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Characterization of a Structural Adhesive in Automotive Environments

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ABSTRACT

In order to achieve better fuel economy without sacrificing vehicle size, the automotive industry is motivated to utilize advanced materials, such as polymer composites, in primary structural components. When polymer composite structures are used, adhesive joining is often an attractive alternative to traditional fastening techniques and may in some cases be an enabling technology. However, successful integration of adhesively bonded materials in structural applications requires a thorough understanding of material performance.

This research effort addresses characterization methods for adhesives emphasizing the durability of the material with exposure to the aggressive environments that may be encountered in typical automotive applications. Specifically, test method selection and fixture design for submerged tests will be discussed. Data will be presented for a candidate epoxy adhesive material. Studies include tensile, fatigue, and creep characterization at 21°C, 90°C, and –40°C and with exposure to brake fluid, distilled water and alcohol.

INTRODUCTION

Previous works [1,2,3] have addressed the experimental methods and analysis techniques to characterize the fracture toughness of adhesive joints in non-aggressive environments. These studies considered the joining of combinations of composite and metallic adherends, employing a candidate automotive adhesive, BFG582E, developed by SIA Inc. a subsidiary of Sovereign Specialty Chemicals (formerly B.F. Goodrich). This work emphasized the need to develop a complete understanding of the performance of the adhesive system which can contribute greatly to the overall toughness of the joint.

The current work extends the previous studies of the BFG582E adhesive system by considering the affects of several aggressive environments on mechanical performance with particular attention paid to time-dependent material response.

MECHANICAL TESTING

For all tests, dog-bone type specimens were machined from 0.125 in (3.175 mm) thick cast epoxy panels employing a Tensile-Cut router/template. Specimen dimensions were 9 x 0.75 x 0.125 in (225 x 18.75 x 3.175 mm) with a 0.5 in (12.7 mm) reduced gage section width. A typical specimen is shown in Figure 1.

Environmental conditions consisted of three temperature levels, 21°C, 90°C, and –40°C, in addition to testing at room temperature after 1000 hours of immersion in automotive fluids. For the longer duration immersed specimen tests (low stress level fatigue and creep), custom in-house designed submerged environmental chambers were employed to avoid desorption of fluids (see Figure 2). For short-term tests lasting less than several hours (tensile and high stress level fatigue) specimens were tested immediately after removal from the fluids in air. The room temperature, hot and cold tests were conducted in lab-air (liquid nitrogen cooled). A summary of the complete test matrix is given in Table I. A typical hot/cold test chamber designed and built in-house is depicted in Figures 3 and 4. This versatile chamber was designed specifically to minimize the volume within the chamber to obtain better temperature control and more economical liquid nitrogen consumption during cold tests.

Testing was carried out on in-house designed and fabricated servo-hydraulic test machines consisting primarily of MTS components. Typical capacities for the various test machines consisted of 1-10 kip (4.448-44.48 kN) load ranges, 0.5-3 inch (12.7-75 mm) stroke ranges with 11 kip (48.9 kN) actuators.

For all tests, extensometers were used to measure strain. For the submerged conditions, it was necessary to employ spring loaded MTS 632.51B-01 extensometers (25 mm gage length) with quartz rod attachment arms which can be inserted through liquid tight ports on the environmental chambers. Providing a seat for the extensometer rods on the specimen surface was accomplished by attaching nylon washers or indented metal-epoxy targets to the specimens within the gage length. This strain measurement technique was designed to avoid damaging clip-gage type extensometers from exposure to the aggressive fluids. For the hot and cold tests, the more common clip-style extensometers (MTS 634.11E-25 and 634.11E-20) were employed with three-point knife edge mounting and spring clips.

Data acquisition, control and function generation for the various tests were accomplished using a combination of MTS 407 servo-hydraulic controllers with the remote serial control option in conjunction with National Instruments PCI-MIO-6031E data acquisition boards.

TENSILE TESTS

For each set of tests at a different environmental condition, a new series of room temperature (21°C) baseline tensile tests were conducted for comparison with the environmental results. Since there can be some variability in material properties among the various batches of specimens, running these additional tests provided a means to identify any significant changes in material properties due to manufacturing techniques, and avoid attributing these differences to environmental exposure. Specifically, the hot, cold, and room temperature tests were conducted from a single batch of specimens, while the immersed tensile tests were conducted using a second batch of specimens with their corresponding baseline tests conducted prior to fluid exposure.

Tensile tests were carried out in stroke control at a displacement rate of 0.05 in/min (1.27 mm/min). Approximately 6-8 specimens were tested at each condition to access material reproducibility. Ultimate strength, stiffness and strain at ultimate strength were calculated from the stress-strain record.

The tensile results for the three different temperature levels are shown in Figure 5. It can be seen that tensile strength decreases significantly with increasing temperature, while strain to failure increases. Interestingly, the strain to failure in the cold condition is not significantly lower than that at room temperature.

Similar plots for the immersed adhesive specimens are shown in Figures 6, 7, and 8. The results for the fluid exposure reveal an increase in strain to failure accompanied by a loss of strength. Surprisingly, the most noticeable change from baseline data is observed for the specimens exposed to methanol, and not brake fluid which is considered the more aggressive environment.

The tensile results for all conditions are listed in Table II, and comparative bar graphs for stiffness and strength are shown in Figures 9 and 10.

FATIGUE TESTS

A study of fatigue behavior was undertaken to determine the effects of the various environments on specimen life. All tests were run in load control with R-ratios (minimum stress amplitude/maximum stress amplitude) of 0.1 at a frequency of 1HZ (to avoid specimen heating). Fatigue stress amplitudes were based on percentages of ultimate strength from tensile tests for a given environmental condition. Since the ultimate strengths for each environment varied considerably, the S-N data was also plotted as a function of absolute applied stress in addition to percentage of ultimate strength. In general, there is quite a bit of scatter in the fatigue life data, therefore numerous replicate tests were run at each stress level for each condition.

Fatigue life, S-N curves, for the various environmental conditions are plotted in Figures 11a and 12a as percentages of ultimate strength, and as a function of absolute applied stress in Figures 11b and 12b. Least squares fits were overlaid for each data set.

From Figures 11a and b, it can be seen that the fatigue life of the adhesive with respect to percentage of ultimate strength for a specific temperature increases with temperature. This agrees with the increased strain to failure observed in the tensile tests at higher temperatures Additionally for the three temperatures considered, the relative scatter is more pronounced for the cold tests at –40°C, to the extent that S-N curves for the cold and room temperature tests overlap at lower stress levels. This could indicate that the adhesive is exhibiting some degree of embrittlement at lower temperatures. However the strain to failure from the tensile tests for both the cold and room temperature tests were in close agreement, which is not descriptive of a brittle condition. This contradiction may be explained by considering the effect temperature may have on the time dependent behavior of the material.

For the submerged fatigue specimens, the S-N curves for the various conditions overlap quite significantly, making it difficult to make a clear distinction between the three sets of environmental data over the entire range of applied stress. Nevertheless, from Figure 12a, the general trend is an increase in fatigue life when viewed by percentage of ultimate applied stress for a particular immersed case. This agrees with the increased ductility

observed in the tensile data for the immersed specimens.

CREEP TESTS

Creep tests were conducted in the same temperature and fluid environmental conditions as the tensile and fatigue tests. Applied stress levels during creep were based upon 20, 40, and 60% of tensile strength for each condition. The creep loading history consisted of one day of creep, maintaining a constant applied stress followed by three days of recovery at zero load to measure strain recovery and any permanent deformation present in the adhesive.

In–house designed creep testing systems were used since they have several novel features which are lacking in dead-weight systems typically employed for this type of testing. Because the adhesive system exhibits significant permanent elongation for the higher stress level creep tests after creep loads are removed, it is important to achieve a zero load condition during recovery in order to record the permanent deformation during the recovery period. Additionally, it is necessary to run tests in a very consistent manner to minimize scatter between replicate tests. With this in mind, servo-hydraulic test machines in conjunction with an automated data acquisition and control software package were employed. This system ensures that replicate tests have identical load-time histories. Furthermore it is possible to control the loading and unloading rates in a precise manner during the critical periods at the start and end of the creep loading.

Plots of the strain-time history for the creep tests are given in Figures 13-18. From the first three figures, it can be seen that there is a significant variation in strain for the three temperature test conditions during creep. This behavior indicates the time dependent sensitivity of this adhesive to temperature. It is important to keep in mind the large difference in strain values occur in spite of the fact that the applied stresses for the three different conditions were percentages of the tensile strengths at each temperature level. For example, although the creep stress levels at 90°C are about half the value of the room temperature tests, the corresponding strain levels are more than double (Figures 13-14 and Table II). This behavior is consistent with the cold test results. Comparing Figure 15 with Figure 13, it can be seen that although the stress levels are greater than those at room temperature and the resulting strain is lower. It should be noted that for several of the cold tests plotted in Figure 15 there are jumps in the strain during the recovery portion of the tests. This was caused by a loss in cooling, and should not be confused with material behavior.

Another interesting observation that should be noted is the creep data for the submerged specimens also resulted in significantly larger strain levels during creep than in the baseline tests. In the cases of the 60% tests for methanol and brake fluid, the specimens consistently attained large enough strain values to fail the specimens before completing the day long creep period.

CONCLUSION

This study has shown that the environmental conditions considered have significant effects on the material behavior of this adhesive system.

Tensile tests reveal that temperature and fluid exposure play a significant role in measured strength, stiffness and ductility. These changes in material performance are consistent with the behavior of the adhesive during fatigue and creep testing. For example, the apparent increase in ductility with increasing temperature and after immersion result in longer lives in fatigue and larger strain levels in the creep tests.

Although this work included a fairly large test matrix, there is a need for additional data to deal with the scatter inherent with the adhesive. In particular, more fatigue tests need to be conducted for the submerged specimens.

ACKNOWLEDGMENTS

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Figure 1. Cast Epoxy Dog-Bone Specimen

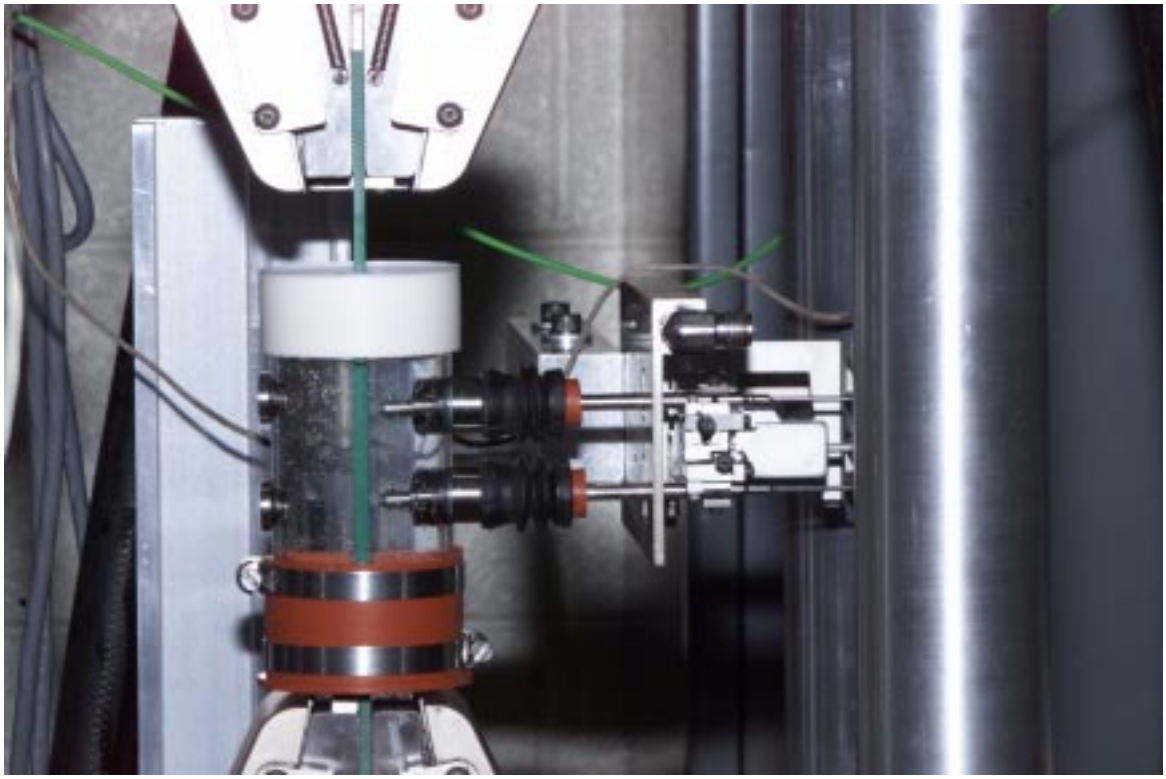


Figure 2. Immersed Specimen Chamber

Test Type	Number of tests	Pre-Soak Duration	Test Temperature	Comment
TENSILE TESTS				
Baseline 1 (Rm. Temp.)	6	N/A	21°C	
90°C	7	N/A	90°C	
-40°C	8	N/A	-40°C	
Baseline 2 (Rm. Temp.)	27	N/A	21°C	9 tests for each fluid
Distilled Water	9	1000 hours	21°C	Tested Unimmersed
Methanol	9	1000 hours	21°C	Tested Unimmersed
Brake Fluid	9	1000 hours	21°C	Tested Unimmersed
FATIGUE TESTS				
Room Temp.	21	N/A	21°C	
HOT	35	N/A	90°C	
COLD	32	N/A	-40°C	
Distilled Water	17	1000 hours	21°C	Tested Immersed*
Methanol	11	1000 hours	21°C	Tested Immersed*
Brake Fluid	20	1000 hours	21°C	Tested Immersed*
CREEP TESTS				
Room Temp.	18	N/A	21°C	
HOT	13	N/A	90°C	
COLD	14	N/A	-40°C	
Distilled Water	14	1000 hours	21°C	Tested Immersed
Methanol	12	1000 hours	21°C	Tested Immersed
Brake Fluid	17	1000 hours	21°C	Tested Immersed
* Short duration fatigue tests (high stress levels) were tested unimmersed				

Table I. Test Matrix

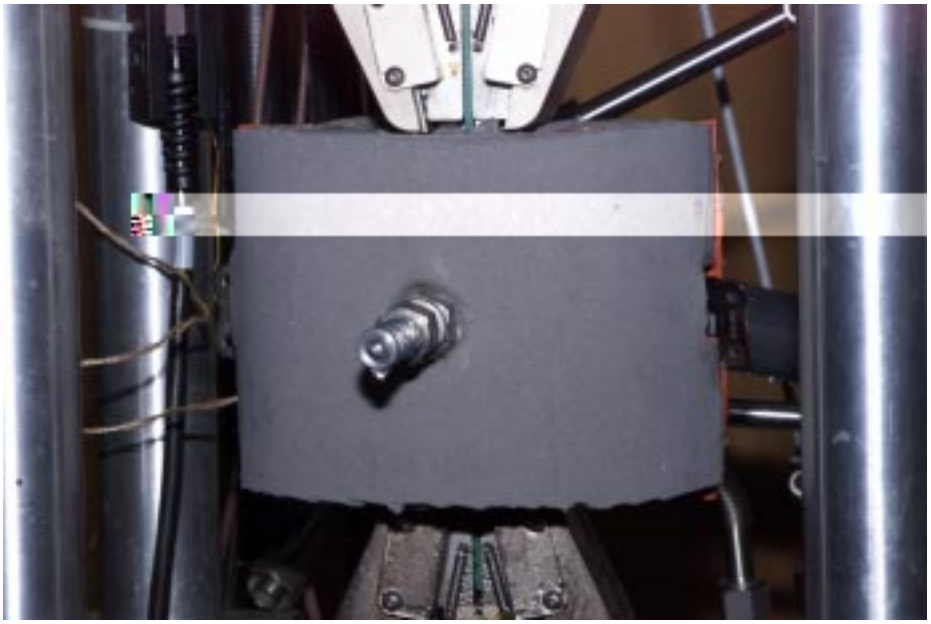


Figure 3. Cold Test Environment Chamber

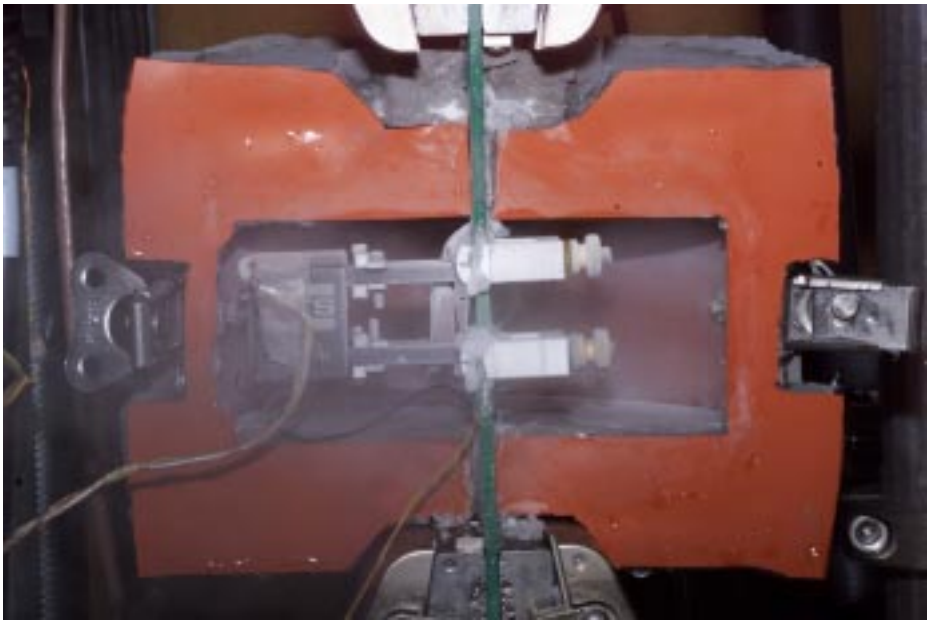


Figure 4. Cold Test Environment Chamber Interior

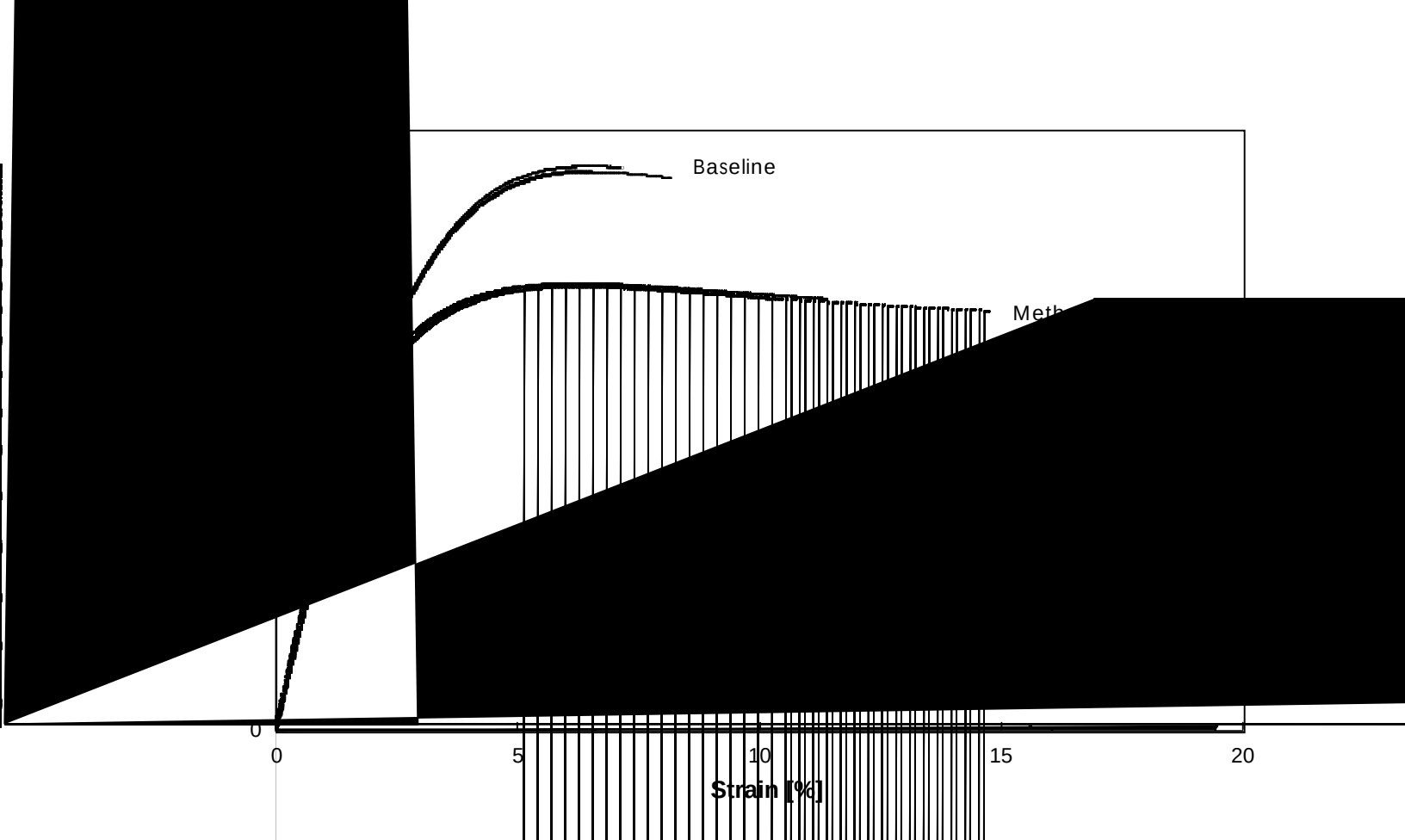


Figure 7. Tensile Tests After 1000 Immersion in Methanol

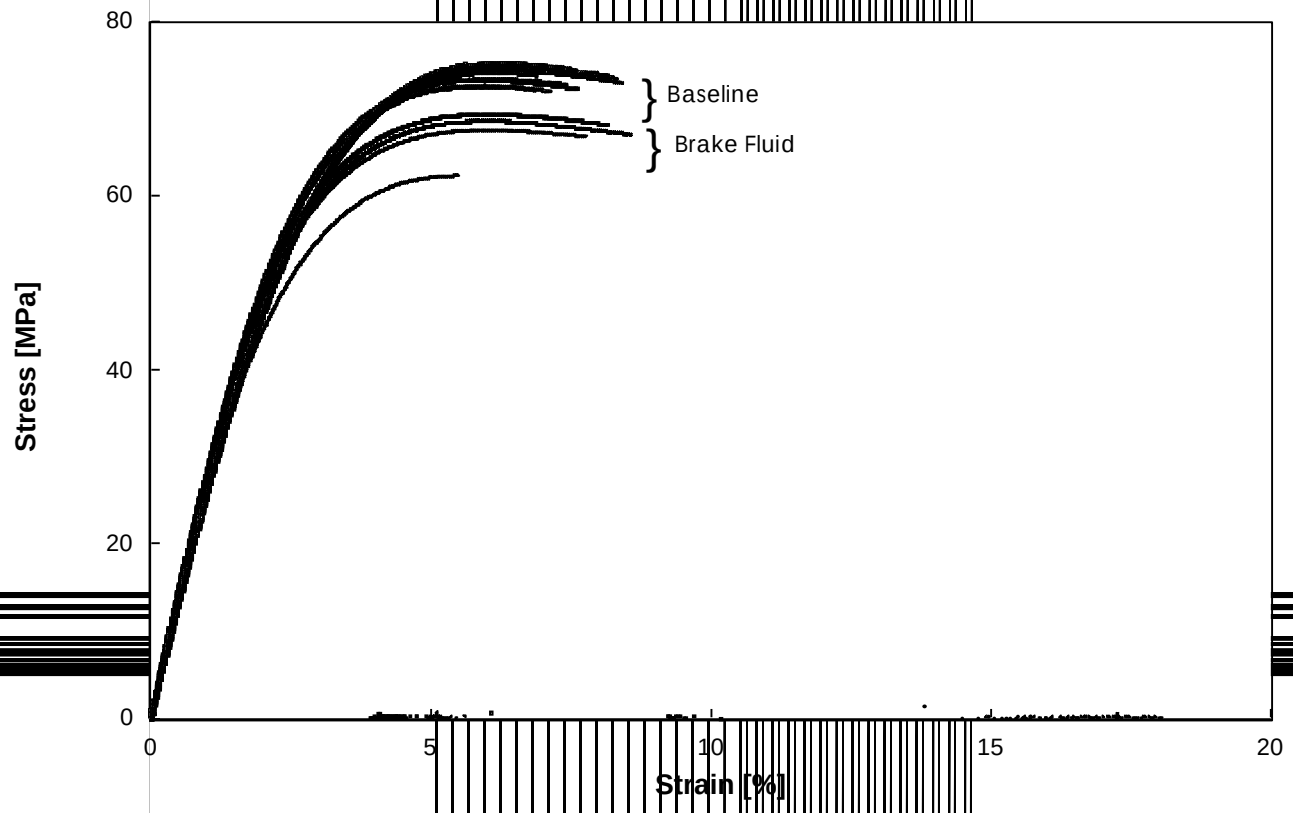


Figure 8. Tensile Tests After 1000 Immersion in Brake Fluid

Condition	Number of tests	Strength (MPa)	Stiffness (GPa)	Strain (%)
Baseline1	6	84.8 ± 2.8	3.13 ± 0.03	4.81 ± 0.71
90°C	7	44.4 ± 1.5	2.10 ± 0.04	3.69 ± 0.10
-40°C	8	101.6 ± 8.0	3.59 ± 0.10	4.79 ± 0.81
Baseline2	27	74.6 ± 1.2	2.59 ± 0.03	6.17 ± 0.40
Distilled Water	9	61.8 ± 0.7	2.79 ± 0.08	6.72 ± 0.17
Methanol	9	59.1 ± 1.9	2.72 ± 0.04	5.61 ± 1.06
Brake Fluid	9	71.4 ± 2.2	2.78 ± 0.08	5.87 ± 0.26

Table II. Summary of Tensile Test Results

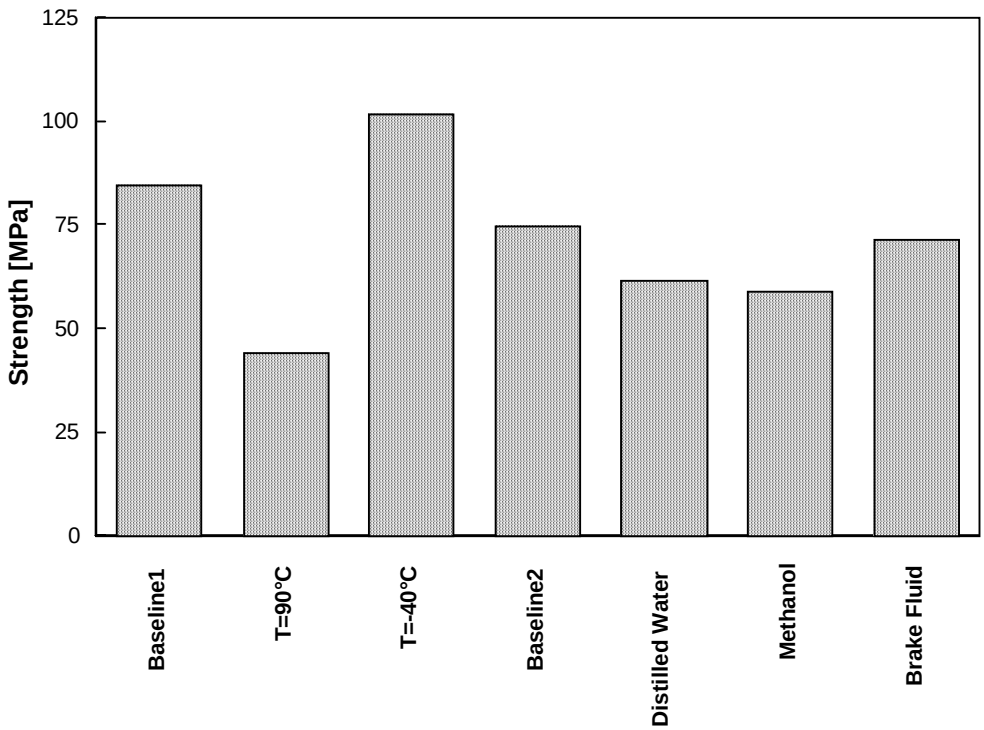


Figure 9. Tensile Strength for Different Environmental Conditions

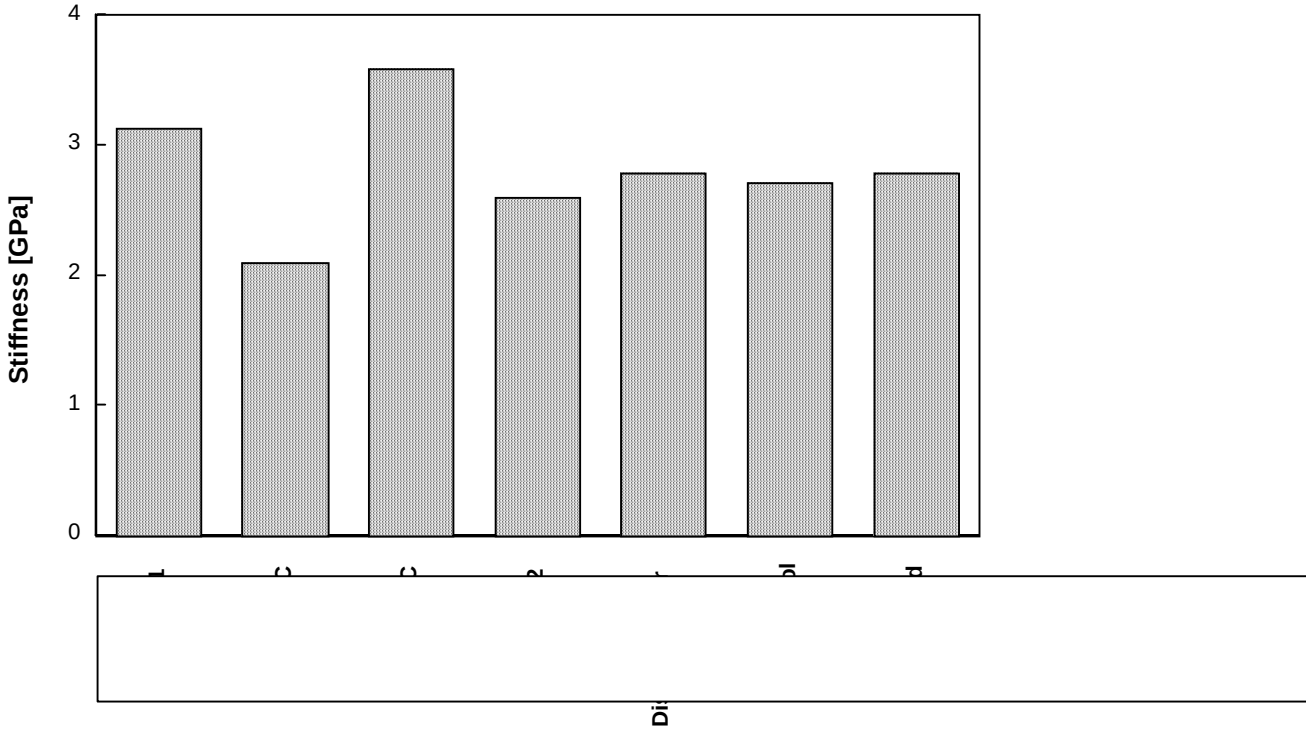


Figure 10. Tensile Stiffness for Different Environmental Conditions

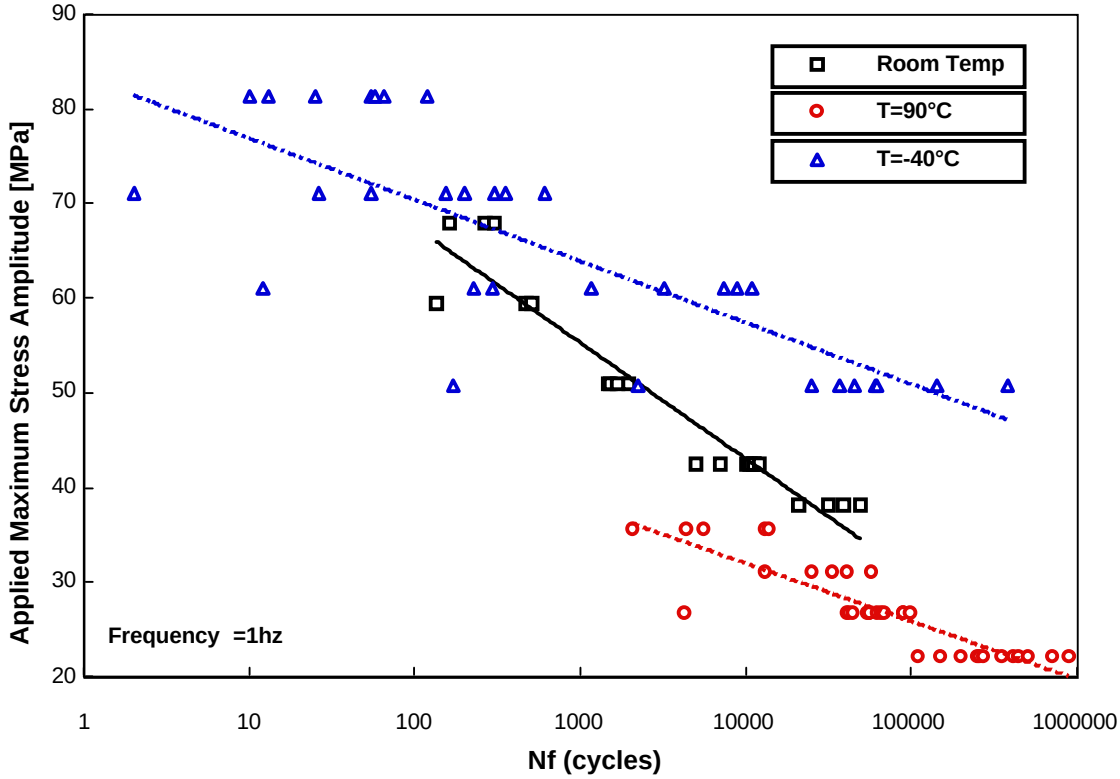


Figure 11b. S-N Data for Room Temp, Hot and Cold FatigueTests

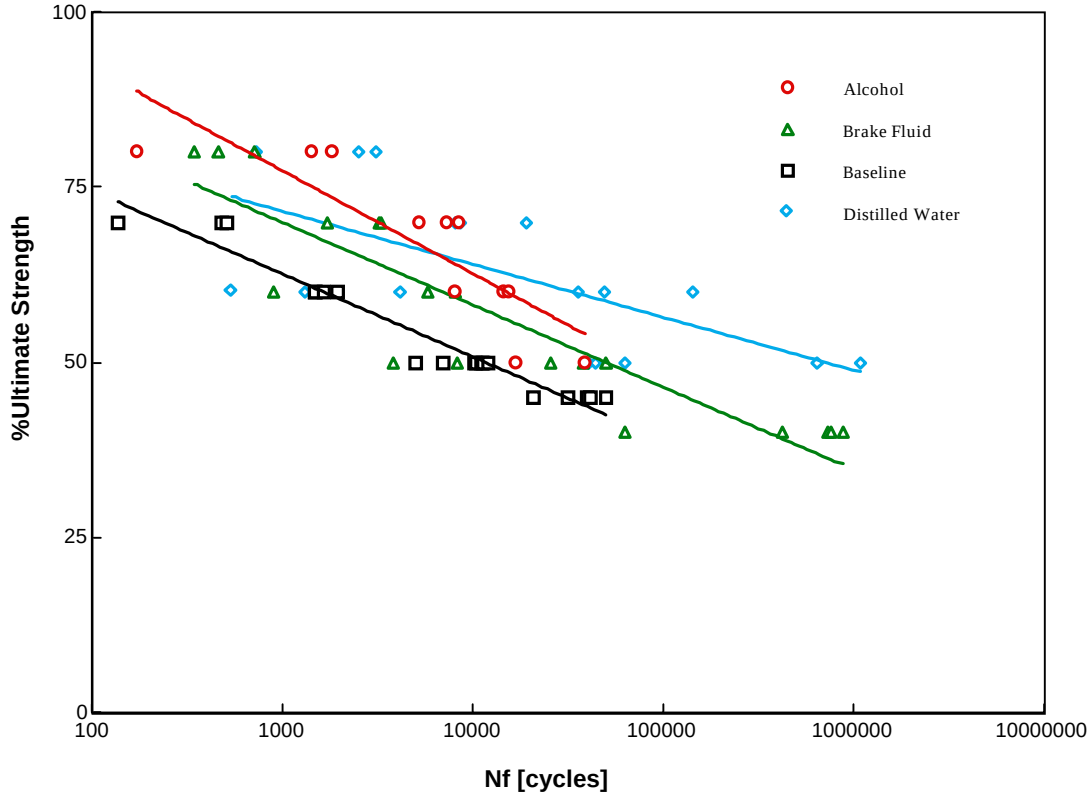


Figure 12a. S-N Data for Immersed Fatigue Tests

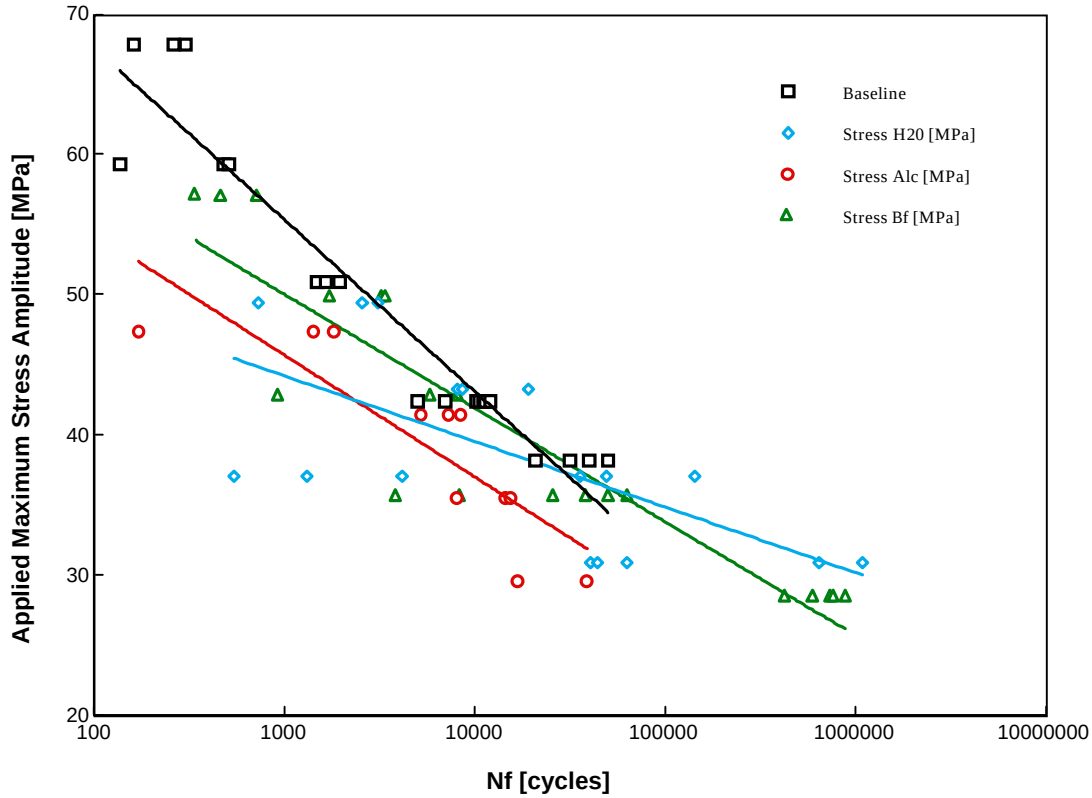


Figure 12b. S-N Data for Immersed Fatigue Tests

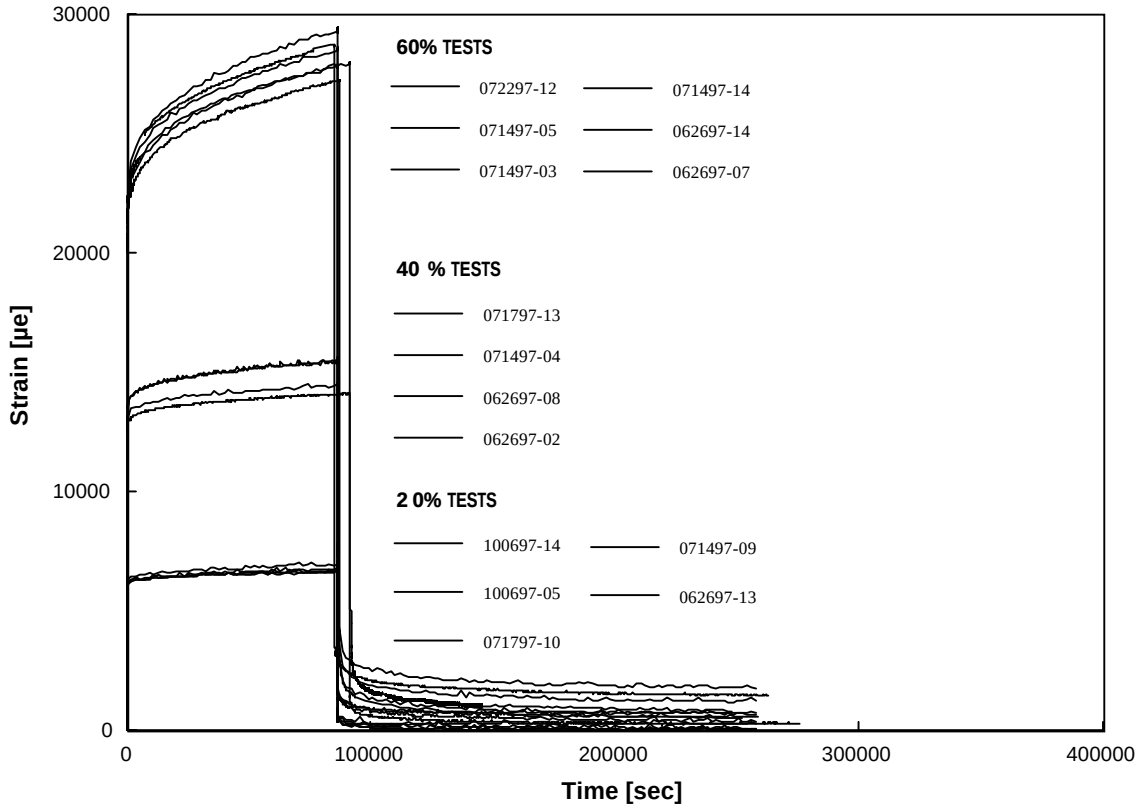


Figure 13. 20,40, and 60% Room Temperature Creep Tests

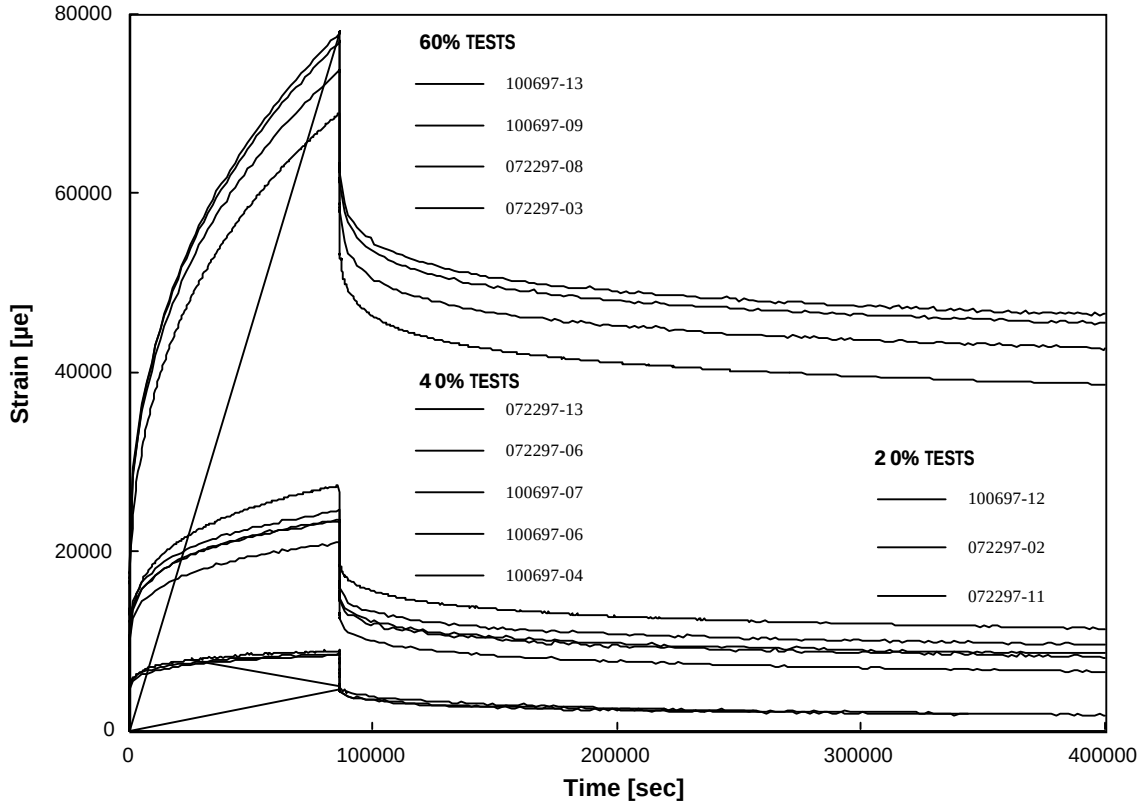
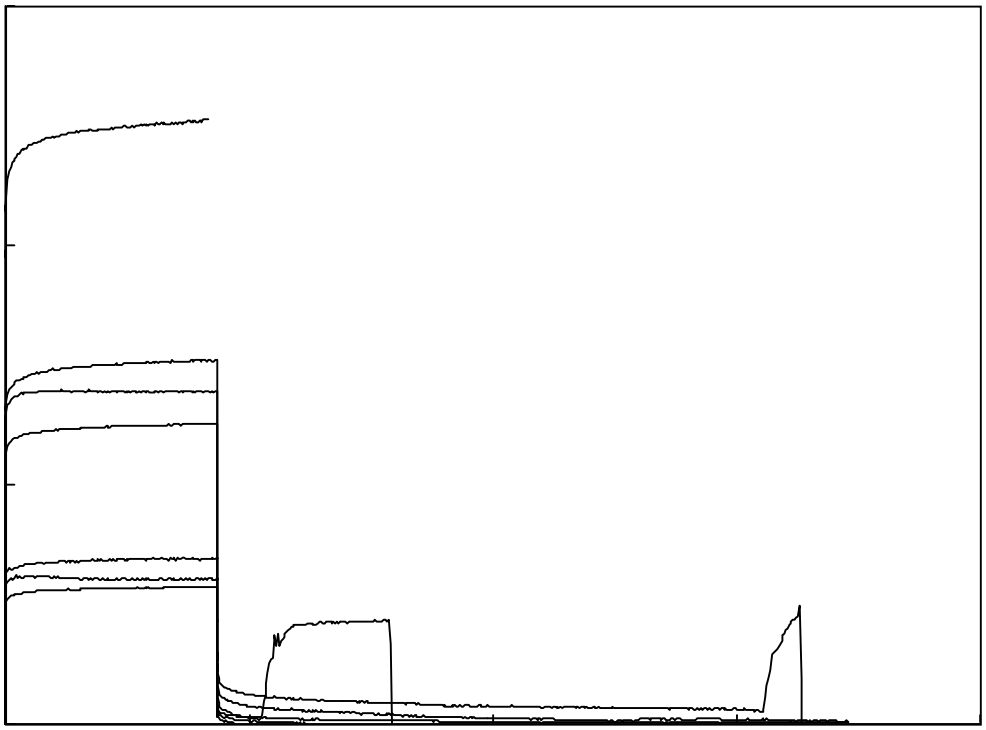


Figure 14. 20,40, and 60% Creep Tests at 90°C



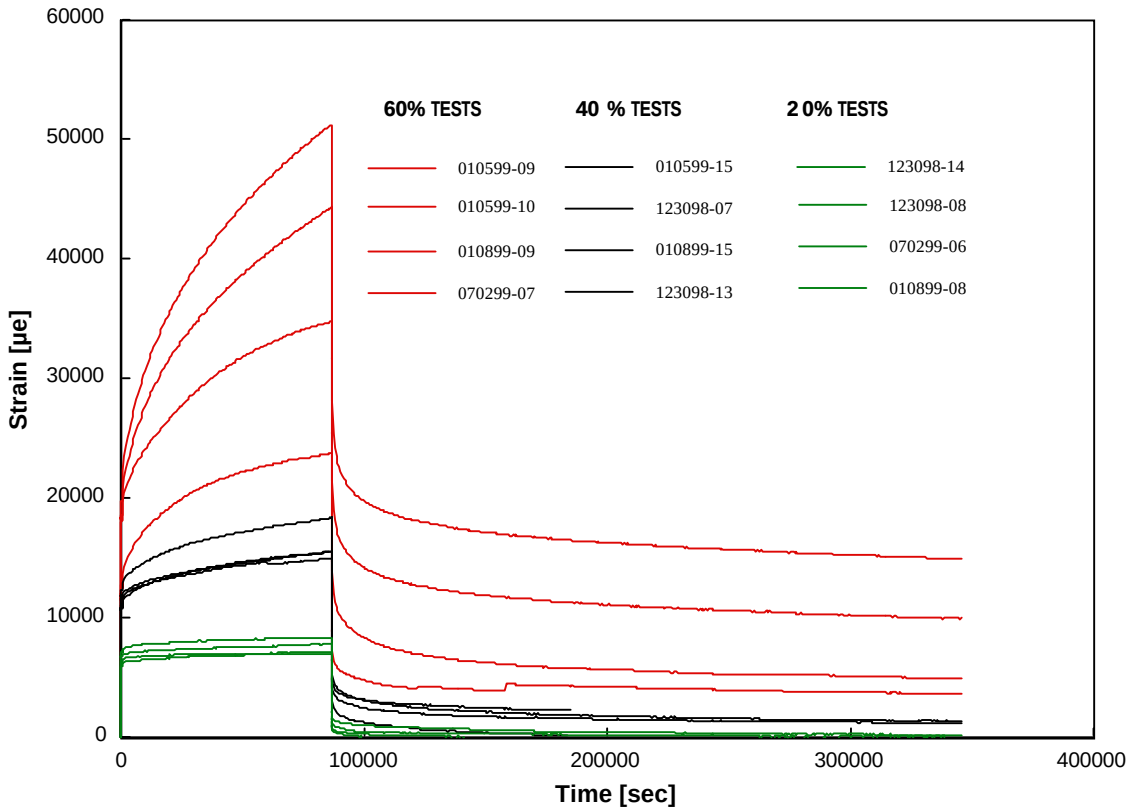


Figure 16. 20,40, and 60% Creep Tests After 40 Days Immersion in Distilled Water

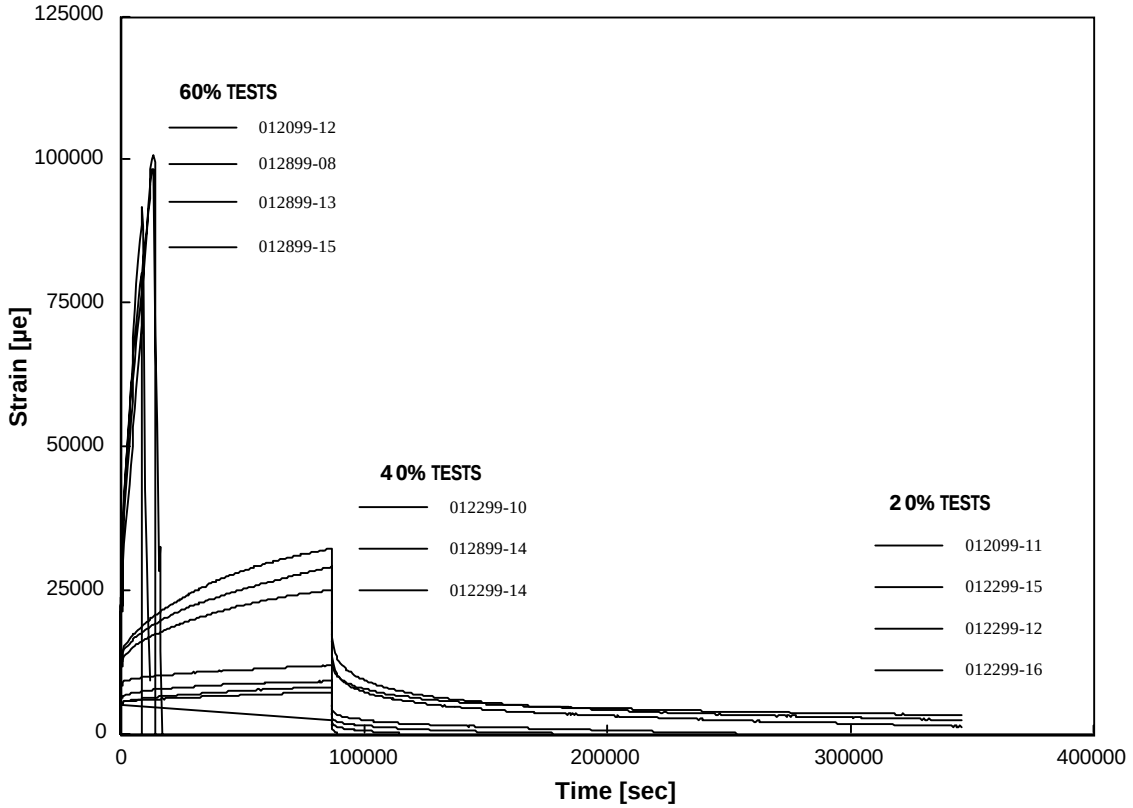


Figure 17. 20,40, and 60% Creep Tests After 40 Days Immersion in Methanol

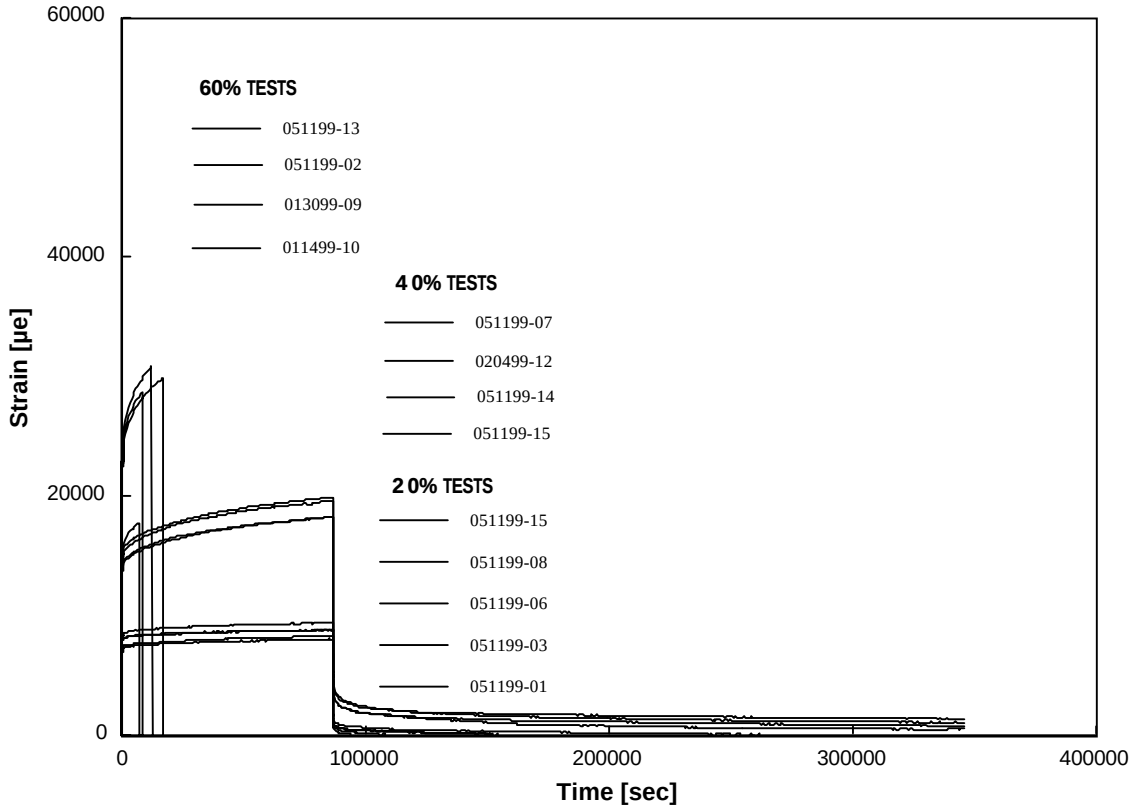


Figure 18. 20,40, and 60% Creep Tests After 40 Days Immersion in Brake Fluid