were all very enthusiastic to be part of such a novel laboratory experience. A few upgrading ideas are being considered for improving the functioning, monitoring, and utility of the tester.

## Introduction

Probably the least discussed failure modes in engineering design is creep. Not only it is almost absent in strength of materials texts but also it receives the least amount of attention among students and instructors of engineering design. These do not necessarily decrease the importance of creep as the principal failure modes in many industries and applications such as turbine blades, high temperature pressure vessels, mounting/assembly bolts in engines etc. Creep failures demonstrate themselves as deformations at higher temperatures over relatively

long period of times. The steady state creep deformation (strain  $\epsilon$ ) rate is predicted by equation 1, where  $K$  and  $n$  are constants,  $Q$  is the activation energy<sup>1</sup>,  $R$  is the gas constant and  $T$  is the absolute temperature.

$$(\partial\epsilon/\partial t) = K_2 * (\text{stress})^n * \text{EXP}(-Q/(R*T)) \quad (1)$$

For a given material and under similar testing conditions, the  $K_2$ ,  $n$ , and  $Q$  values will remain constant. Then at a given temperature the simplified relation will take the form shown in equation 2.

$$(\partial\epsilon/\partial t) = K_1 * (\text{stress})^n \quad (2)$$

On the other hand if the stress is maintained the same, the creep rate will depend on the absolute temperature of the specimen is subjected to. This may be expressed by equation 3,

$$(\partial\epsilon/\partial t) = K_3 * \text{EXP}(K_4/T) \quad (3)$$

where  $K_3$ ,  $K_4$  are respective constants.

While the equations predict creep at any temperature and stress levels, in reality creep does not become apparent until the temperatures reach a certain critical or threshold level. This level often termed as homologous temperature<sup>3</sup> given by equation 4.

$$T_{\text{homologous}} = T_{\text{service}}/T_{\text{melt}} \quad (4)$$

This nondimensional number for metallic materials ranges between 0.4 – 0.5. For polymers the threshold temperature often described by what is known as glass transition<sup>4</sup> temperature  $T_g$ , when polymers transform from brittle glassy state to ductile state. For ceramic materials it is usually higher than metallic materials. These strains rates in the above equations are for steady state deformations.

While vast majority of creep testing and analyses are done for metallic materials due to their application in high temperature applications, polymeric and composite materials also need attention as these are increasingly replacing metallic components and are often being used in higher temperature applications. Diffusion phenomenon of creep deformation in metals is simpler to explain due to their simple crystalline and bonding structures. Interplanar and intergranular slips are the foremost reasons of creep in metals and alloys. The creep mechanism in polymers<sup>5,6</sup> would be a mixture of a combination of mechanisms including polymer chain alignment, interchain slip, unfolding of crystalline regions etc. A large percentage of composite materials contain some type of polymers as matrix and/or reinforcing agent and are thus also subject to creep failure at elevated temperatures.

### Design of the Tester

A group of students from Engineering Materials, when enrolled in Machine Design class, were prompted to design and build a creep tester which would be incorporated in our Materials

Testing Lab. The enthusiastic group, motivated by the fact their design will be used by fellow students, gladly accepted the proposal and ended up with a creep chamber that is electrically heated, dead weight loaded, and instrumented to monitor temperature and strain of the specimen.

Little experimental creep information is available in the open literature which posed great challenge and instilled fear among the research students. The primary study of creep started with the Callister<sup>1</sup> text book. Then the students quickly looked up creep on the Wikipedia<sup>2</sup> which, to the comfort of the students, provided further information in plain English. The author/instructor provided more encouragement than prompting in this information gathering process.

The design group started with a set of customer requirements and followed by developing a set of satisfying design specifications that would be compared with the functionalities of the tester being developed<sup>7</sup>.

The completed tester was designed with hinged door, insulated panels, two incandescent light bulbs for heating, a temperature controller that was manually set, and a pair of Pasco transducers to monitor temperature and elongation of the specimens over time. The load was applied via a pair of dead weights used for weight lifting. These are shown in figures 1 and 2.



Figure 1: The tester and set up. Tensile load is applied from the bottom.



Figure 2: The Pasco data interfaces.

## Specimen

Dictated by the size of the tester a smaller standard dog bone specimen was chosen. The shape and dimensions of the specimen are shown in figure 3. To facilitate accurate dimension and productivity, a special machining jig was developed for use on the CNC mill on which the specimens were cut from sheet materials. The jig is shown in Figure 4. Three industrial

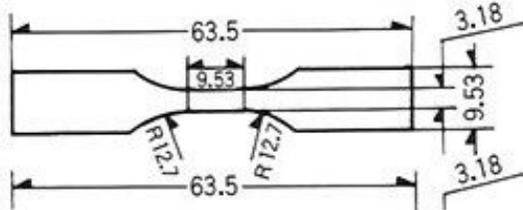


Figure 3: Dog bone specimen miniature version per ASTM D-18822-L.  
All dimensions are in mm. Specimen is 0.318 mm thick.

Table 1: Customer and Design Specifications

Customer Requirements	Design Specifications
Polymer specimens	Material permitted by budget. ABS, Nylon, Delrin, PVC, etc. Small CSA (~0.125 in <sup>2</sup> ) similar to ASTM D1822 specimens
Adjustable load, linear application	No more than ~100lbf load Stress dictated by - ASTM 1822 material, max stress = 6400 psi Easy adjustment of load force
Adjustable temperature	Max Temp dictated by specimen material. = 300°F Multiple thermocouples to verify even temperature in chamber Heating method capable of maintaining constant temperature
Safe reading	Direct gage length elongation measurement
See-through visibility	viewport to allow for elongation measurements and visual effect
Extremely cost-effective	less than \$2000.00 to reproduce

Wish List	
3 parallel test chambers	1 chamber likely for prototype
Autonomous elongation data collection	High accuracy (0.0005") extensometer attached to PC for data AQ
Autonomous load data collection	In-line load-cell optional
Autonomous temperature stabilization	Proportional derivative/integral controller OR PLC control

polymers were used as test samples: ABS, Derlin, and Nylon. The limited set of polymer types was taken up for avoiding excessive time consuming task and keep the focus of experimental research on learning than generating data.



Figure 4: The CNC milling jig to manufacture the dog bone specimens.  
The positions of a raw blank and a finished part are shown

## Results

To isolate the effects of temperature and applied stress, each of the three materials were tested multiple times. Each specific material was tested under various stresses and then under each stress level, at various temperatures. A sample of data plots are shown below in figures 5 through 7. Each of three plots is developed for a specific material and at a specific temperature. The varying factor was the applied stress. There is no attempt in these results to quantify the creep properties but demonstrate general trends of such. Similar data plots were developed for testing under a specific stress level and at various temperatures. The results were similar, meaning higher temperatures resulted in higher creep rates. Failure is defined when the material started to creep at an accelerated rate and before rupture. This is because excessive deformation via creep or any other method deforms the specimen beyond its functional geometry.

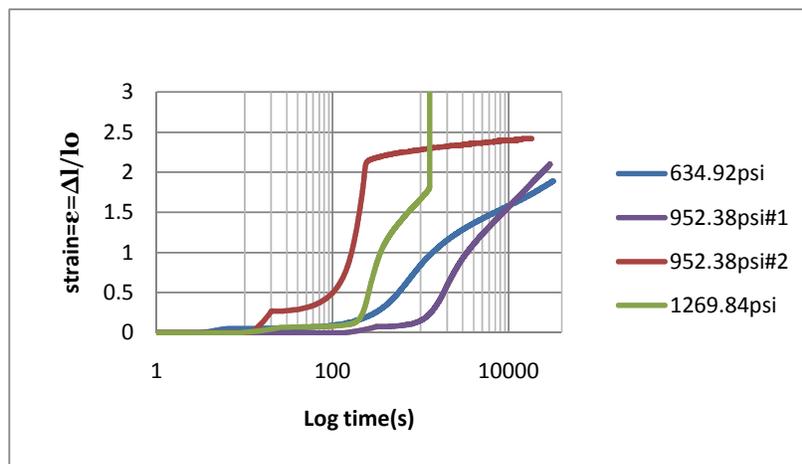


Figure 5: ABS creep plots at 200F and under various stresses.

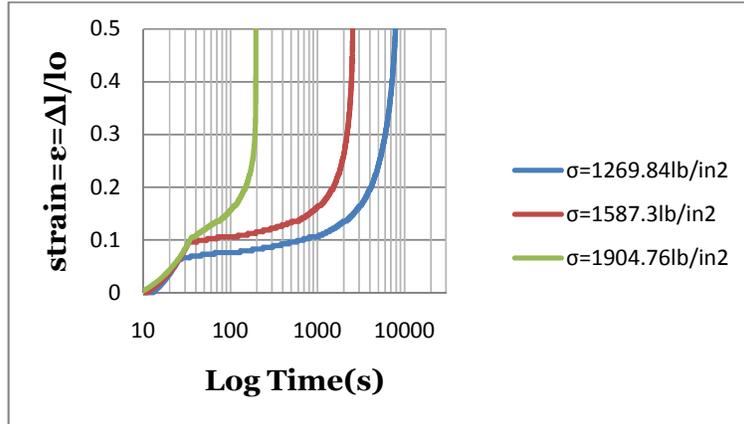


Figure 6: Derlin creep plots at 250°F and under various stresses

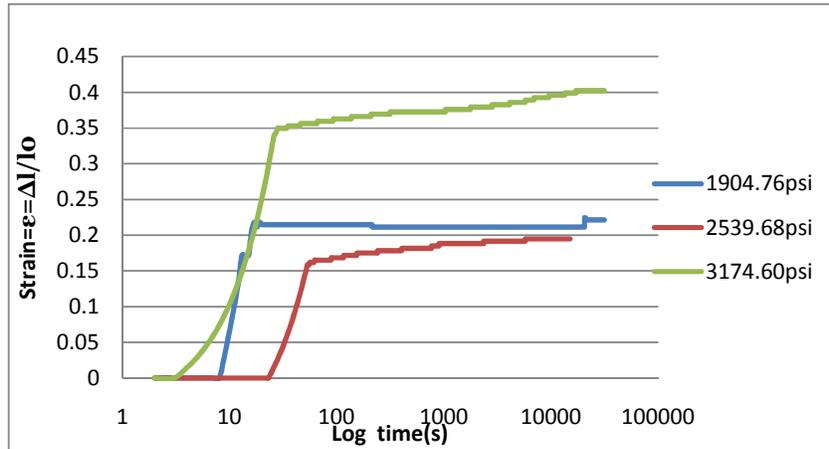


Figure 7: Nylon creep plots at 250°F and under various stresses



Figure 8: A typical sample, from left to right: undeformed, partially and then extensively crept.

Furthermore most specimens at higher temperatures did not rupture within the maximum deformation that can be accommodated within the limited size of the test chamber. In all three plots shown, both the creep rate and the life demonstrated direct correlation to stress levels. Similar correlation was evident for specimens tested at constant load/stress but at varying temperatures.

Several difficulties were encountered during the testing phase. The significant problem was posed by the non-uniformity of the temperature within the test chamber. Two high power light bulbs were used for heating but at steady state situation the temperatures were higher at the upper level compared to the bottom of the chamber. This resulted in different amount of deformation on either end of the specimen especially at higher temperatures. This could be solved by using a high temperature circulating fan inside the chamber to constantly circulate the air. The insulation was not very good and the chamber was running hot from outside posing various risks to users. Another but not the last difficulty was to monitor and record the deformation. Originally a laser beam mounted on a height gage was being used to manually monitor and record the elongation over time, which proved too tedious even for a dedicated researcher. Later a rotary transducer was available and a string under dead weight was used to transform linear elongation of the specimen into rotary motion of the transducer. Then the rotary motion was converted to linear elongation through the software for display and recording.

The minority research students used the tester and figured out most of the set up and tuning issues. They presented their efforts and findings in a student conference and won the 1<sup>st</sup> prize for research among engineering and technology oral presentations. Most of the data presented here were obtained by two undergraduate honors research students. A part of these results have been presented by these students in a poster session designed for internal dissemination of undergraduate research.

### Summary and Conclusion

Enthusiasm of some students was utilized by guiding them to design and use a high temperature creep tester. The design and manufacturing prowess of these students were demonstrated through this project. Two minority students and then two honors research students benefitted from studying and testing creep behaviors of polymers on this tester. Limited set of results were developed which clearly conformed to notions that both high temperature and higher stress directly affect thermal properties<sup>8,9</sup> of creep rate and creep life. A lot was learned about creep failure and research experimentation for creep testing. The successful design, construction and

then testing of the tester triggered high interest in materials science in general and undergraduate research in specific. The materials lab at Southern Polytechnic State University will be enhanced by the addition of this 'home made' tester that is a testimony of our students' achievement. A set of improvements were suggested to augment the functionality of the tester, that will be the challenge for the next design group.

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### Bibliography

1. Callister, William D. Material Science and Engineering an Introduction. 7th ed. John Wiley & Sons, Inc., 2007.
2. Creep (deformation) -[http://en.wikipedia.org/wiki/Creep\\_\(deformation\)](http://en.wikipedia.org/wiki/Creep_(deformation))
3. Martin Tarr, "Stress and its effect on materials-Creep," [http://www.ami.ac.uk/courses/topics/0124\\_seom/index.html#4](http://www.ami.ac.uk/courses/topics/0124_seom/index.html#4), University of Bolton, UK.
4. Shogo Saito, Tatsuji Nakajima, "Glass Transition in Polymers," *Journal of Applied Polymer Science*, Vol 2, Issue 4, pp-93-99, 1959.
5. J. A. Forrest, K. Dalnoki-Veress, and J. R. Dutcher, "Interface and chain confinement effects on the glass transition temperature of thin polymer films," *Physical Review E*, Volume 56 » Issue 5
6. Gregory B. McKenna, "On the physics required for prediction of Long term performance of polymers and their composites," *Journal of Research of the National Institute of Standrad and Technology*, Vol 99, No. 2, March-April 1994.
7. Momoh et. Al., "Development of a low cost mechanically operated tensile and creep testing machine," *Journal of Engineering and Applied Sciences*, Vol3, issue 6, pp 491-495, 2008.
8. Godovsky, Y K , "Thermophysical Properties of Polymers," Springer-Verlag (Germany), 1992, pp. 320, 1992.
9. *Engineered Materials Handbook- Desk Edition*, ASM International, 1995.