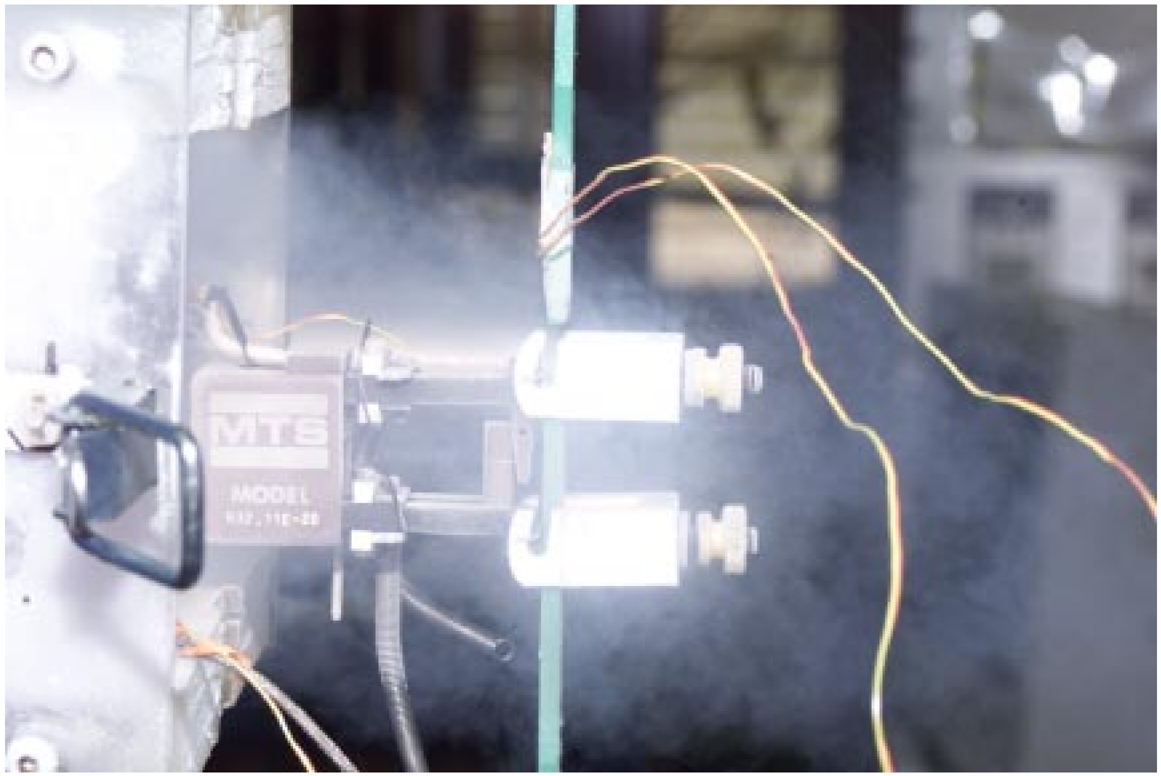
Characterization of a Structural Adhesive in Automotive Environments



Don Erdman, Rick Battiste, Ray Boeman, and Lynn Klett

Adhesive Joining Project
Oak Ridge National Laboratory

2000 Future Car Congress

Characterization of a Structural Adhesive in Automotive Environments

Adhesive Joining Project Overview

•Joint effort between DOE’s Lightweight Vehicle Materials Program and United States Automotive Materials Partnership (USAMP)

•Project executed by Automotive Composites Consortium (ACC) Joining Group, **Oak Ridge National Laboratory (ORNL),** suppliers and subcontractors.

Characterization of a Structural Adhesive in Automotive Environments

Why Use Adhesive Bonding?

•Polymer composites are considered as candidate materials to help reduce passenger vehicle weight for better fuel efficiency.

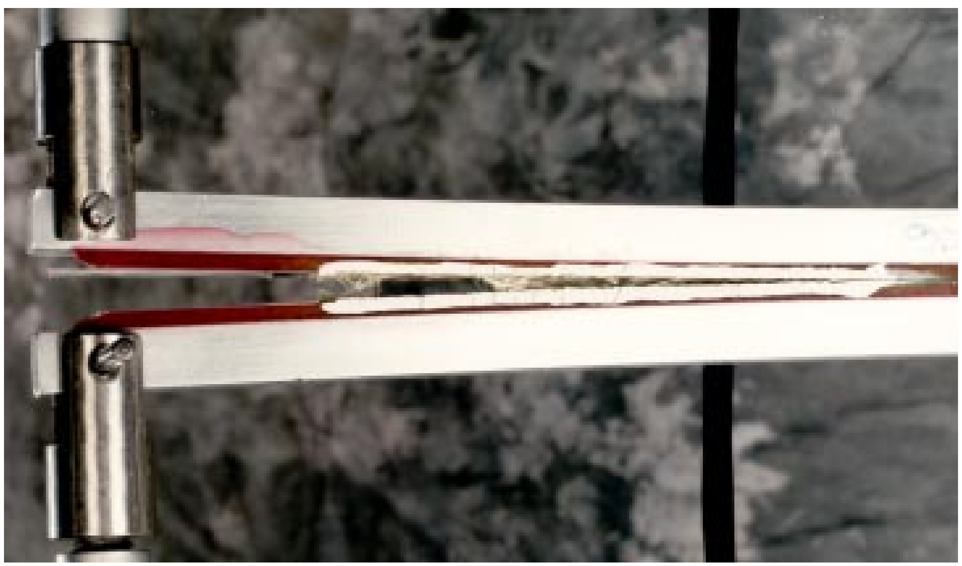
•Adhesive bonding is an enabling technology for composite materials since traditional fasteners used with metals (welds, rivets, bolts, clinching, etc.) are not appropriate.

Characterization of a Structural Adhesive in Automotive Environments

<u>Adhesive Joining Project Primary Objectives:</u>

•Characterization of fracture behavior of adhesive joints with various adherand combinations (composite-metals).

•Develop standardized test methods and automated test procedures to evaluate joint toughness.

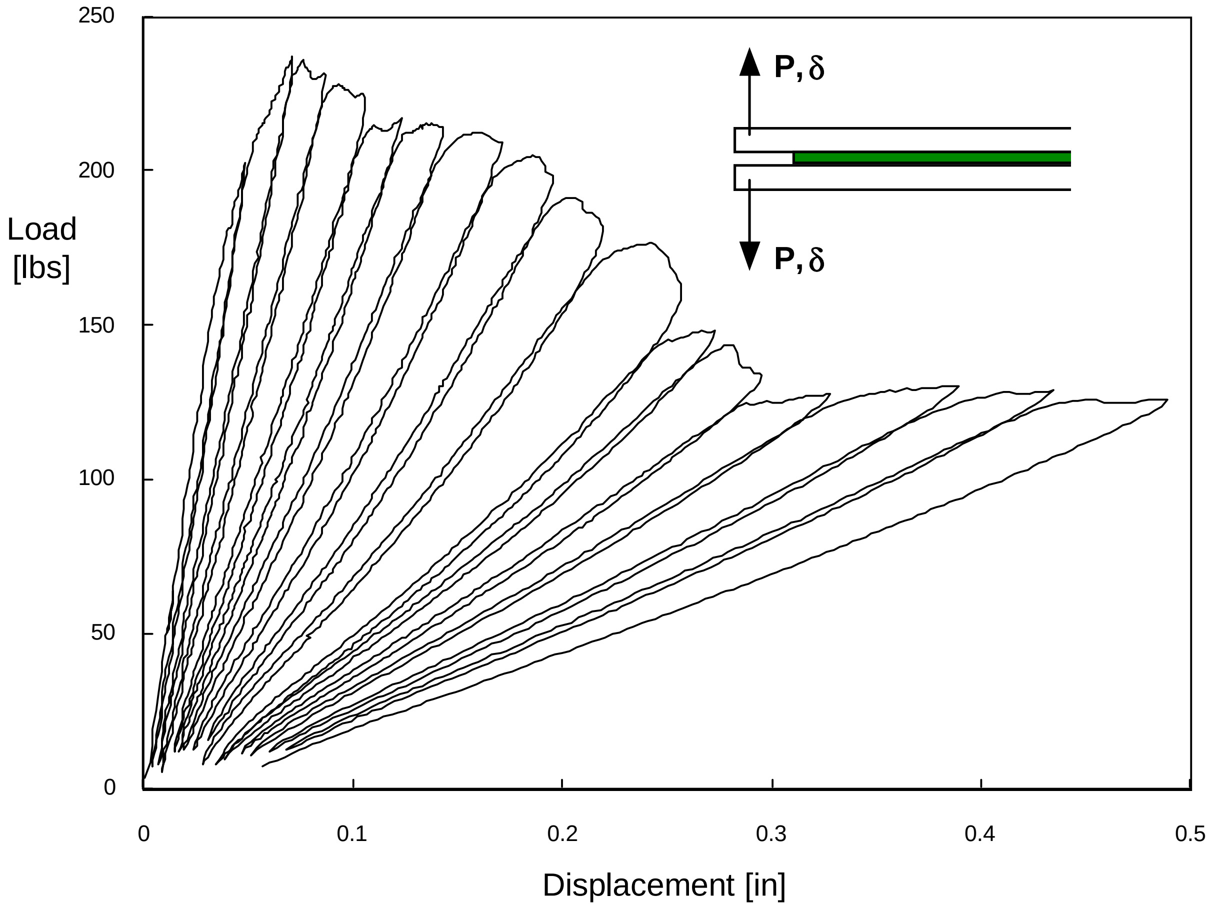


Mode I DCB Test

Mode II ENF Test

Characterization of a Structural Adhesive in Automotive Environments

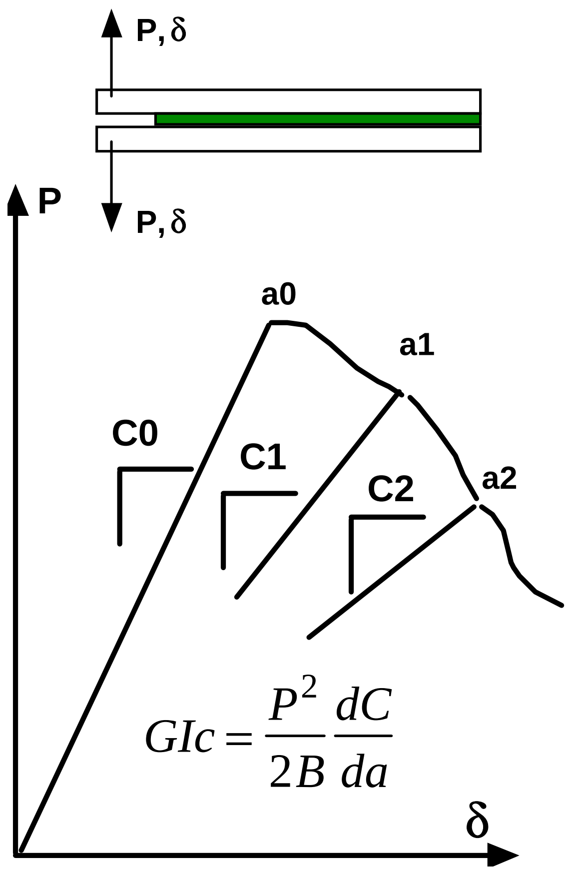
Typical mode I load-displacement curve for a composite-epoxy-composite adhesive joint.

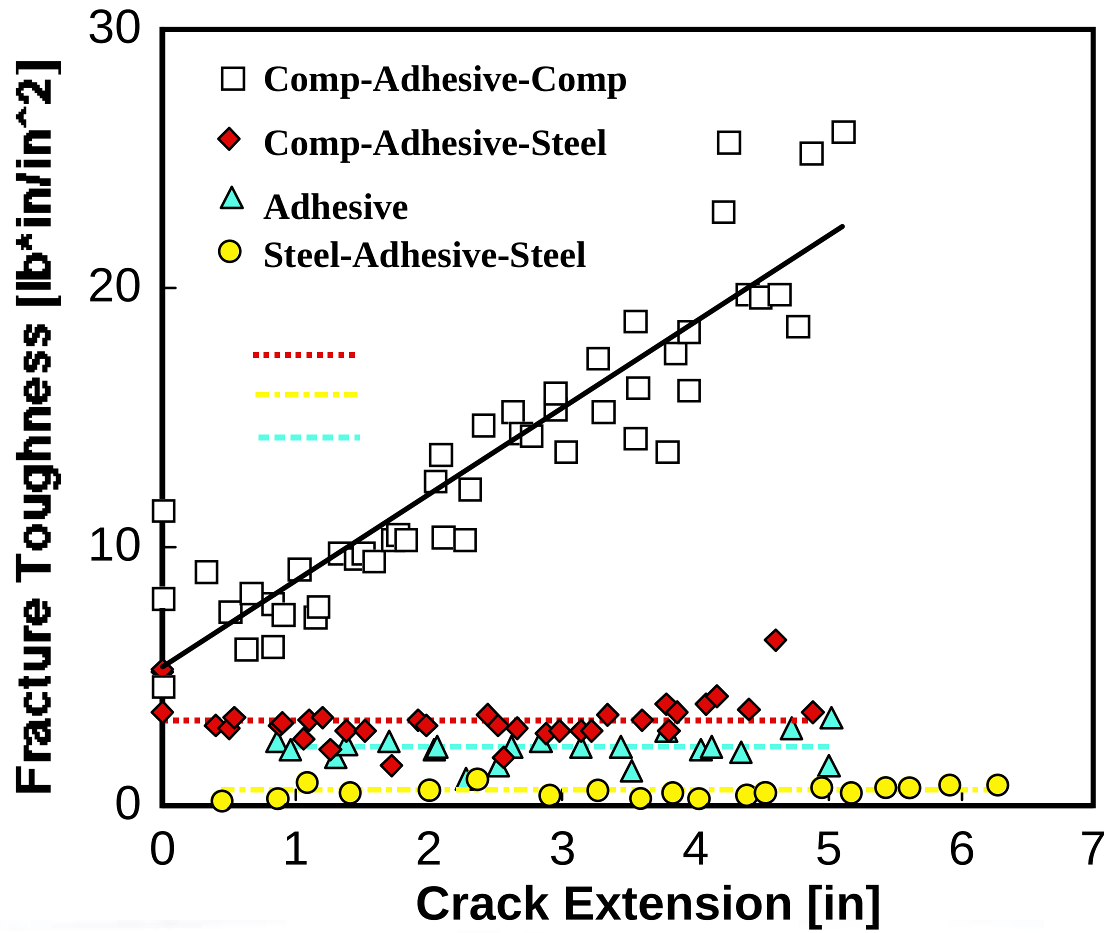


-Hysteresis, non-linearity of loading-unloading curves and permanent deformation are generally attributed to fiber bridging.

-Additionally, adhesive behavior may contribute to the load-displacement response of the joint.

Characterization of a Structural Adhesive in Automotive Environments





Characterization of a Structural Adhesive in Automotive Environments

Characterization of Environmental Effects on Adhesives and Adhesive Joints

Environmental Conditions

•Cold (-40°C), Hot (90°C), Room Temp.
•Exposure to automotive fluids (immersion for 40 days): methanol, distilled water or brake fluid.
•Long term tests (fatigue and creep) conducted in submerged chambers to prevent desorption of fluids.

Adhesive Tensile Testing

•Strength, Stiffness, Elongation

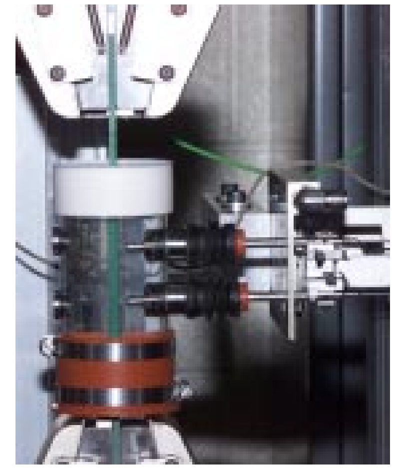
Adhesive Creep Testing

•Time dependent material response
•Non-Recoverable Strains

Adhesive Fatigue Testing

•Fatigue Life-SN Curves
•Damage Accumulation/Progression

Cold Test Chamber (T = -40°C)



Submerged Test

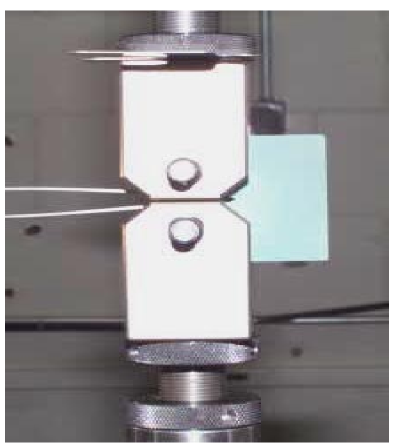
Fracture Testing

Adhesives

- Mode I Compact Tension: KIc of Cast Adhesive Specimens
- Lap-Shear: Comparative Screening Test
- Hat Section Structural Test

Adhesive Joints

-Modes I, II and Mixed Mode
-Joint Adherends:
  Composite/Composite
  Composite/Steel(E-coat)
-Composites:
  Swirled Glass Mat/Urethane
  Carbon Fiber/Urethane



Mode I KIc Test

CT Fracture Surface

Mode I Adhesive Joint Test (T = -40°C)

Characterization of a Structural Adhesive in Automotive Environments

Adhesive Specimens

•Material : BFG582E : experimental adhesive developed by SIA Inc. a subsidiary of Sovereign Specialty Chemicals (formerly B.F. Goodrich)

•Tensile Specimen Geometry:

- Flat dog-bone specimen machined (Tensile Cut Router) from cast panels.
- Dimensions: 9” x 0.75” x 0.125” with 0.5” width gage section.

•Compact Specimen Geometry:

- Standard ASTM 1-T specimen machined from a thick cast adhesive block.

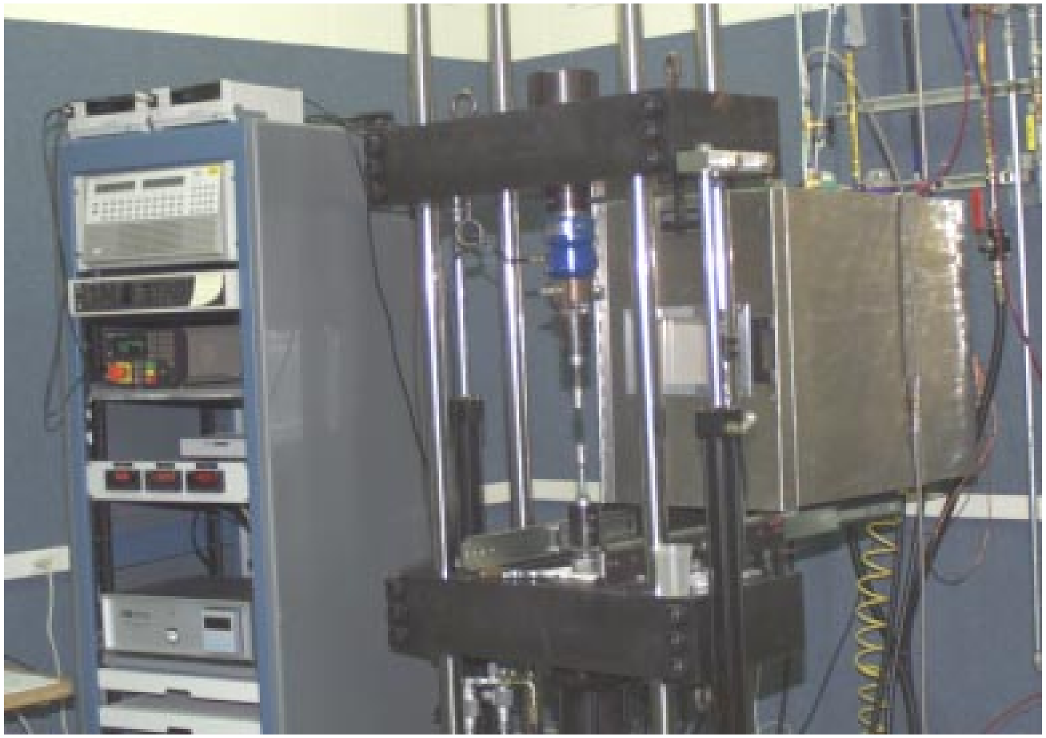
<u>Testing Systems</u>

-MTS and In-House Custom Built Servo-Hydraulic Test Machines
(systems designed or retrofitted for specific testing task at hand)

- **Capacities** : 1-220 kip load ranges, 1/2 - 8 inch stroke ranges
- **Environments** : Cold, Hot, (-130° to 400°C), Submerged
  - ATS standard ovens/environmental chambers
  - In-house specialty environmental chambers

- **Servo-Controllers :** MTS 407 with remote serial capability
- **Extensometers :** MTS (0.5-15%)
- **AD/DA:** National Instruments DAQ and signal conditioning boards (thermocouple,strain gages,etc.)
- **Computers :** PCI based Macintosh and/or PC’s

Characterization of a Structural Adhesive in Automotive Environments



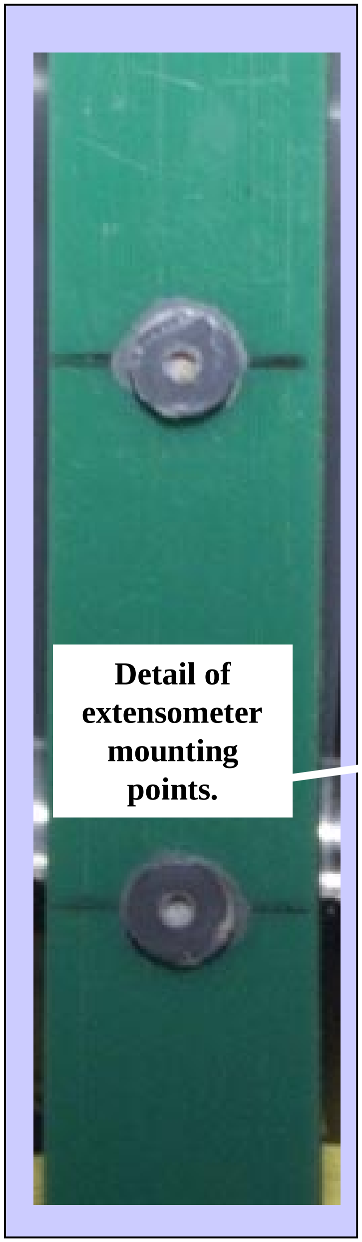
In house designed servo-hydraulic test machines

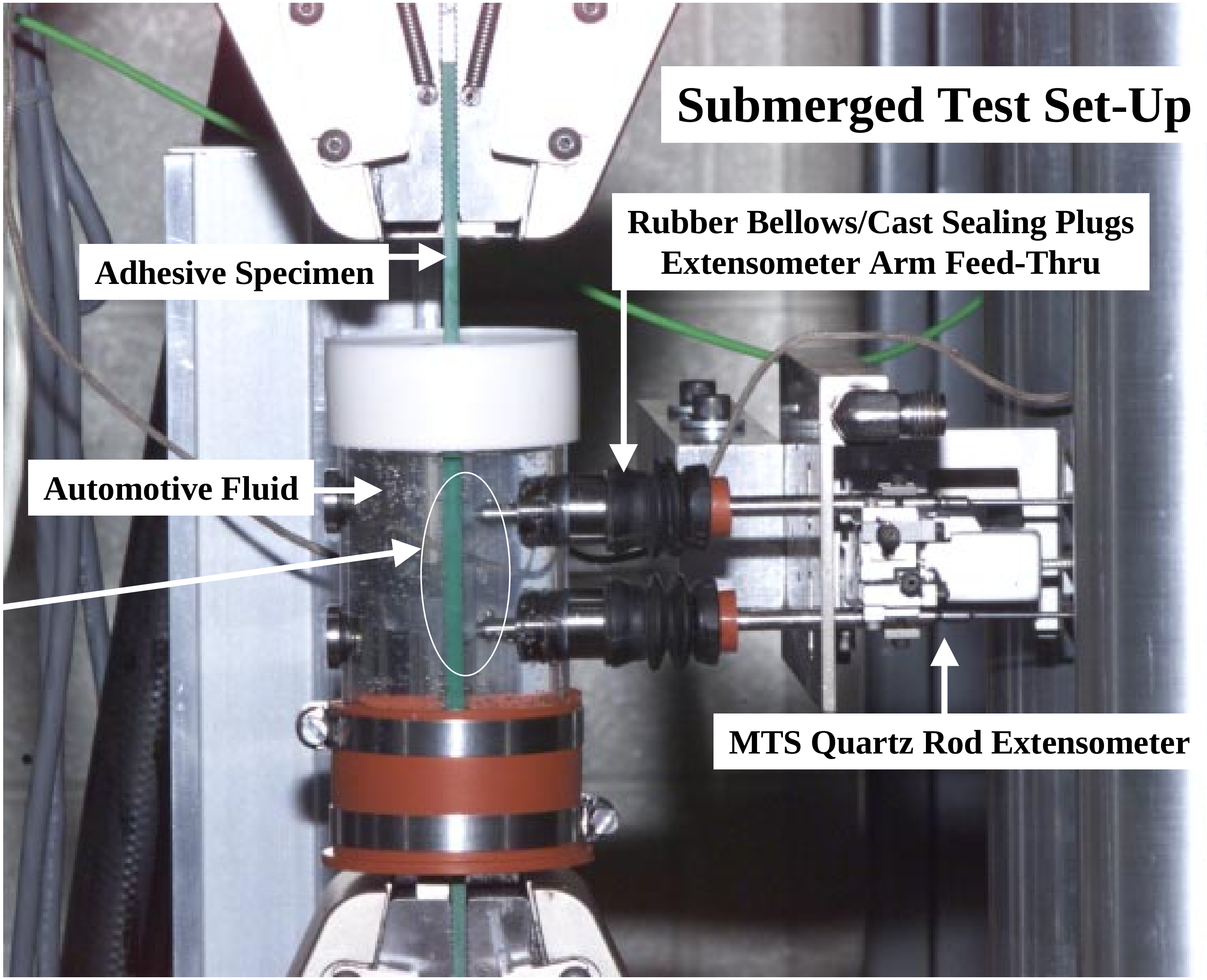
Characterization of a Structural Adhesive in Automotive Environments

Characterization of a Structural Adhesive in Automotive Environments

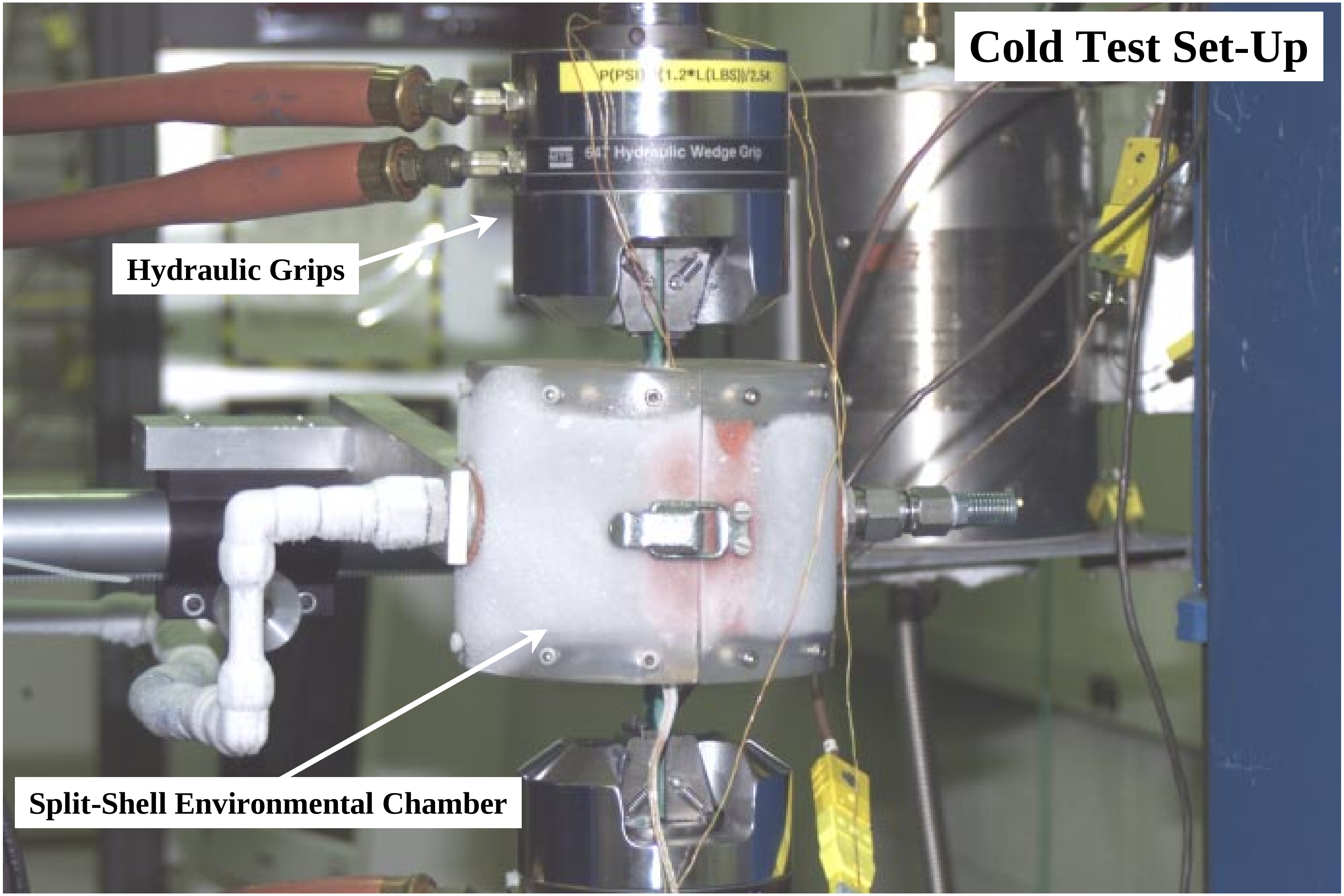
Typical test set-up showing servo-hydraulic test machines, controllers and computers with data acquisition/control cards.

Characterization of a Structural Adhesive in Automotive Environments

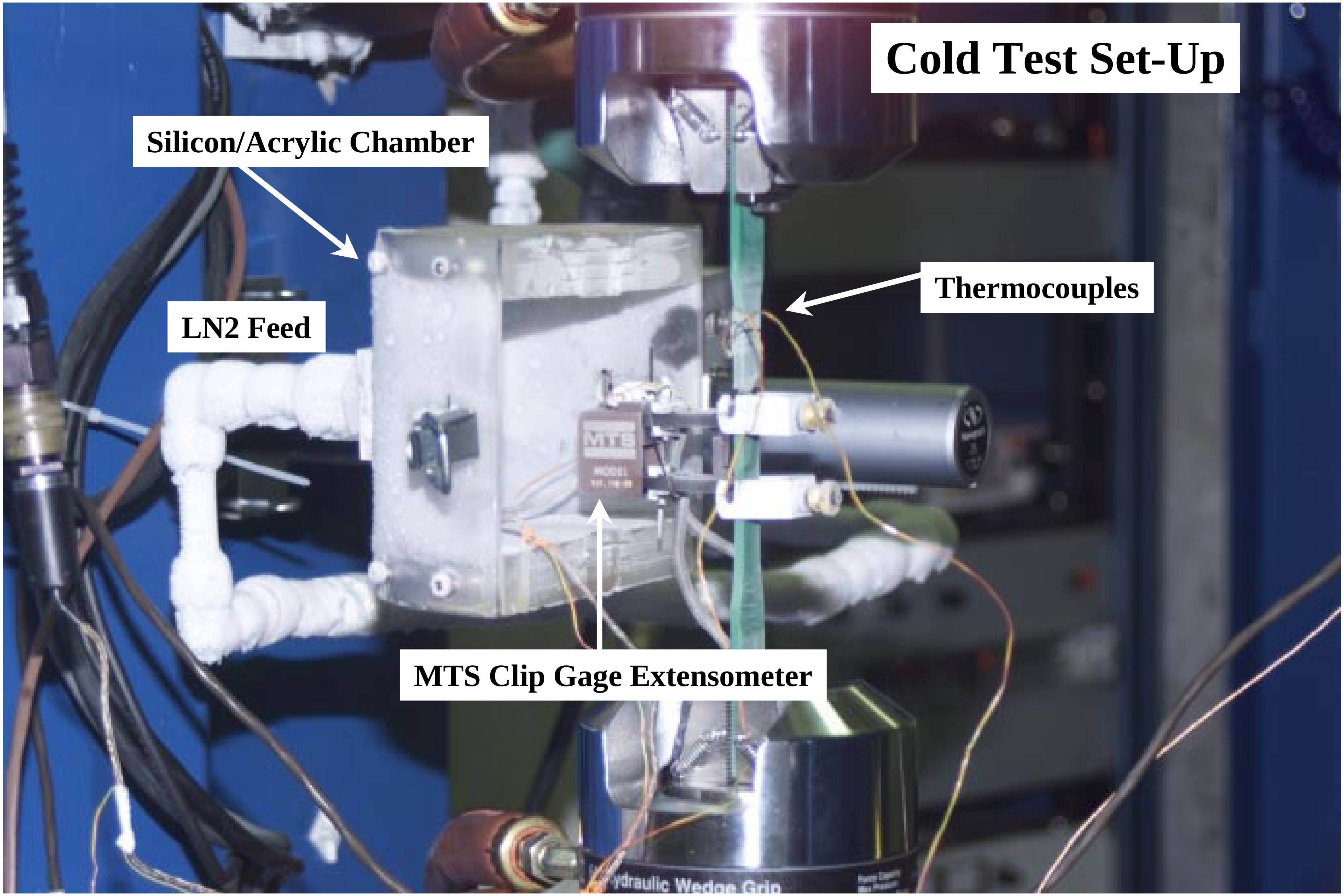




Characterization of a Structural Adhesive in Automotive Environments



Characterization of a Structural Adhesive in Automotive Environments



Characterization of a Structural Adhesive in Automotive Environments

Data Acquisition and Test Control Overview

-National Instruments PCI-MIO-6301 A/D - D/A board

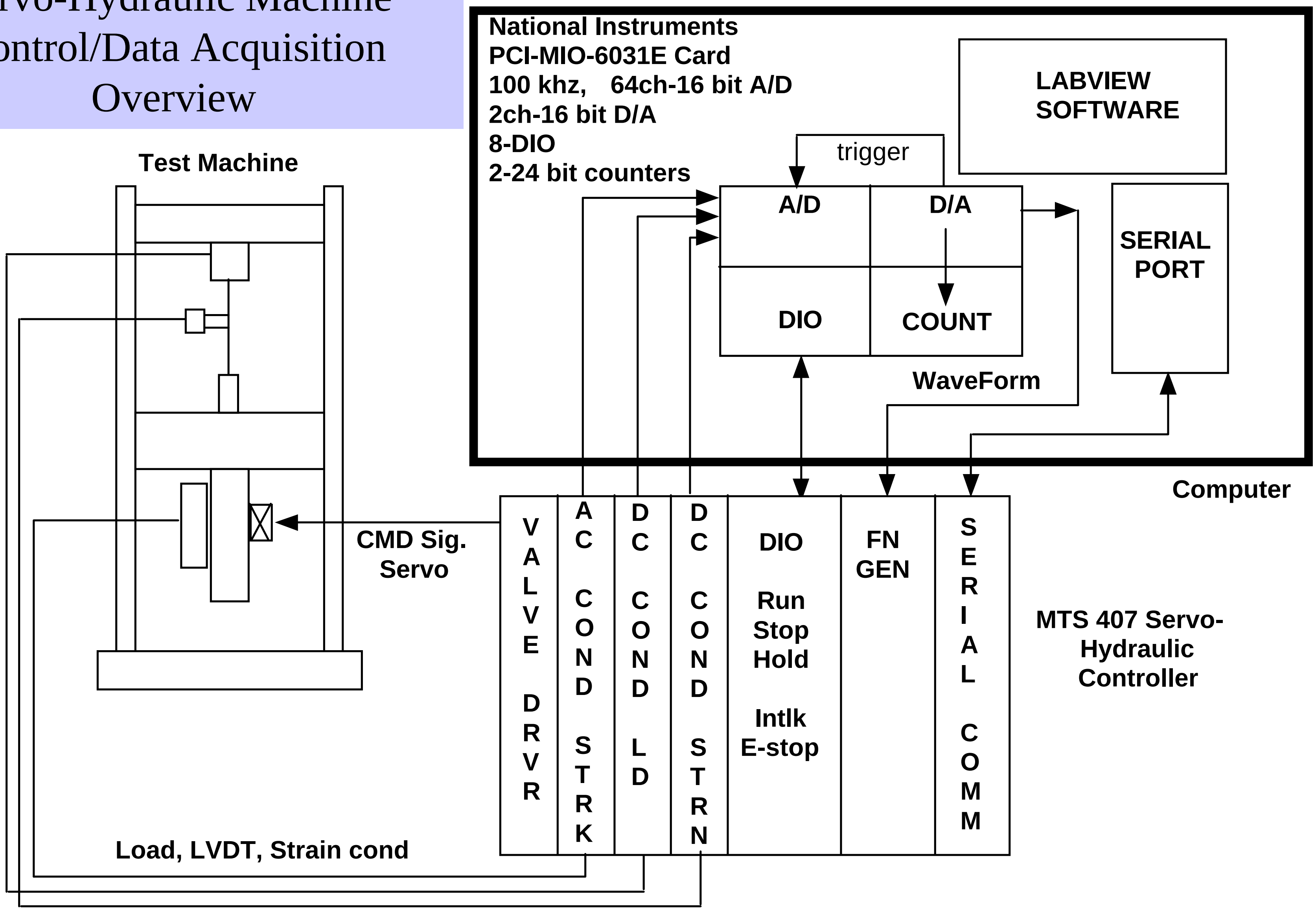
-100 kHz sampling rate
- 64 channels A/D, 16 bit resolution (signal sampling)
- 2 channels D/A, 16 bit resolution (waveform generation)
- 8 digital TTL lines (interface servo-hydraulic interlocks, etc.)
- 2-24bit counters (on-board cycle counter and elapsed time counter)

-All Test Software Written In-House Using LabView (National Instruments)

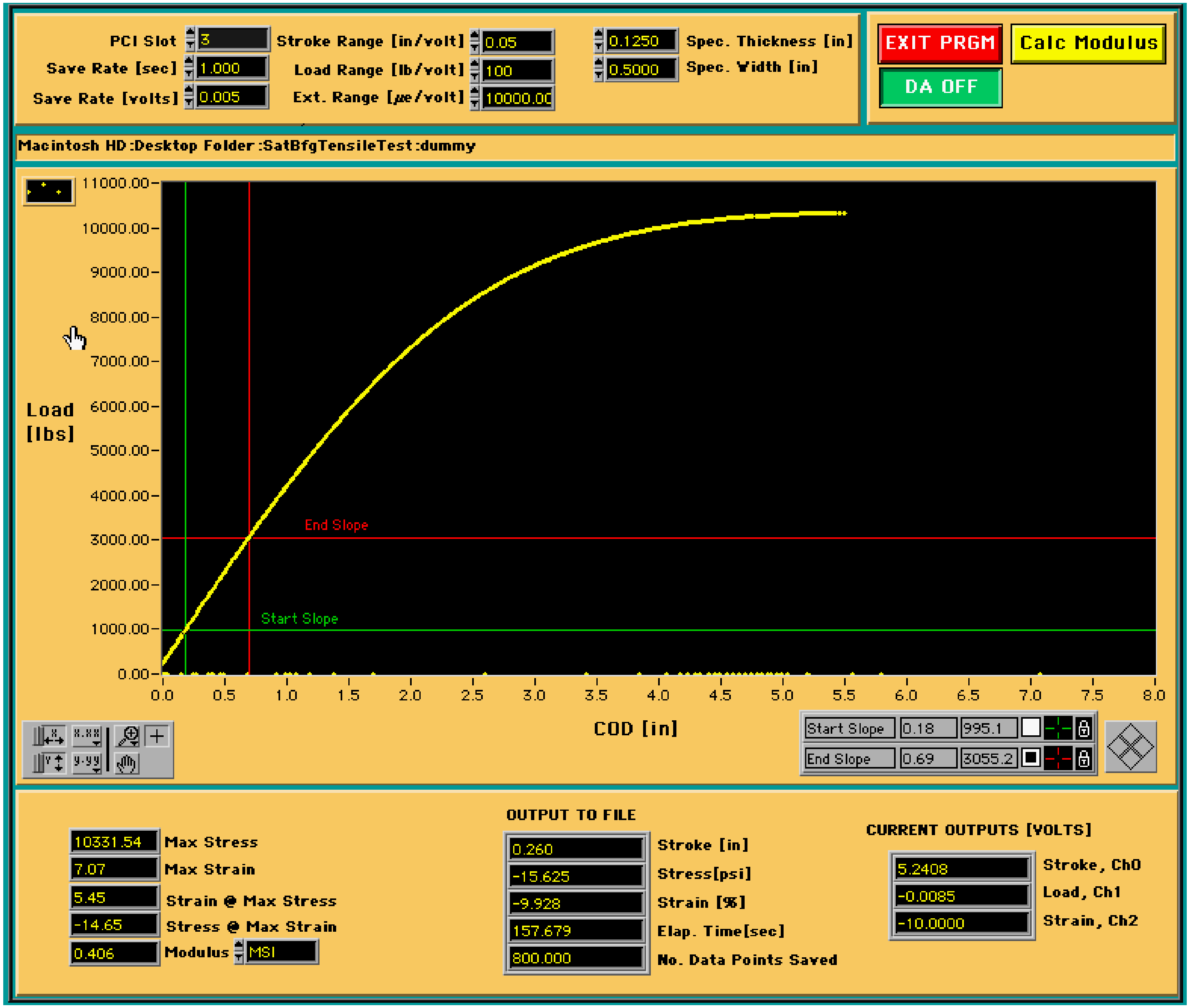
- Stress/Strain-Load/Displacement
- Automated fatigue with custom waveform generation
- Automated creep utilizing serial communication with MTS 407 servo-hydraulic controller.
- Fracture toughness with automated re-loading each crack extension.

Characterization of a Structural Adhesive in Automotive Environments

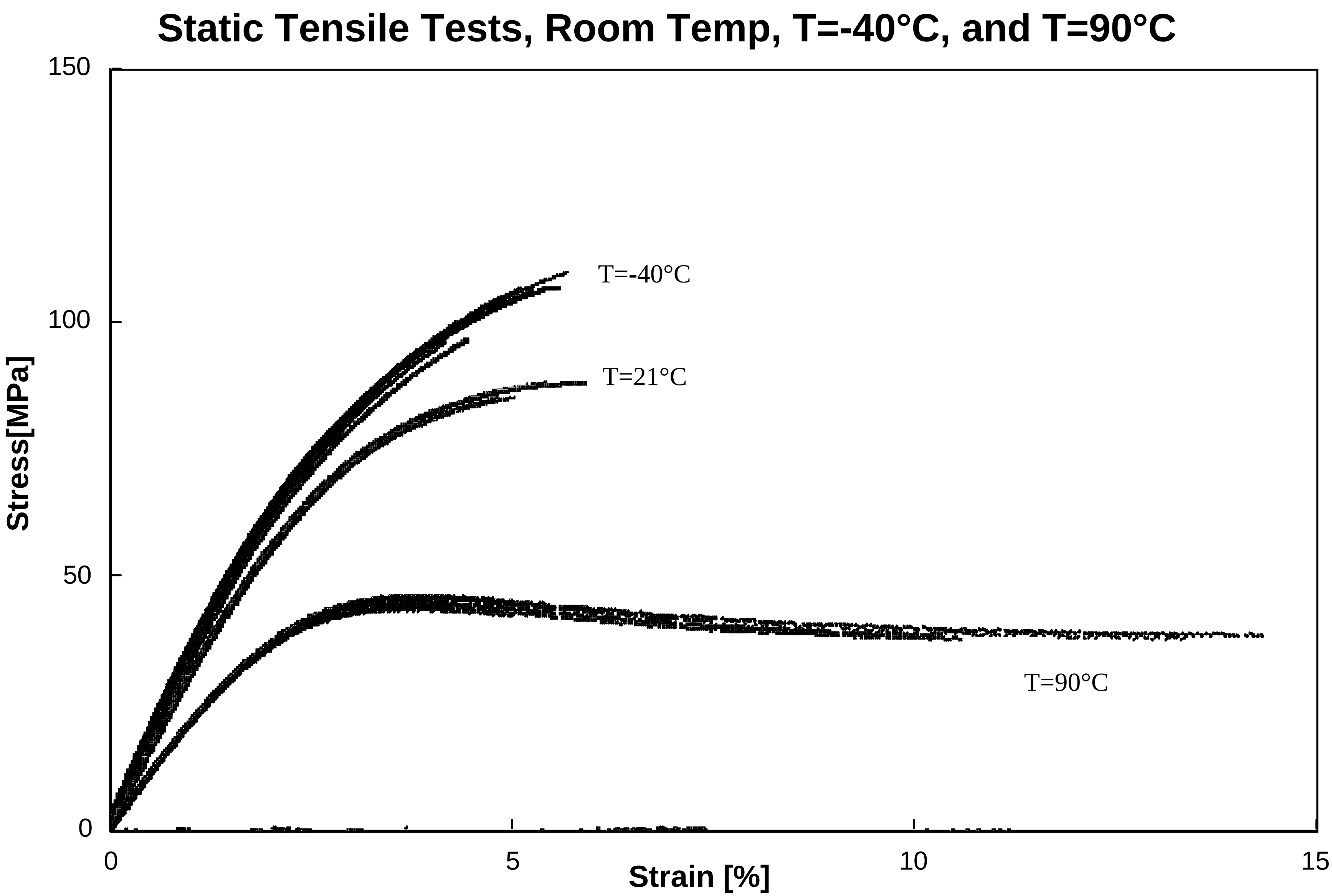
Servo-Hydraulic Machine Control/Data Acquisition Overview



Characterization of a Structural Adhesive in Automotive Environments

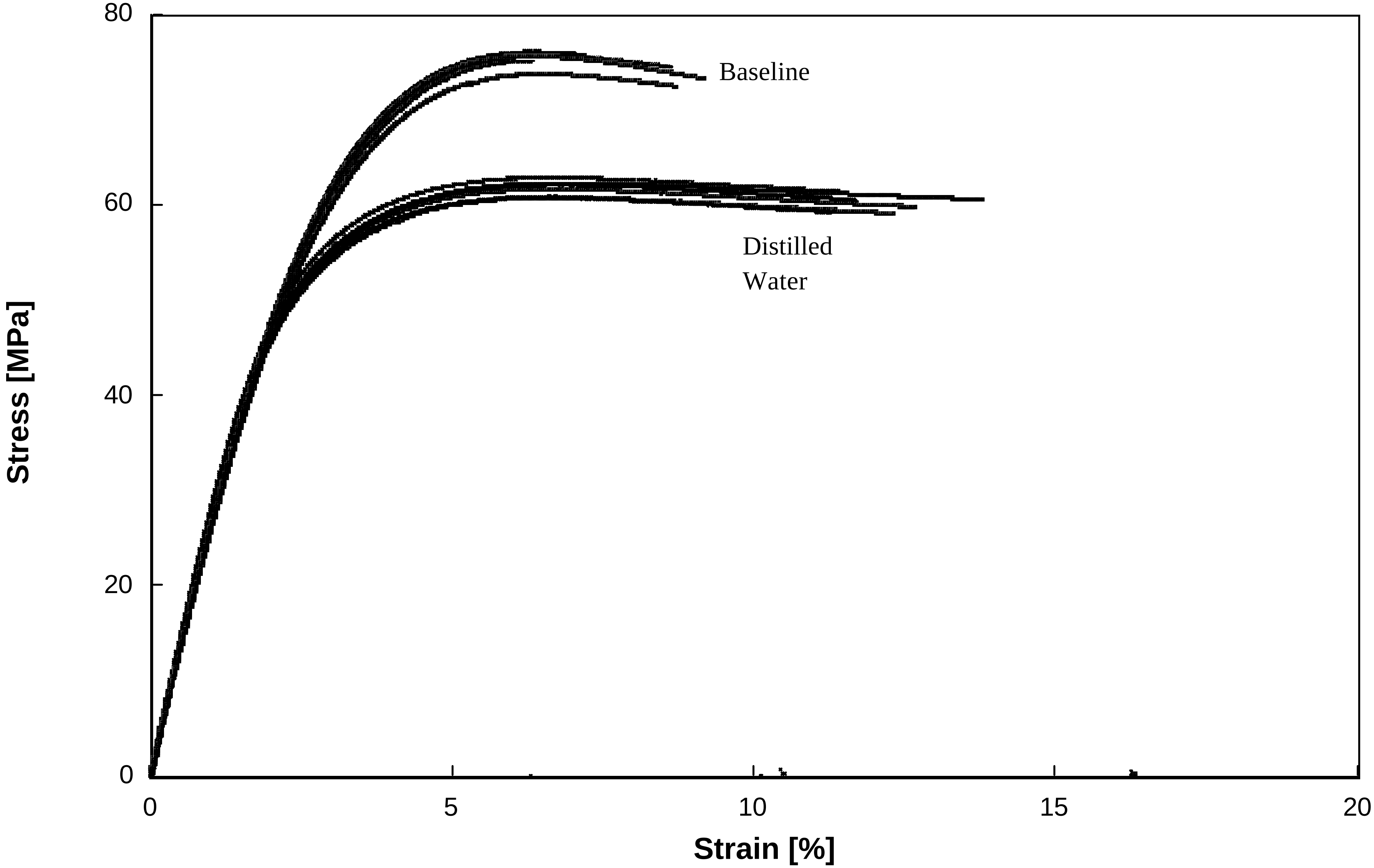


Characterization of a Structural Adhesive in Automotive Environments



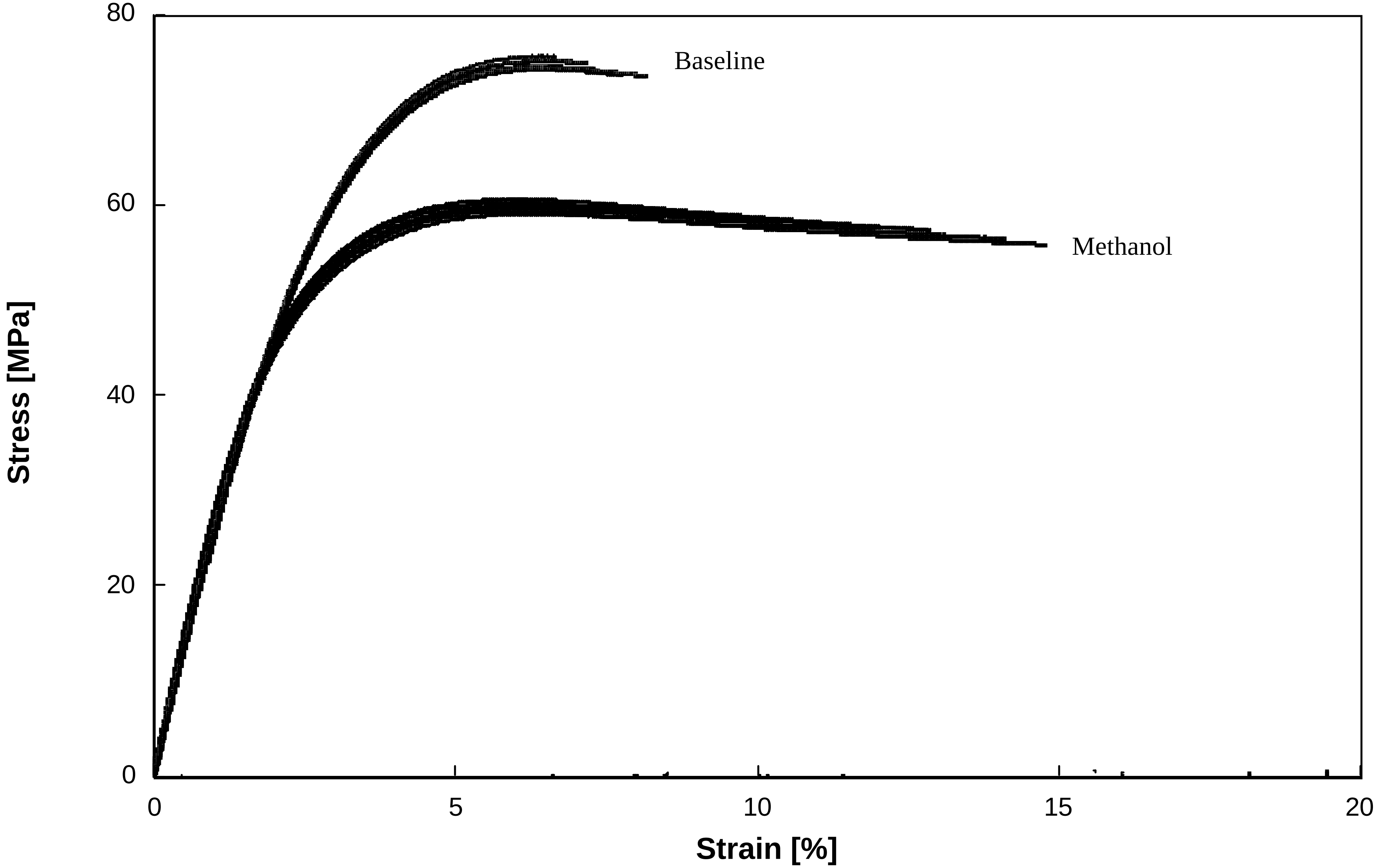
Characterization of a Structural Adhesive in Automotive Environments

Tensile Tests, Baseline and 1000 Hours Exposure To Distilled Water

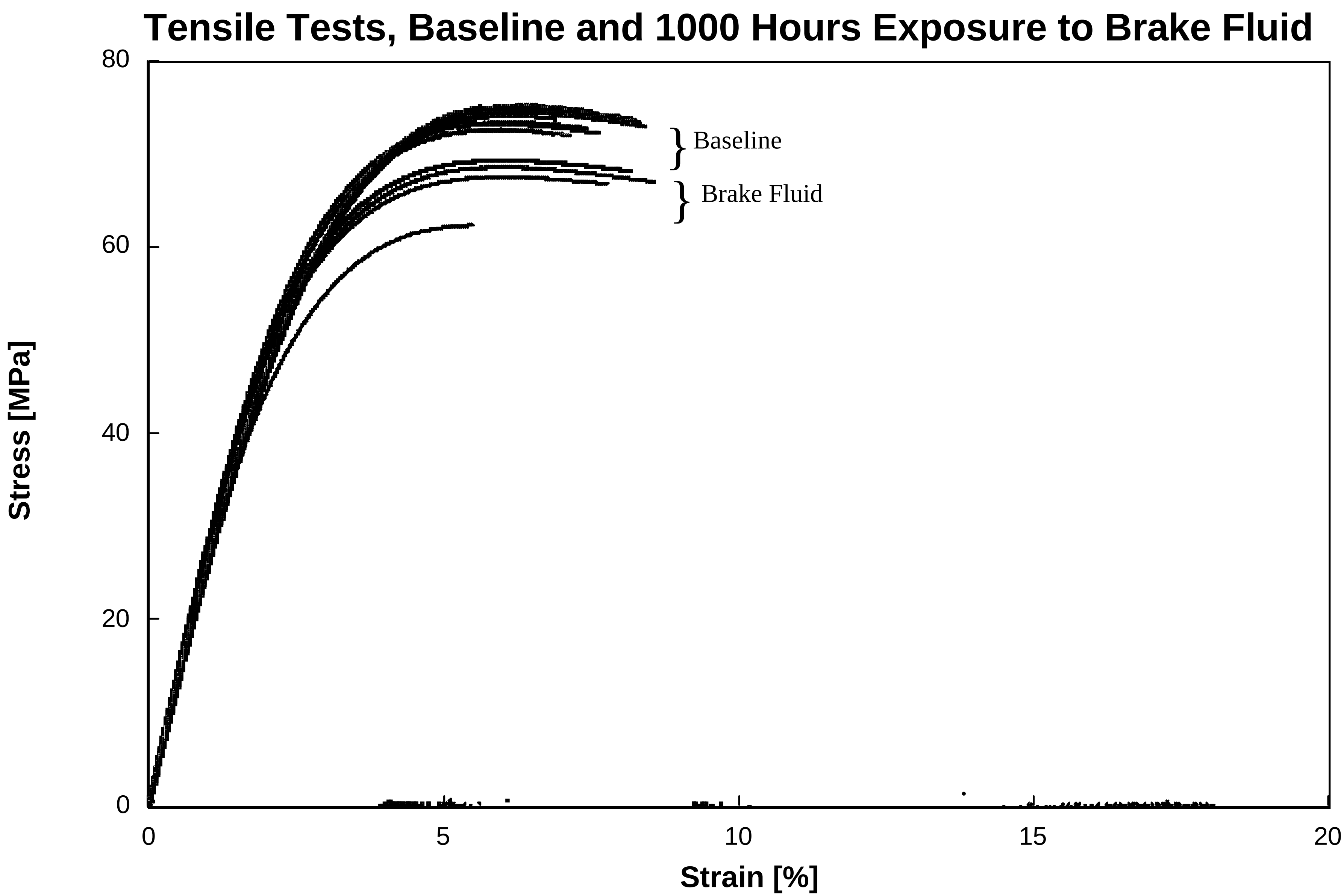


Characterization of a Structural Adhesive in Automotive Environments

Tensile Tests, Baseline and 1000 Hours Exposure to Methanol

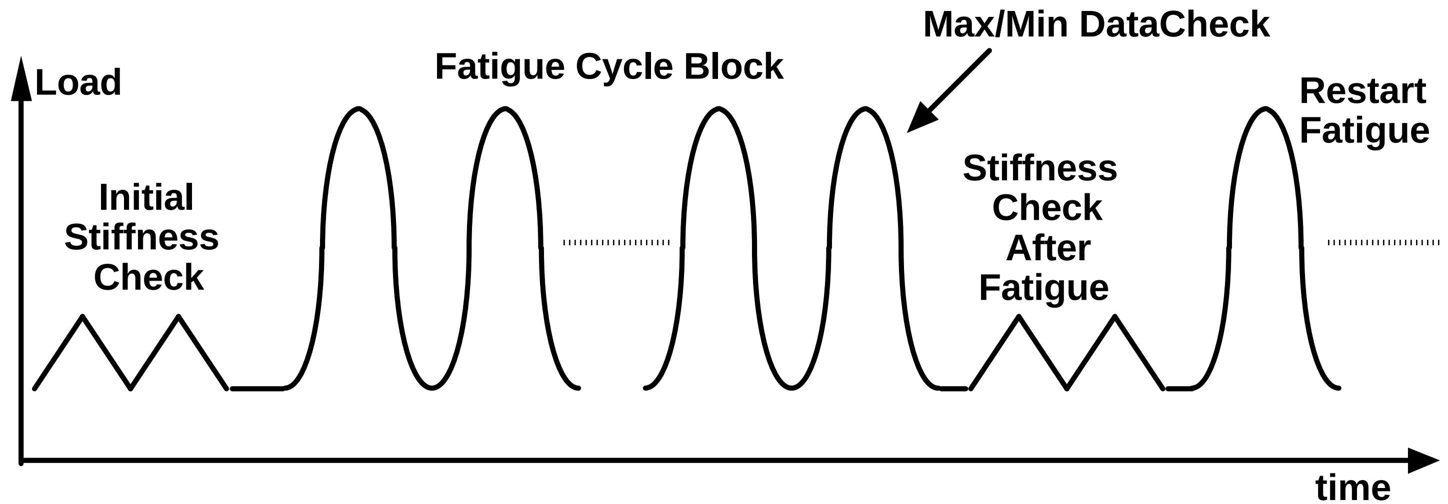


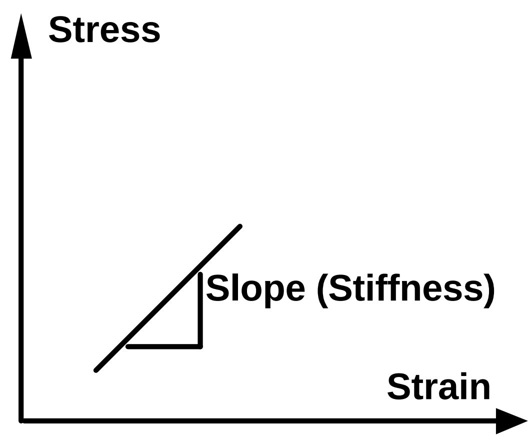
Characterization of a Structural Adhesive in Automotive Environments

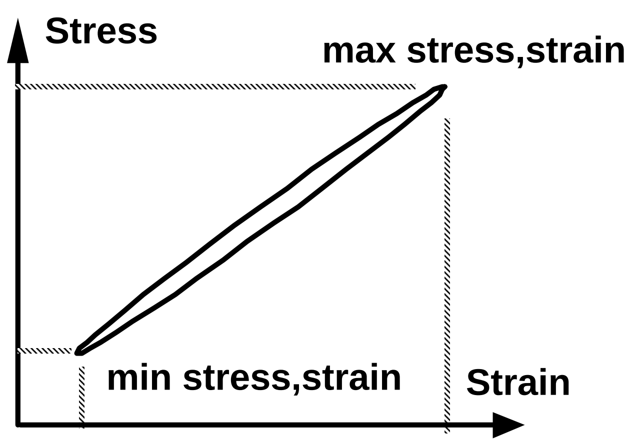


Characterization of a Structural Adhesive in Automotive Environments

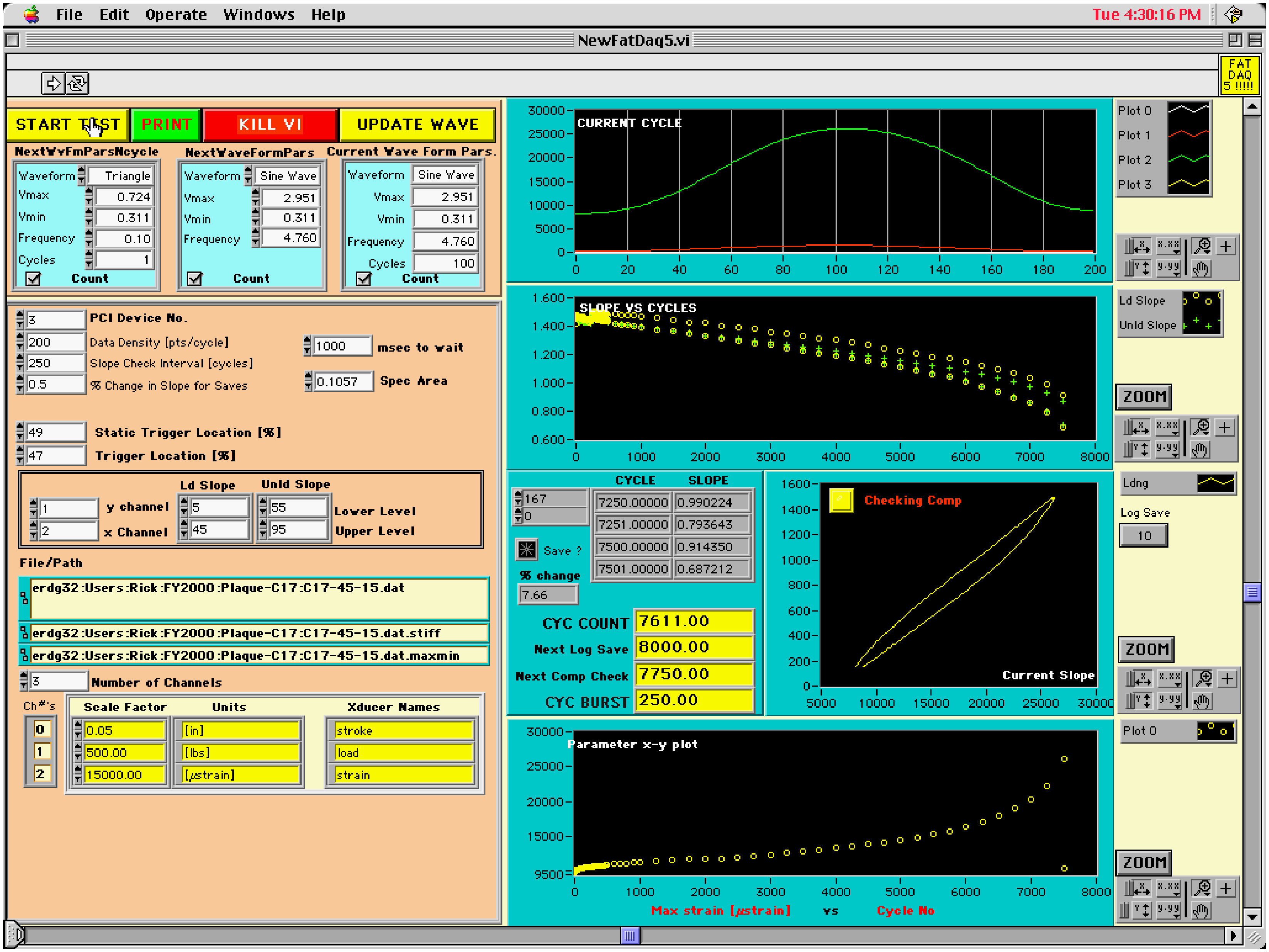
Typical Fatigue Test Sequence





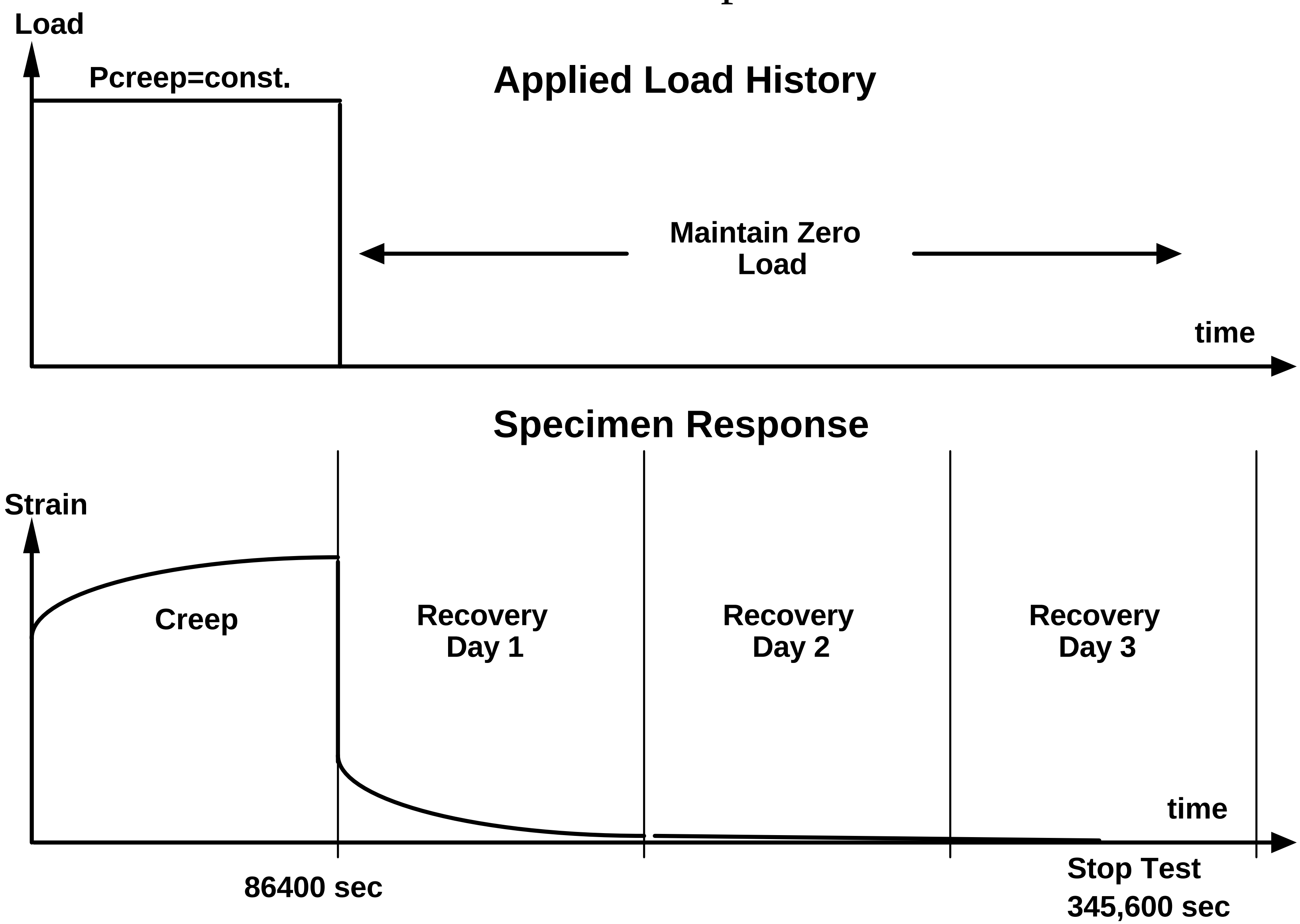


Characterization of a Structural Adhesive in Automotive Environments

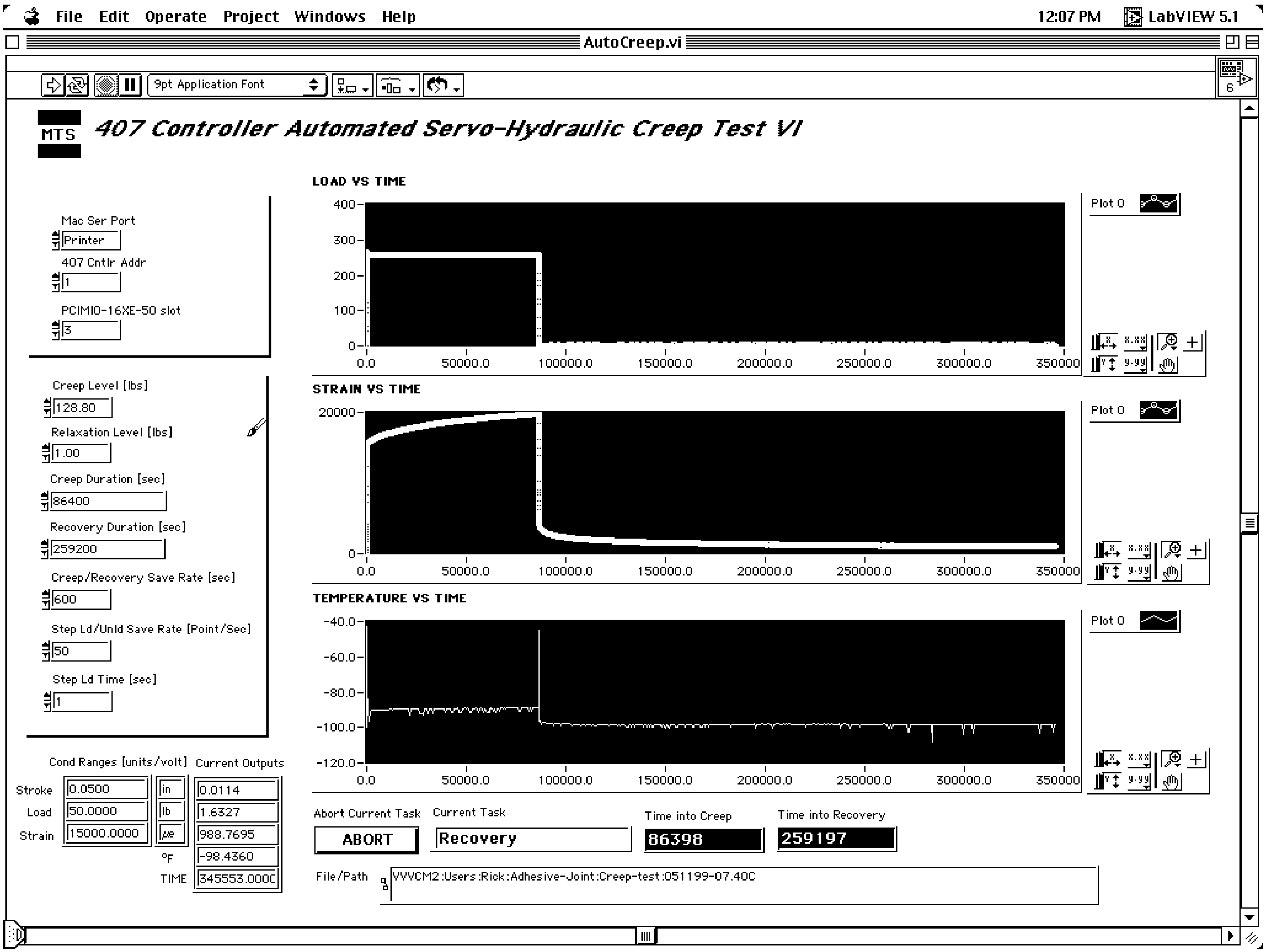


Characterization of a Structural Adhesive in Automotive Environments

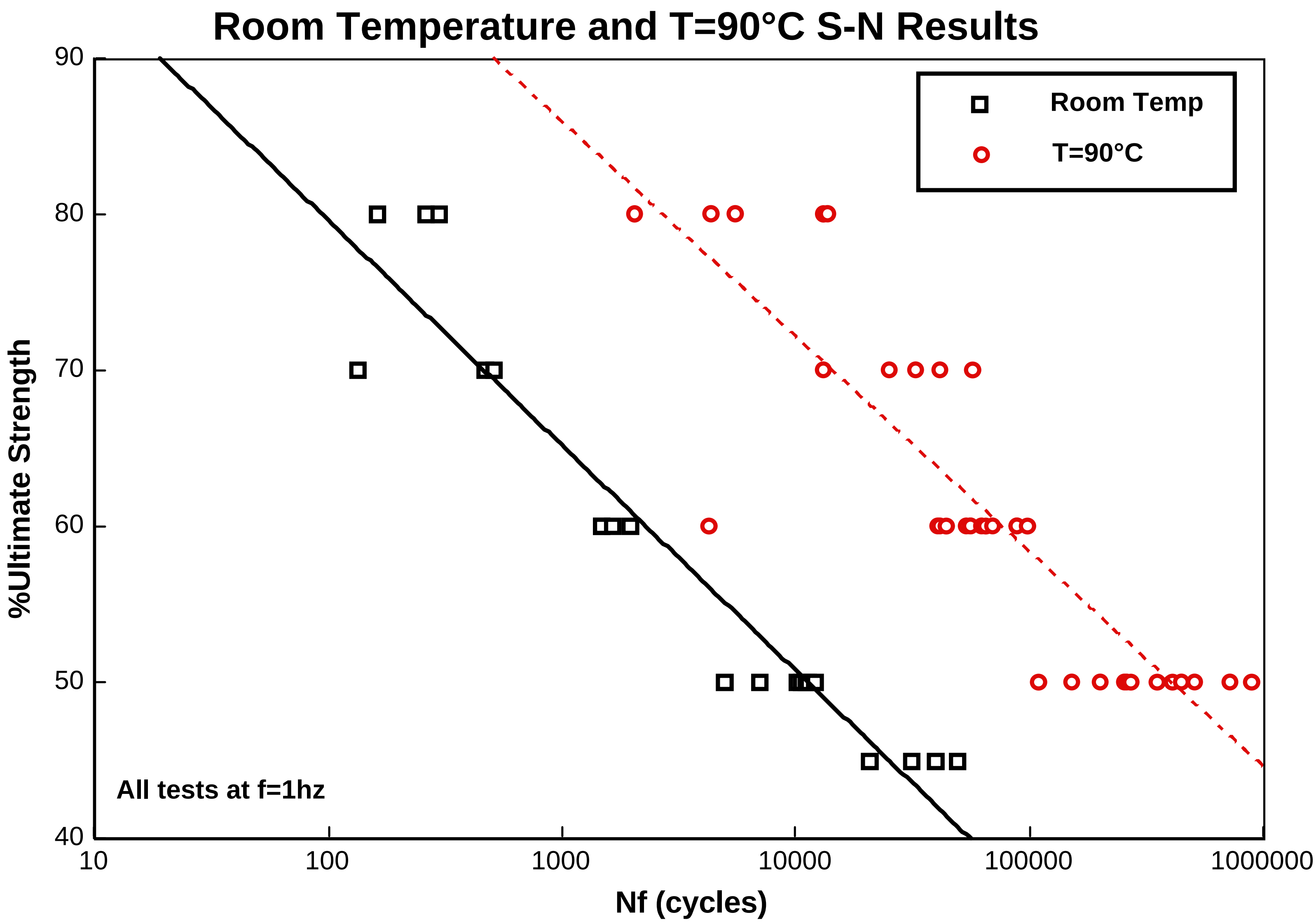
Automated Creep Test Procedure



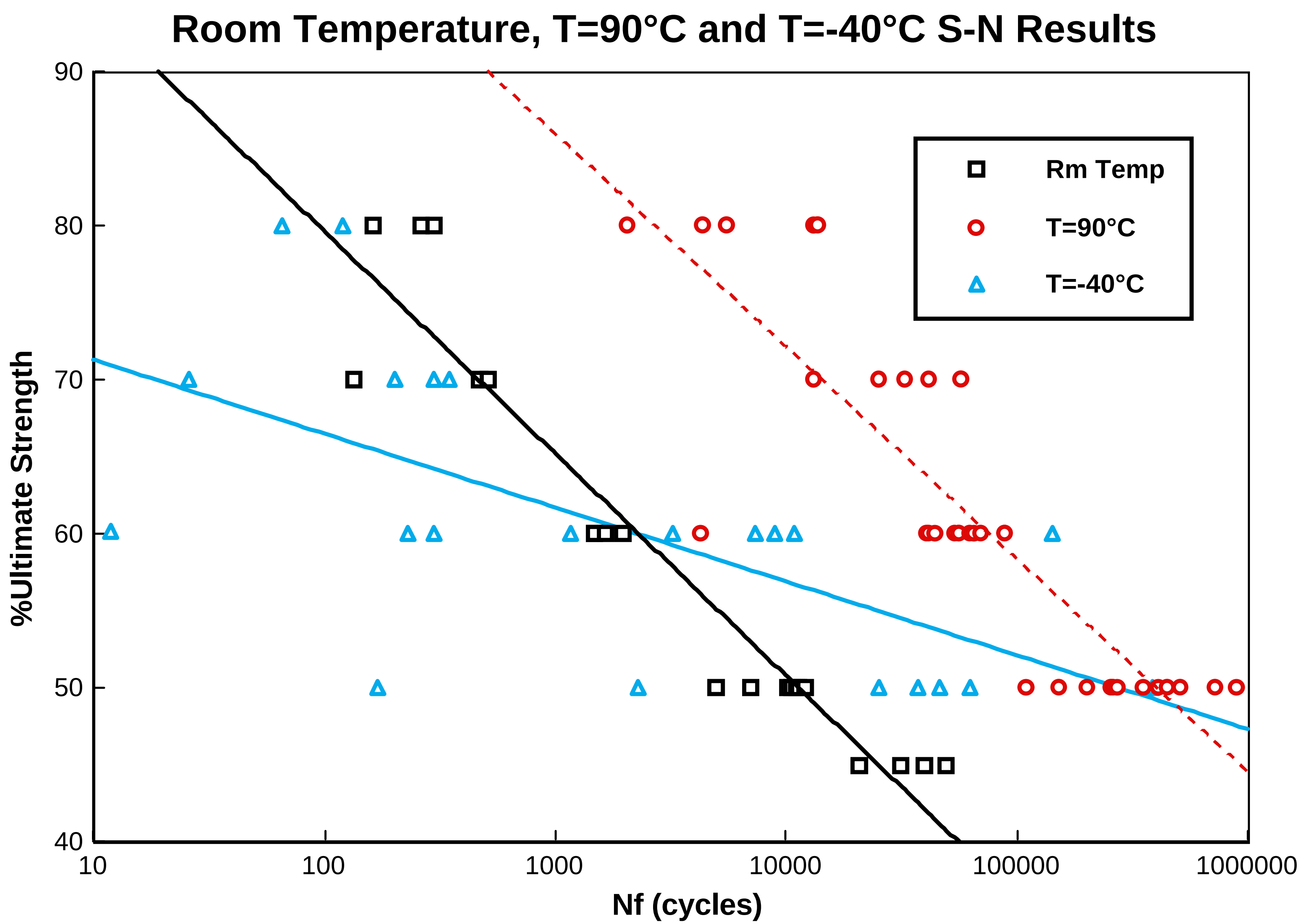
Characterization of a Structural Adhesive in Automotive Environments



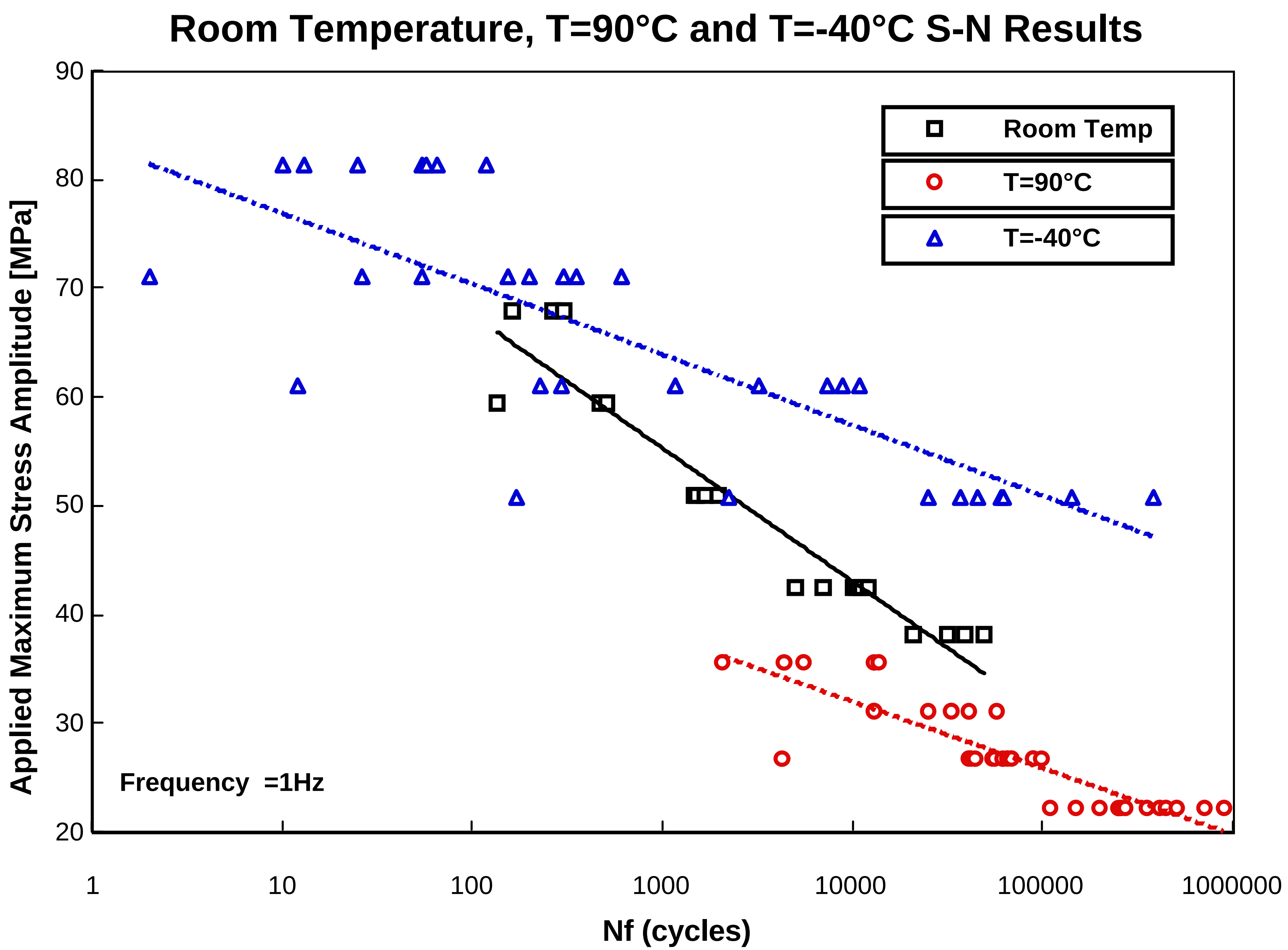
Characterization of a Structural Adhesive in Automotive Environments



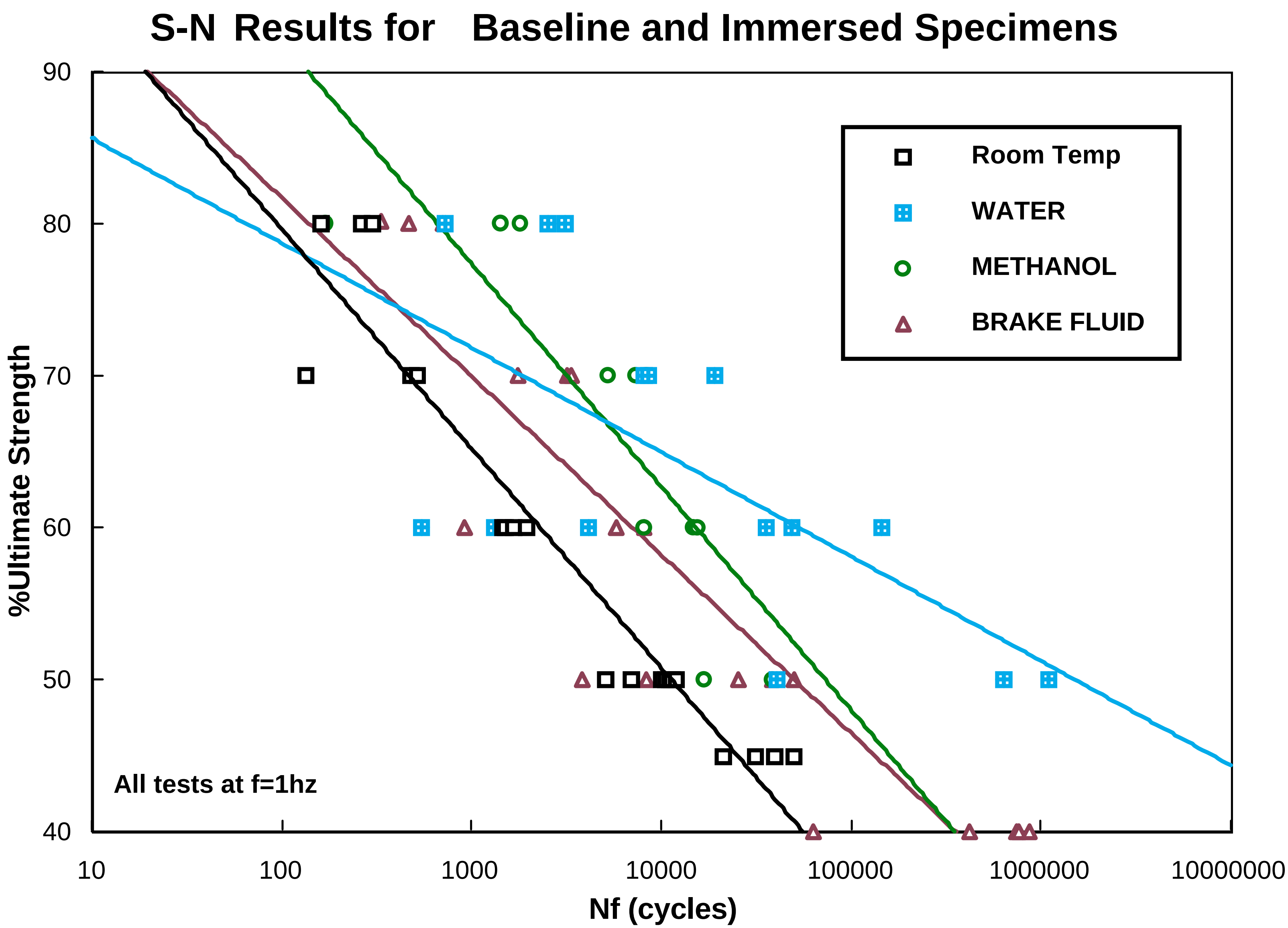
Characterization of a Structural Adhesive in Automotive Environments



Characterization of a Structural Adhesive in Automotive Environments

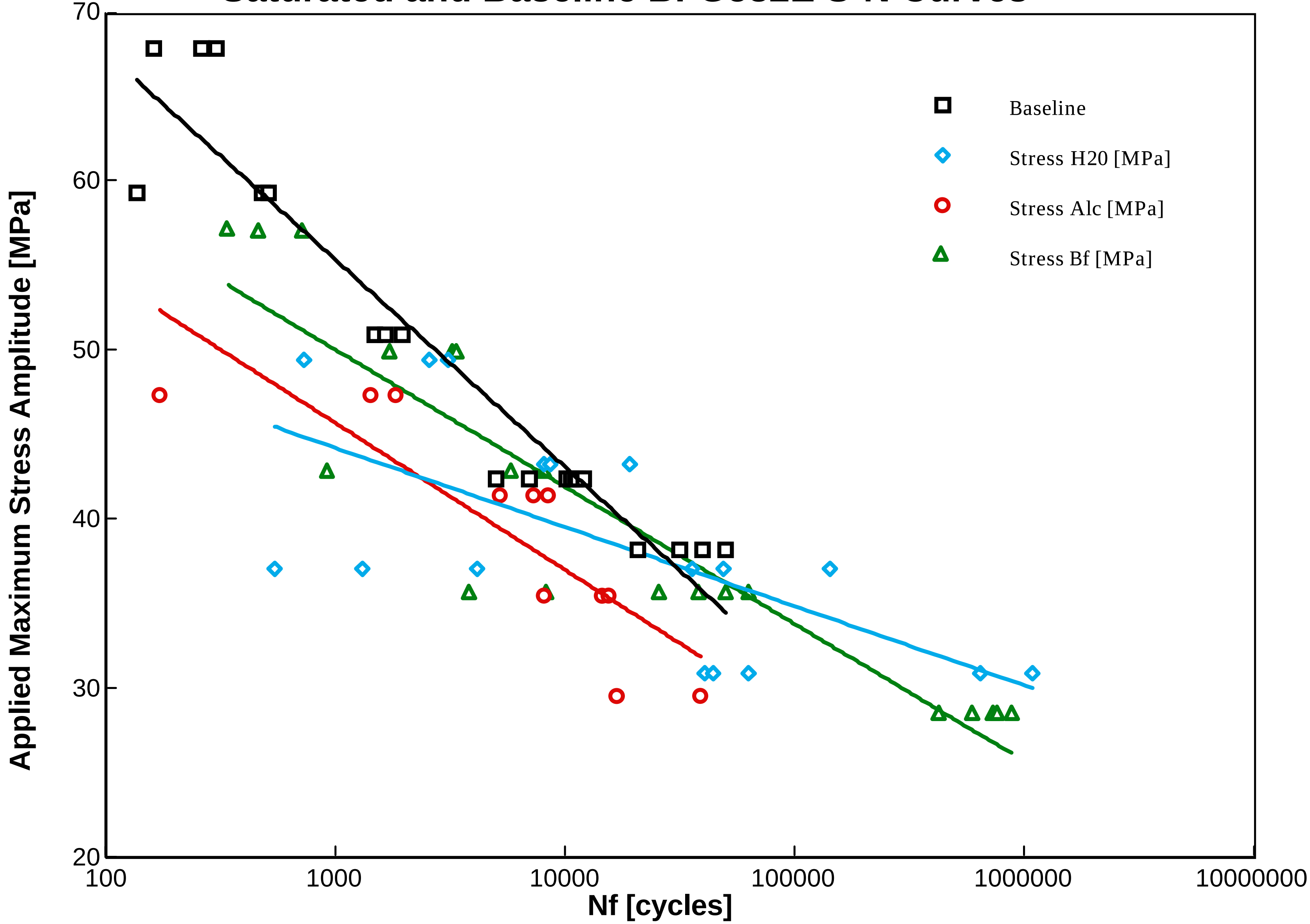


Characterization of a Structural Adhesive in Automotive Environments



Characterization of a Structural Adhesive in Automotive Environments

Saturated and Baseline BFG582E S-N Curves



Characterization of a Structural Adhesive in Automotive Environments

Weibull Statistics / S-N Data

$$1 - P = \exp\left\{ -\left(\frac{N_f}{b} \right)^m \right\} \qquad [1]$$

$1 - P \equiv probability\ of\ survival$

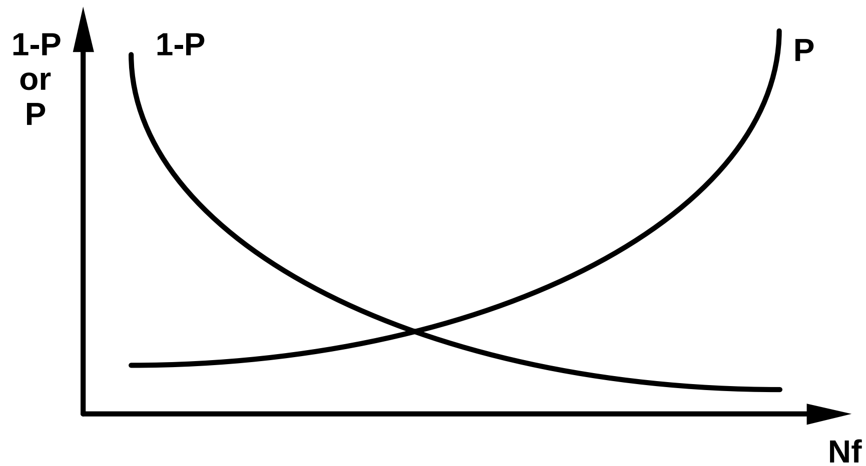
$N_f \equiv no.\ of\ cycles$

$m, b \equiv Weibull\ slope\ and\ shape\ factor$

$$1 - P = 1 - \frac{i}{1 + N} \qquad [2]$$

$N \equiv No.\ of\ data\ points$

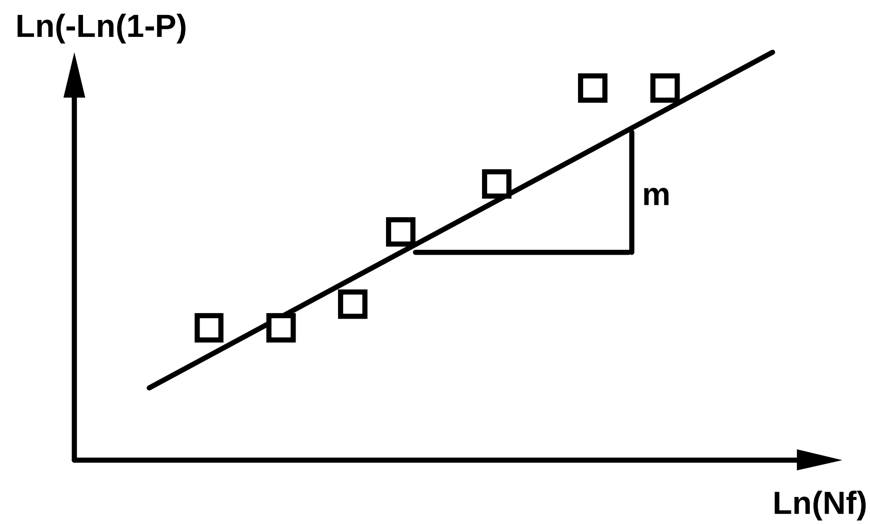
$(1 \le i \le N)$



Take natural logs of [1]:

$$Ln(-Ln(1 - P)) = mLn(N_f) - mLn(b) \qquad [3]$$

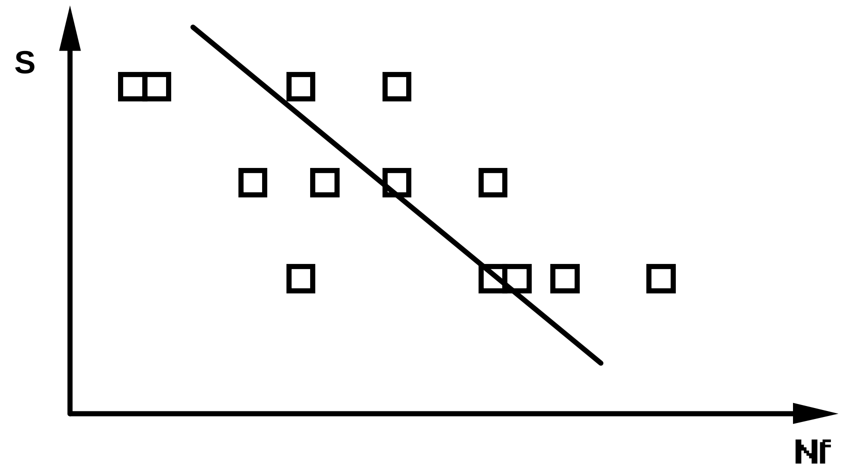
$$m = slope, \quad b = \exp\left(\frac{-y\,\mathrm{int}}{m} \right)$$



Characterization of a Structural Adhesive in Automotive Environments

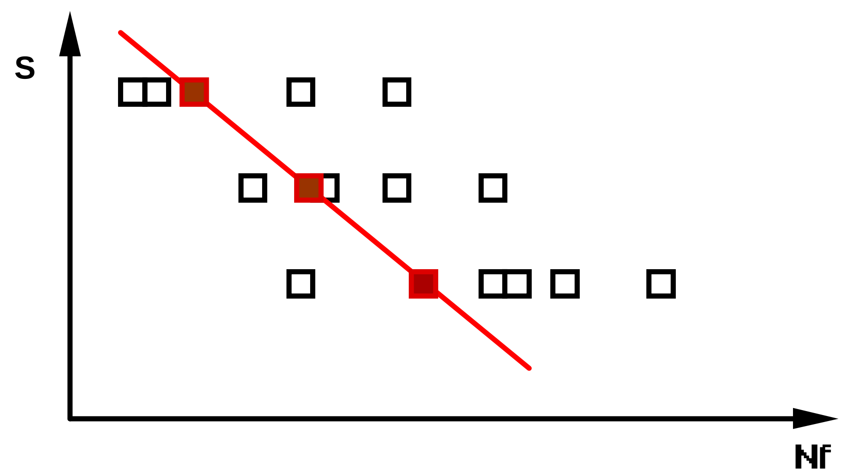
Weibull Statistics / S-N Data

For a given probability of survival or failure can solve for corresponding number of cycles at each dynamic stress level:

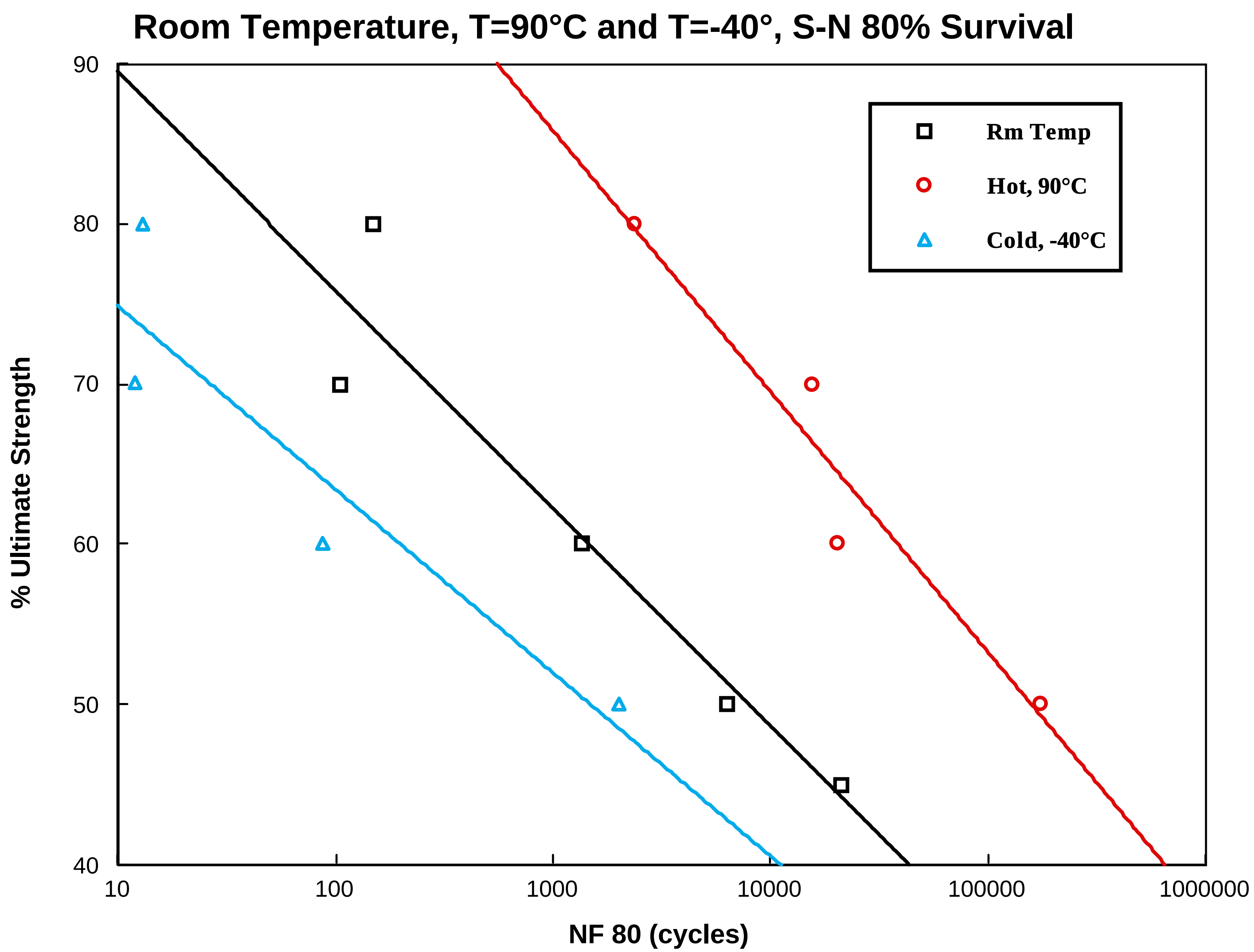


$$N_f = b\{-Ln(1-P)\}^{-1/m} \qquad [4]$$

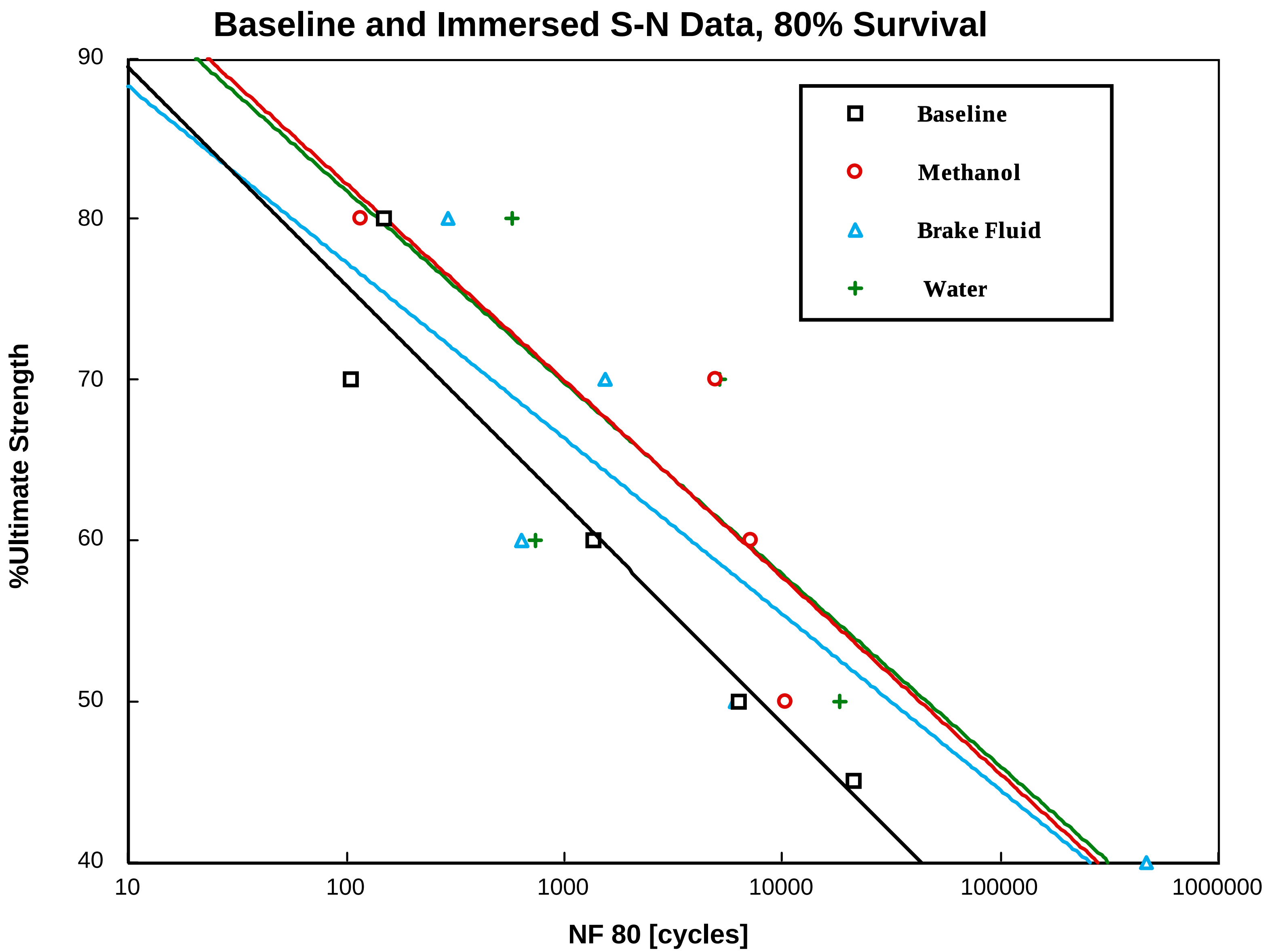
From [4], generate a new S-N curve.



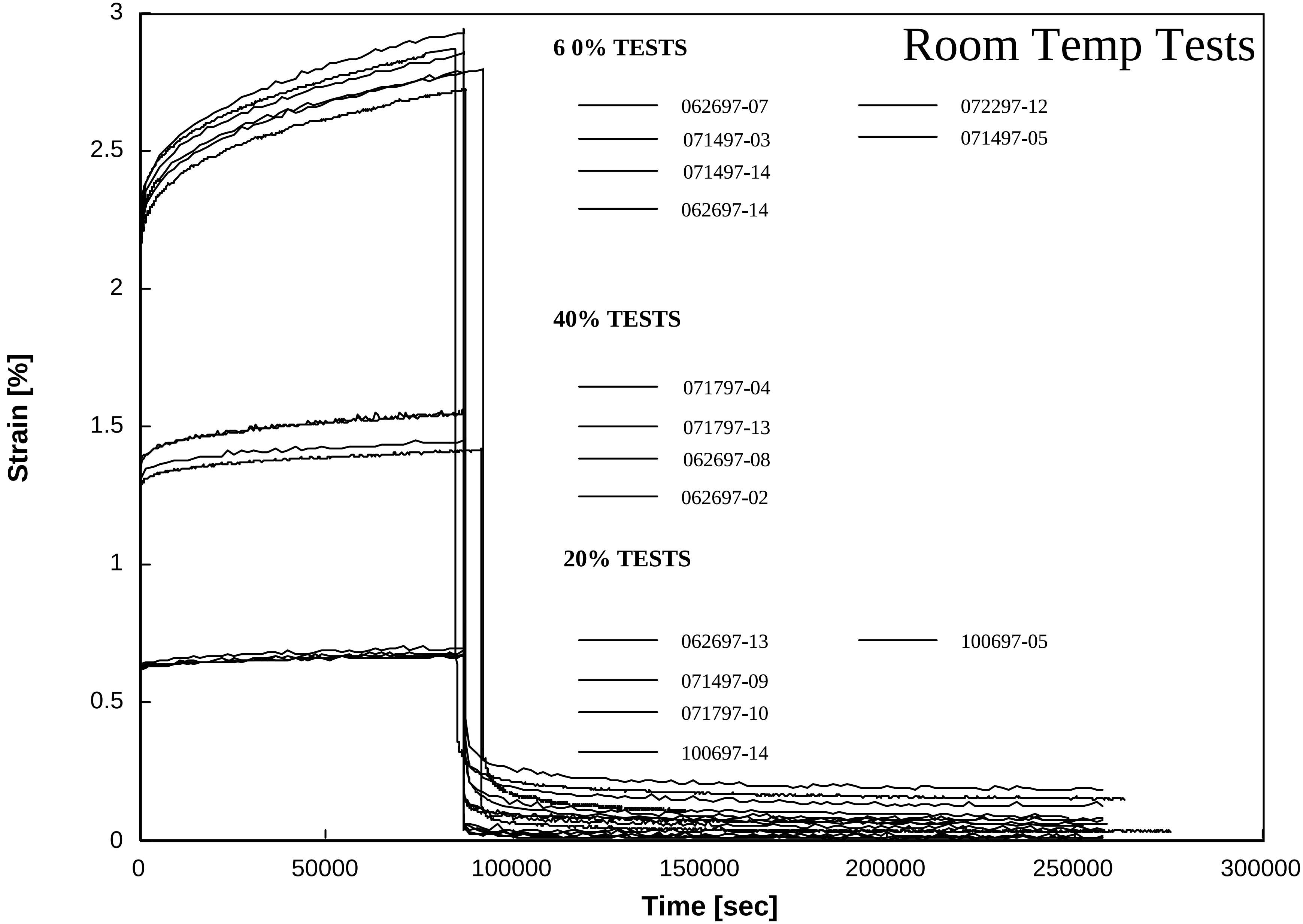
Characterization of a Structural Adhesive in Automotive Environments



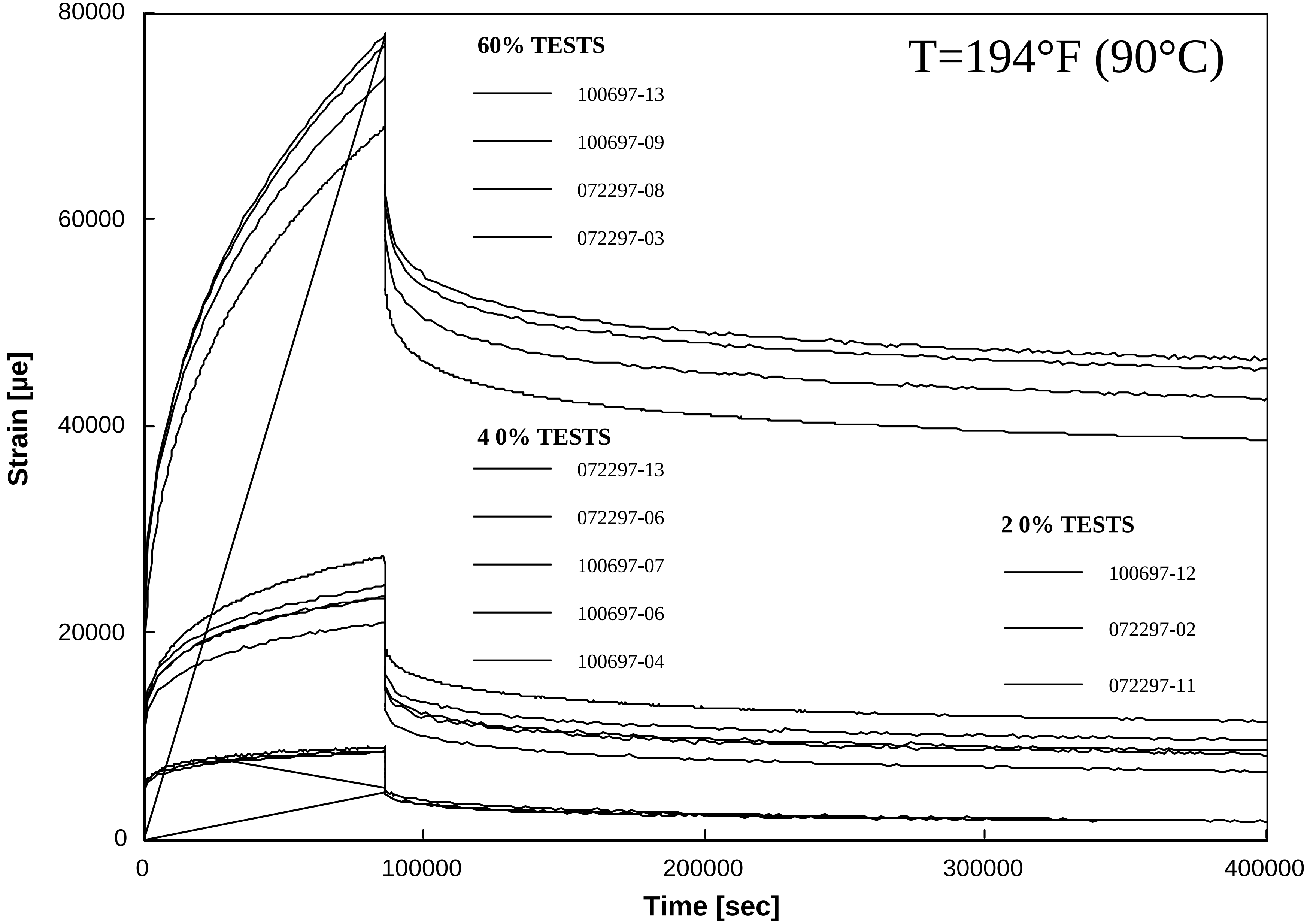
Characterization of a Structural Adhesive in Automotive Environments



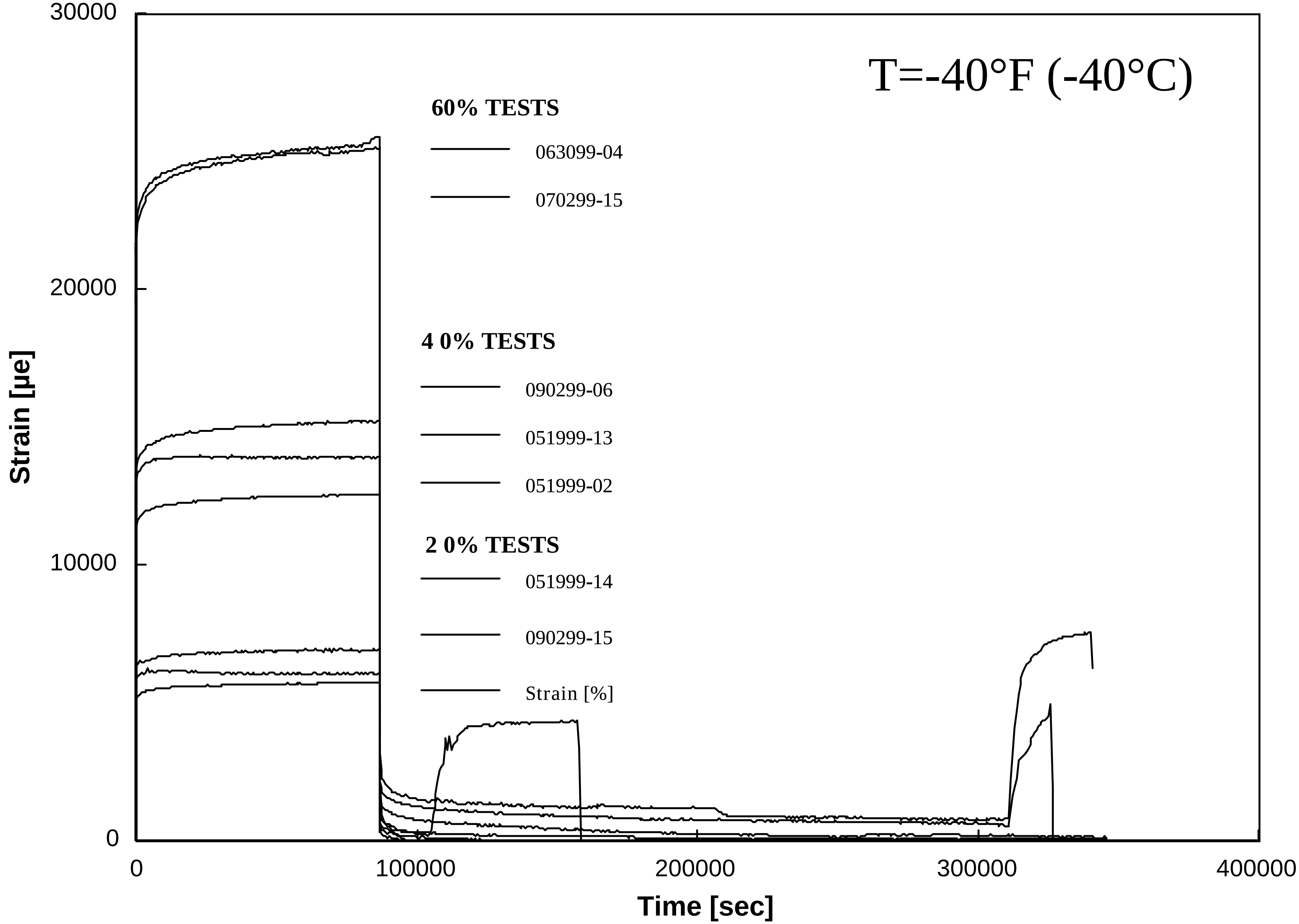
Characterization of a Structural Adhesive in Automotive Environments



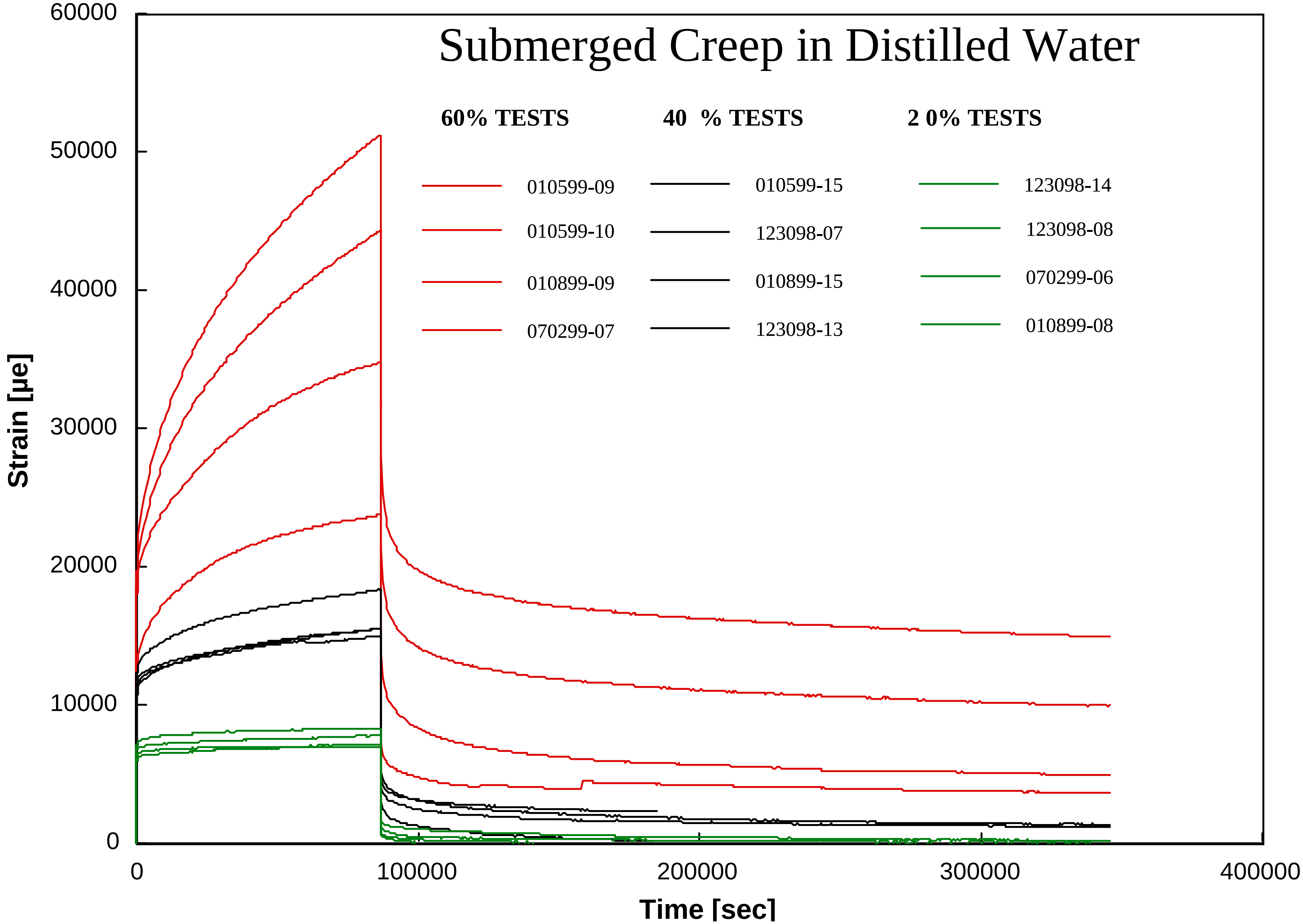
Characterization of a Structural Adhesive in Automotive Environments



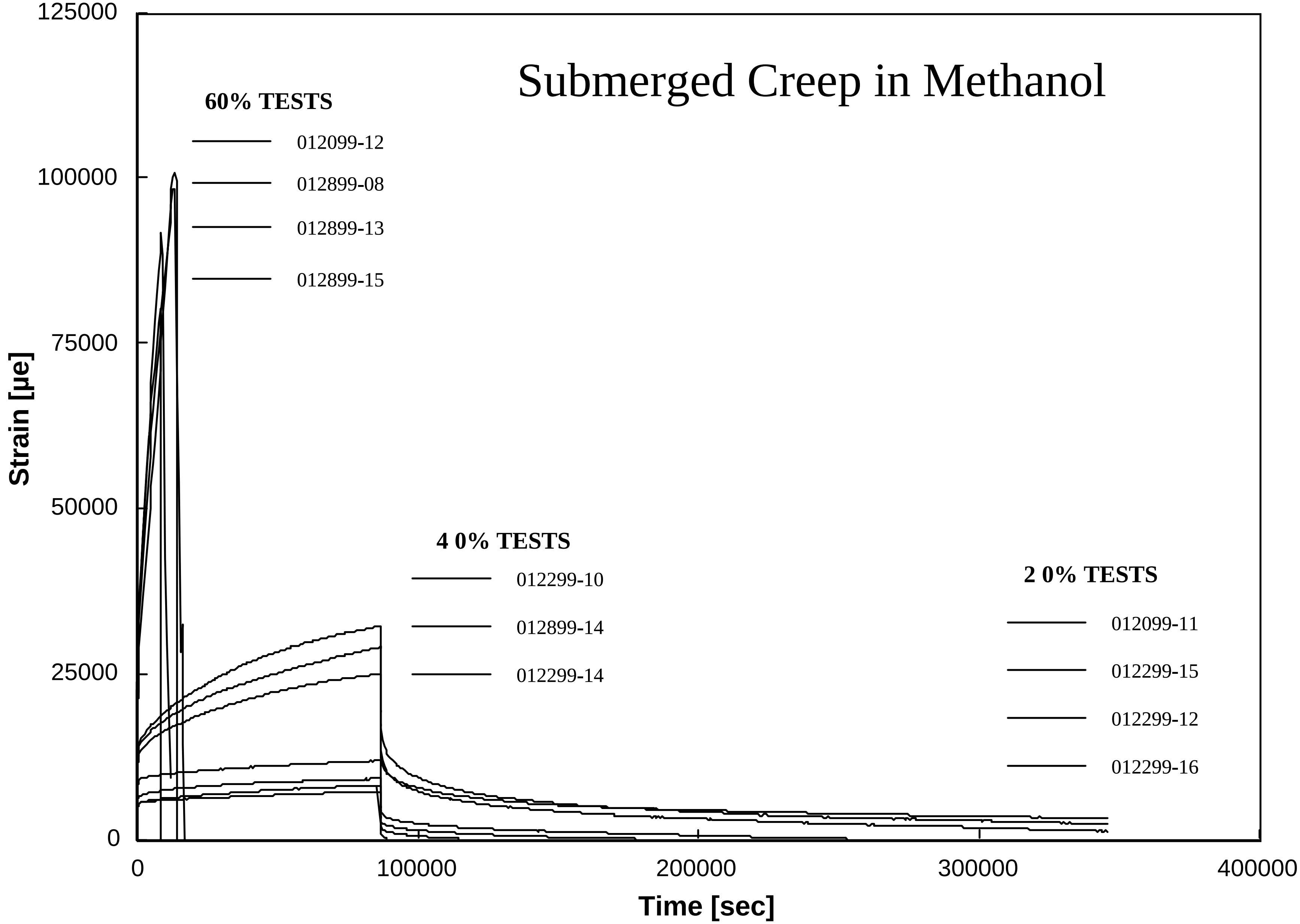
Characterization of a Structural Adhesive in Automotive Environments



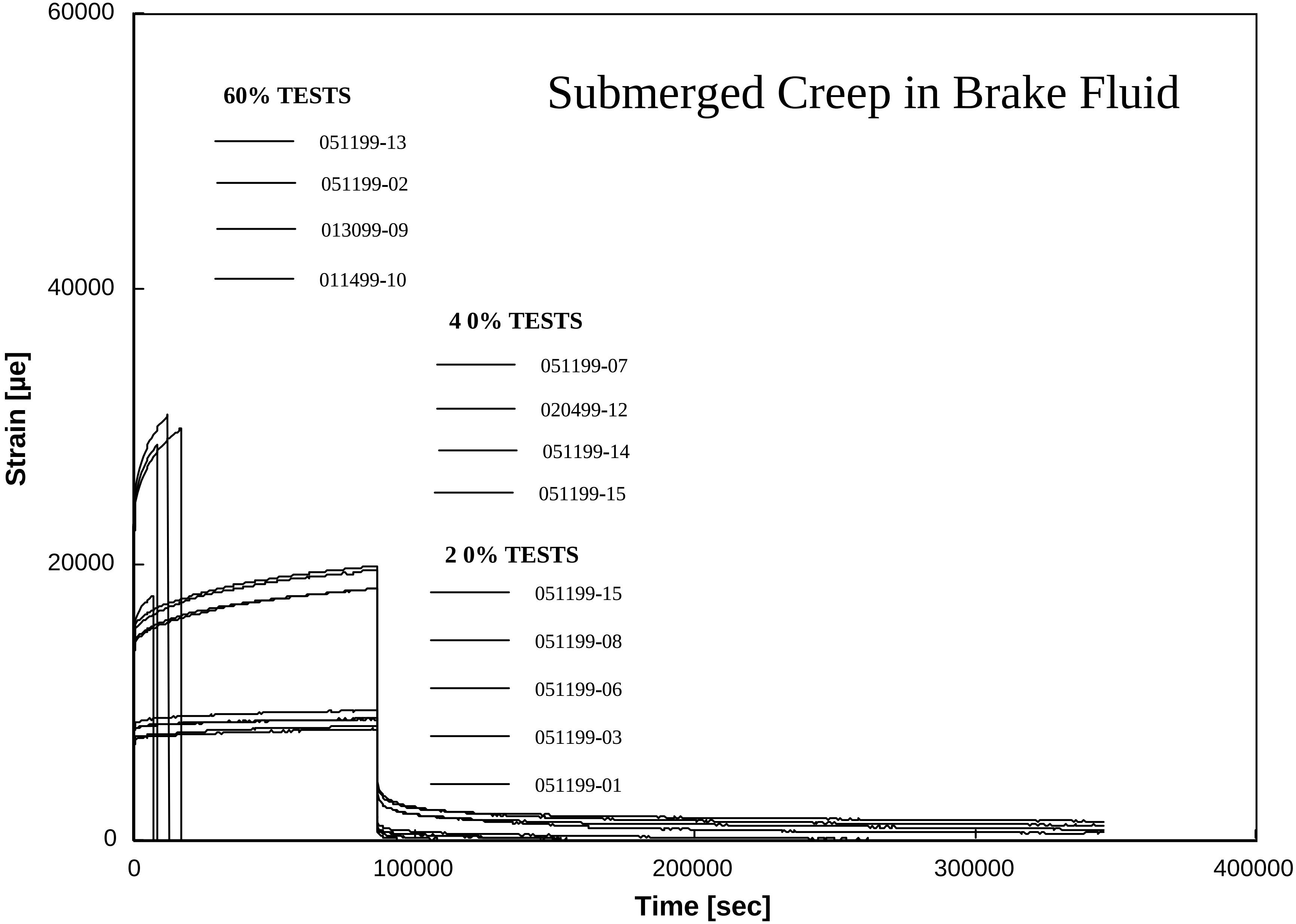
Characterization of a Structural Adhesive in Automotive Environments



Characterization of a Structural Adhesive in Automotive Environments



Characterization of a Structural Adhesive in Automotive Environments



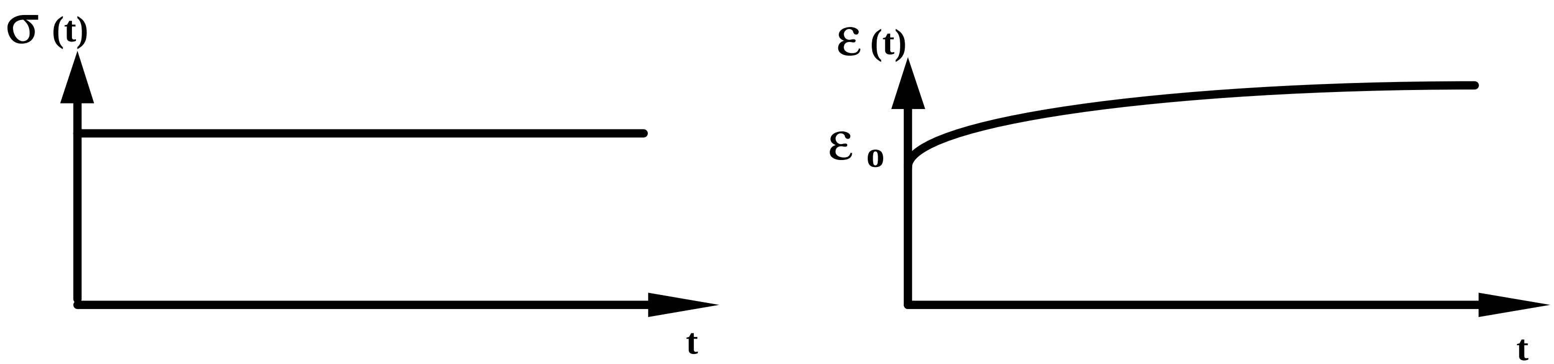
POWER LAW CREEP RESPONSE

The strain response resulting from a general stress input is given as:

$$\varepsilon(t) = \sigma(t)\left\{D_0 + D_1 t^n\right\} \quad [1]$$

In the case of constant stress input (i.e., creep loading) we have:

$$\sigma(t) = \sigma_0 H(t) \quad [2]$$



The strain is given by: $$\varepsilon(t) = \sigma_0 H(t)\left\{D_0 + D_1 t^n\right\} \quad [3]$$

Characterization of a Structural Adhesive in Automotive Environments

To determine the power law parameters separate strain into instantaneous and creep components:

$$\varepsilon(t) = \varepsilon_0 + \varepsilon_{creep}(t) \quad [3]$$

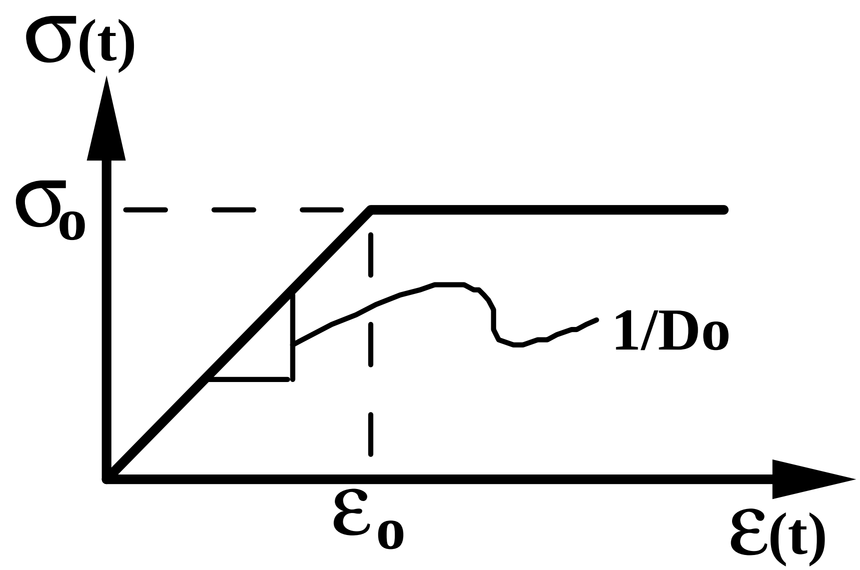
Since creep level, σ_0 is prescribed, and the loading is linear, D_0 can be determined from the initial loading slope.

$$\varepsilon_0 = D_0 \sigma_0 \quad [4]$$

and

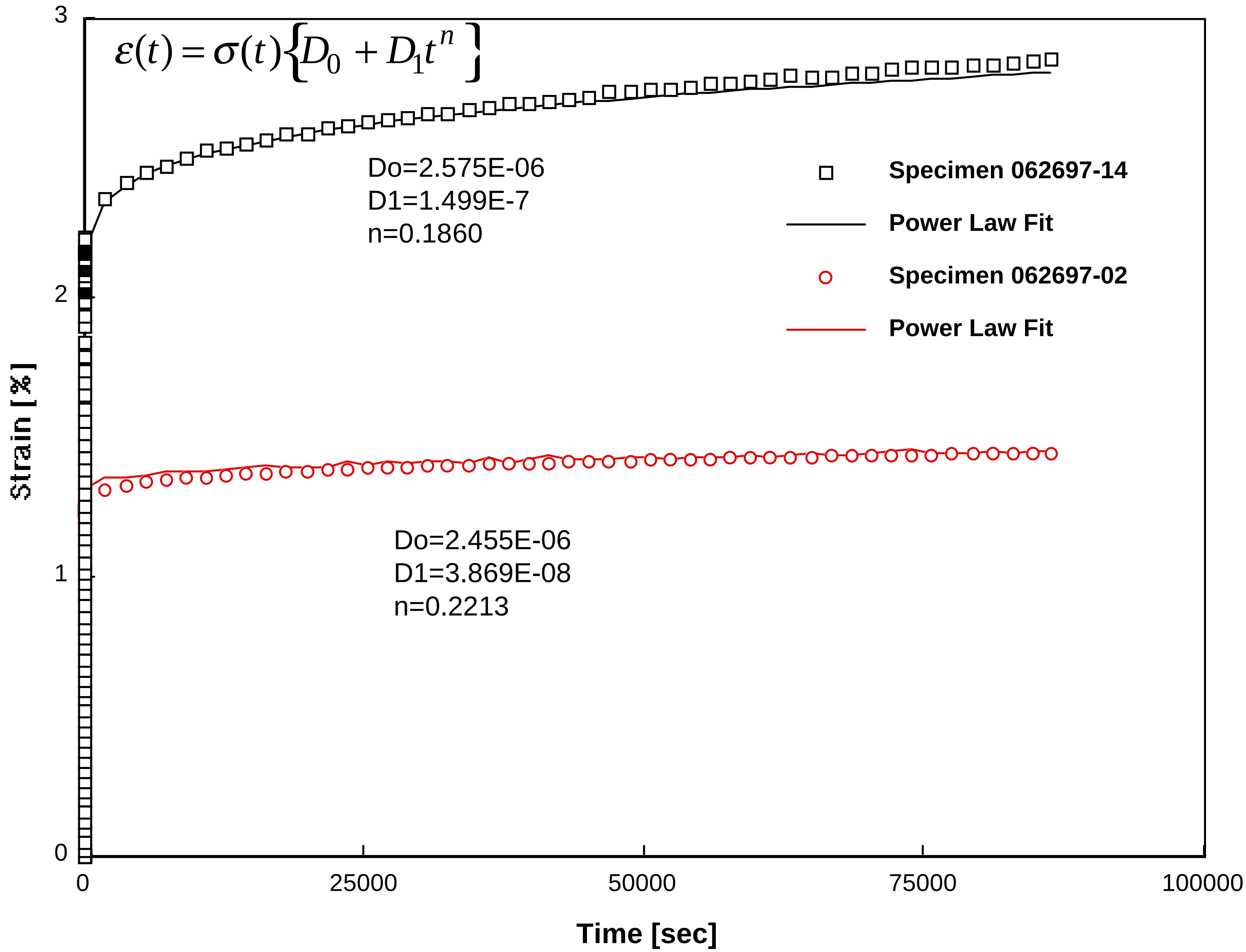
$$\varepsilon_{creep}(t) = \sigma_0 D_1 t^n \quad [5]$$

The final two parameters can be determined by taking the log of both sides of [5] and employing linear regression analysis with the creep data.



Characterization of a Structural Adhesive in Automotive Environments

Characterization of a Structural Adhesive in Automotive Environments



Characterization of a Structural Adhesive in Automotive Environments

Conclusion :

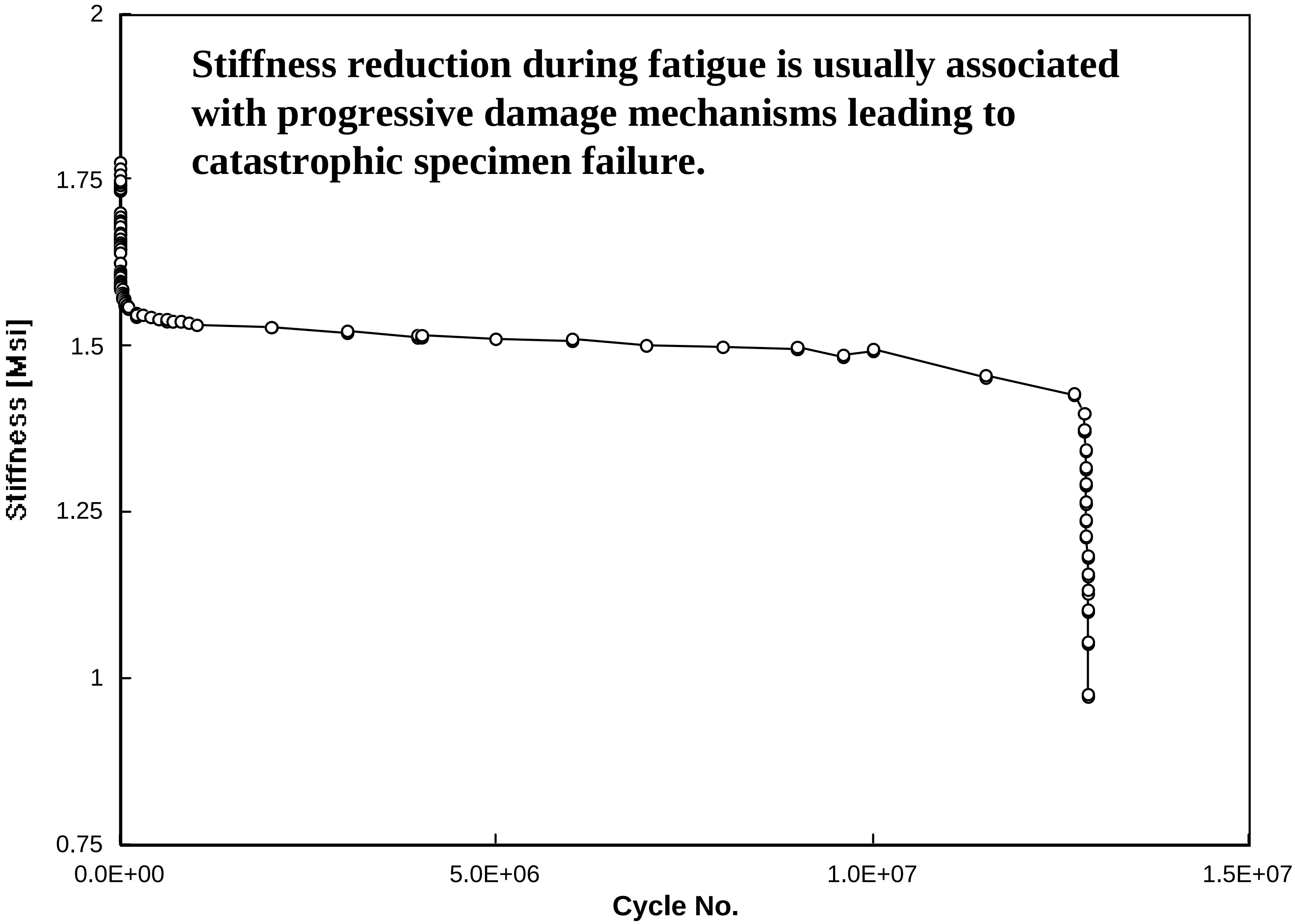
Methods to characterize the mechanical performance of adhesives and adhesive joints have been addressed through the completion of numerous test development efforts.

Comprehensive mechanical testing was carried out to assess the validity of these test methods and provide data for correlation with analytical predictions.

This information provides valuable insight to the applicability of adhesive joints as an alternative fastening technology.

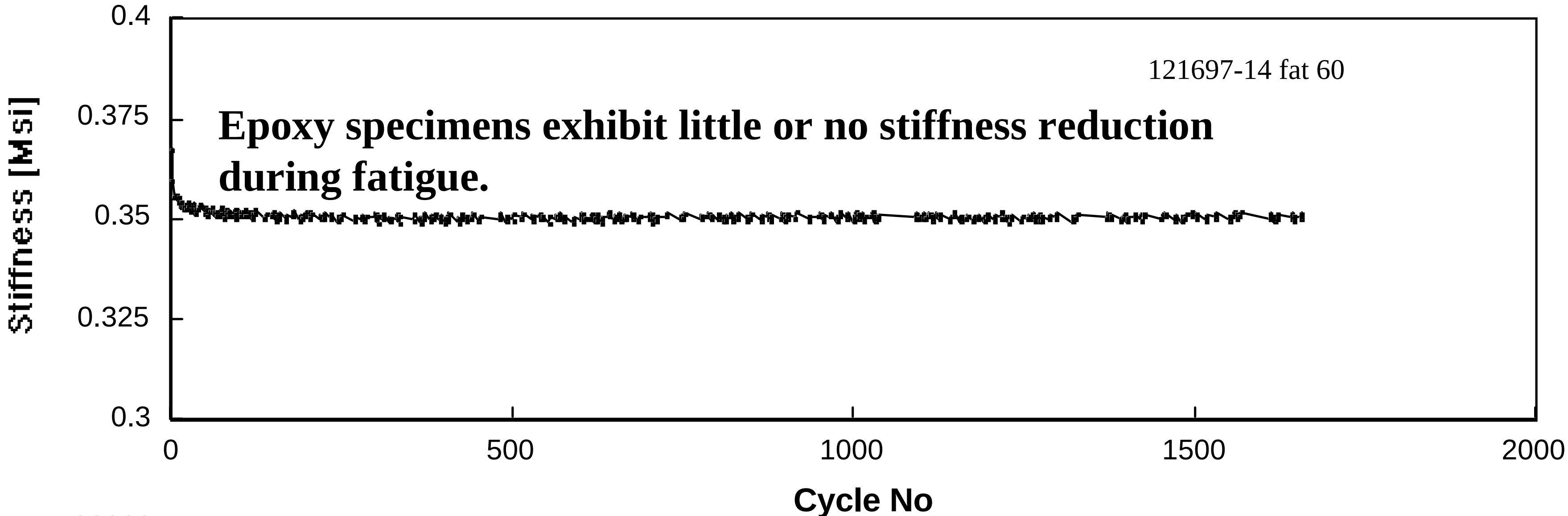
Characterization of a Structural Adhesive in Automotive Environments

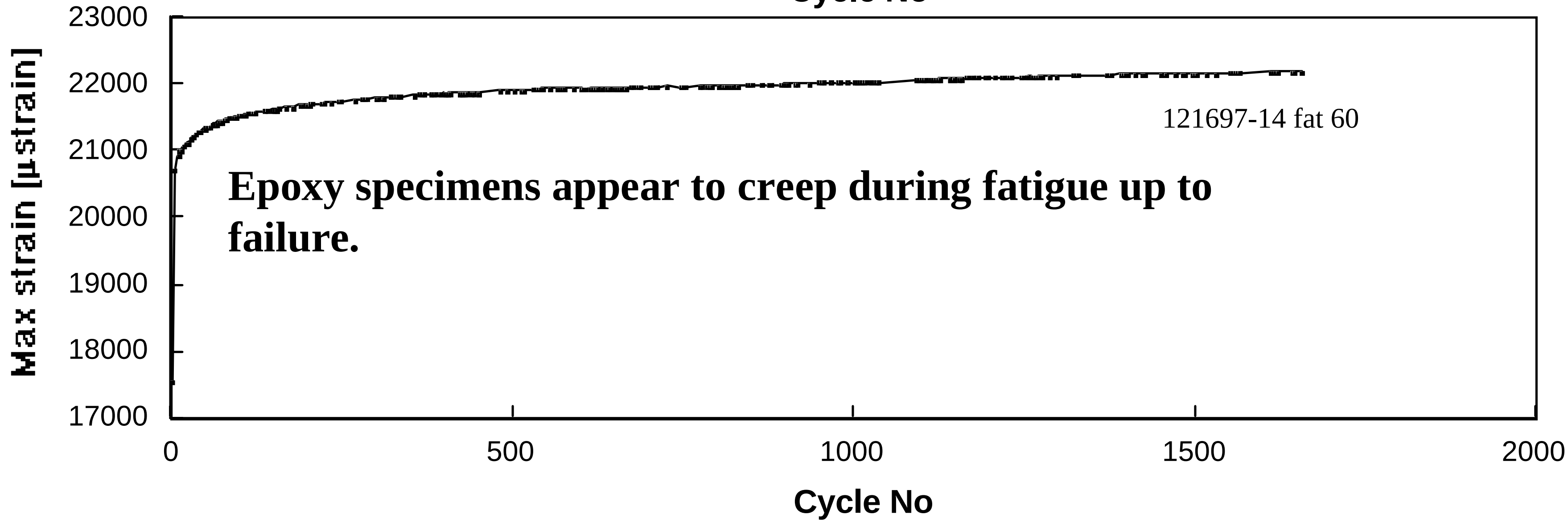
Typical Stiffness Reduction for a Composite Fatigue Test



Characterization of a Structural Adhesive in Automotive Environments

Typical Stiffness Reduction for an Adhesive Fatigue Test





Characterization of a Structural Adhesive in Automotive Environments

STRAIN RESPONSE TO SINUSOIDAL STRESS INPUT

In general, the strain response of a linear viscoelastic material to arbitrary stress input is given as:

$$\varepsilon(t) = \int_0^t D(t-\tau)\frac{d\sigma(\tau)}{d\tau}\,d\tau \quad [6]$$

$$D(t) = \left\{D_0 + D_1 t^n\right\} \quad [7]$$

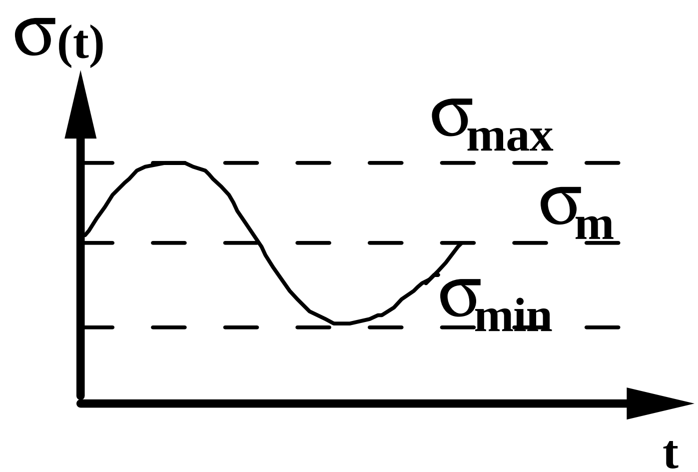
For the case of sinusoidal stress input:

$$\sigma(t) = H(t)\left\{\sigma_m + \sigma_s \sin(\omega t)\right\}$$

$$\sigma_m \equiv mean\ stress = \frac{(\sigma_{\max} + \sigma_{\min})}{2} \quad [8]$$

$$\sigma_s \equiv span\ stress = (\sigma_{\max} - \sigma_m)$$

$$\omega = 2\pi f,\ f \equiv test\ frequency$$



STRAIN RESPONSE TO SINUSOIDAL STRESS INPUT

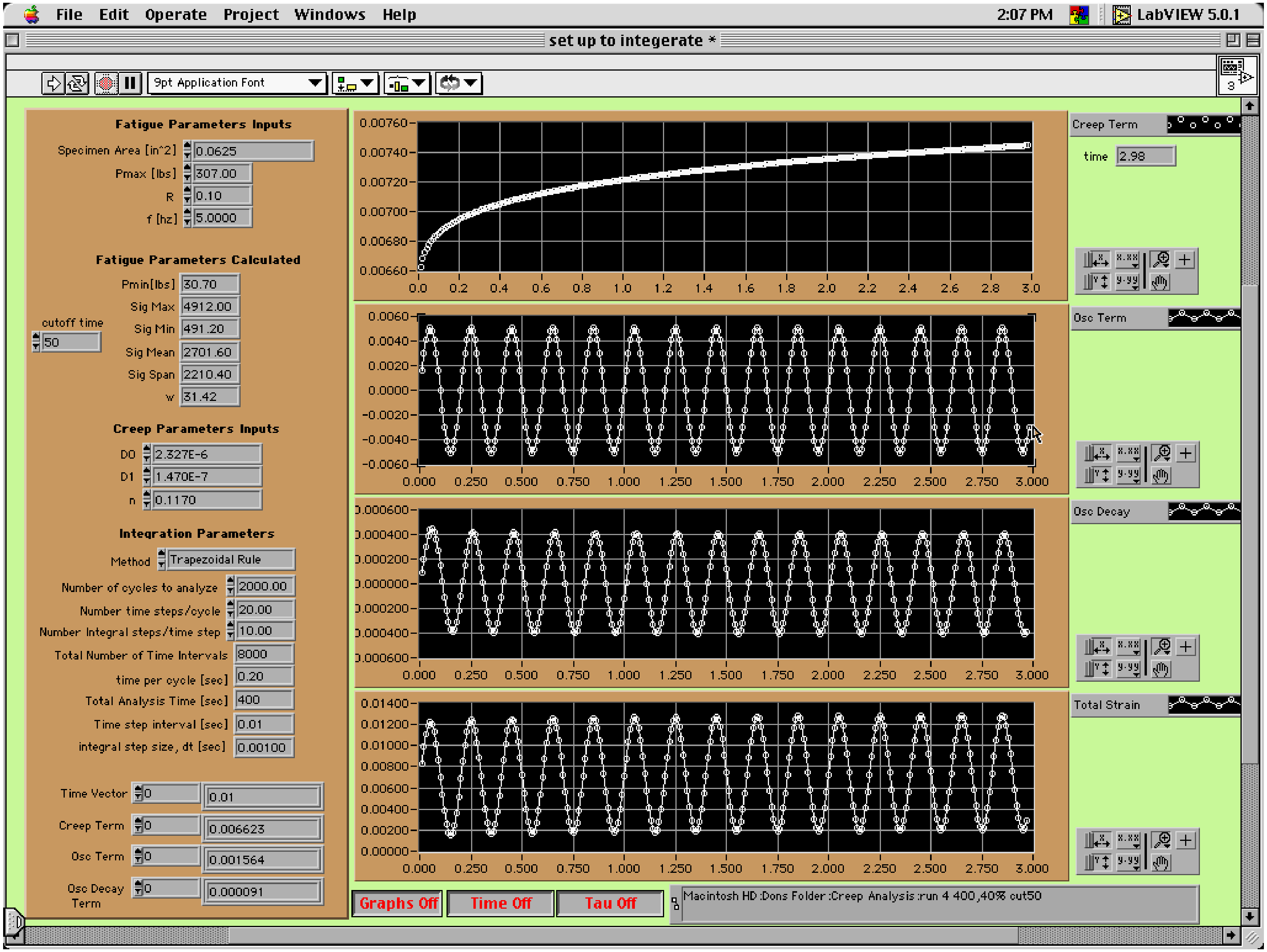
Substituting equations [8] and [7] into [6] and carrying out the integration yields:

$$\varepsilon(t) = H(t)\left\{ \begin{array}{l} \sigma_m \left(D_0 + D_1 t^n \right) + \sigma_s D_0 \sin(\omega t) \\ \sigma_s D_1 \omega \int_0^t (t-\tau)^n \cos(\omega\tau) d\tau \end{array} \right\} \qquad [9]$$

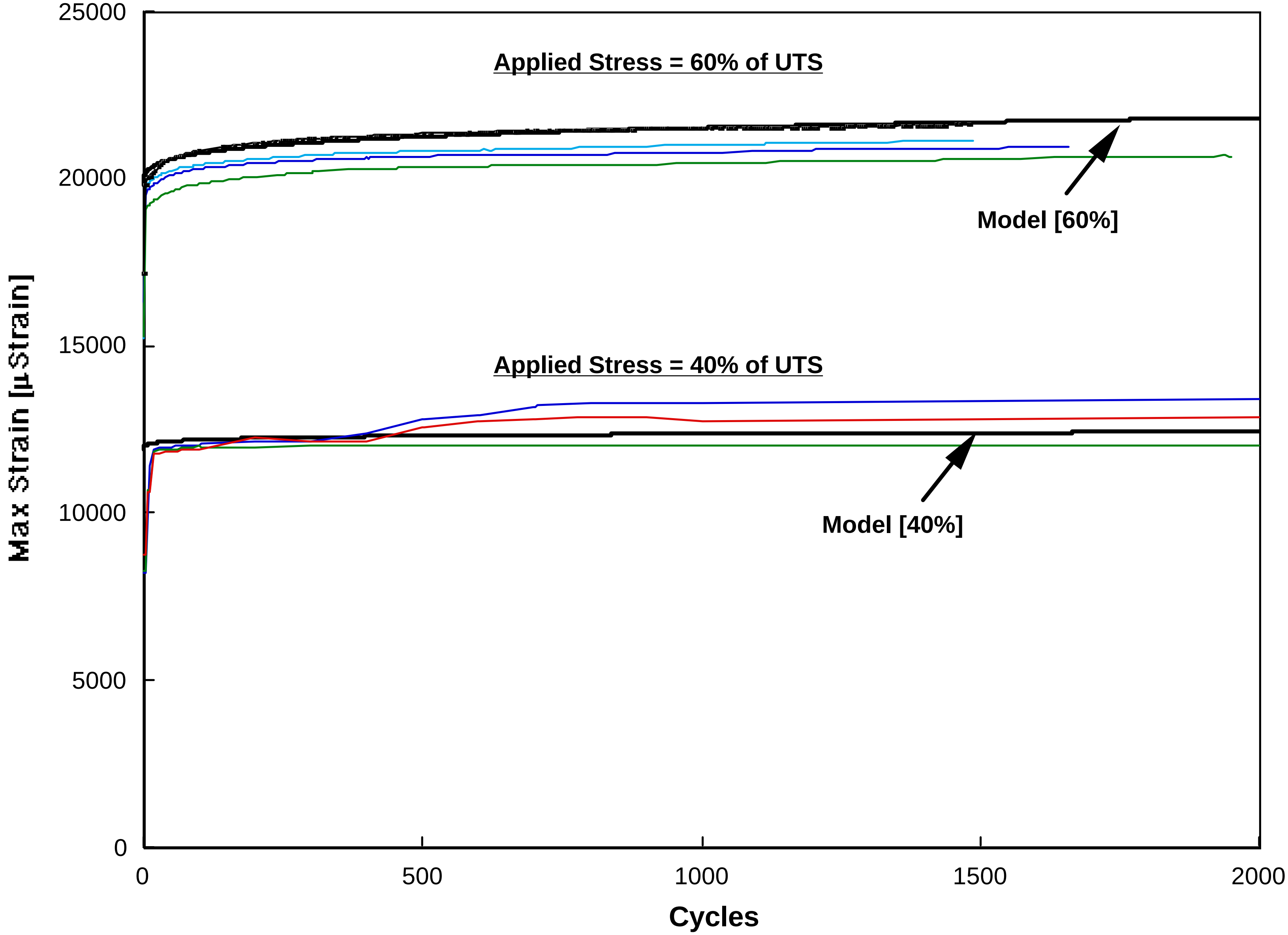
The three terms in [9] represent the total strain as a function of time for all times greater than zero. These terms can be categorized as follows:

Term 1 : “Creep” Response to the mean stress level.
Term 2 : Oscillatory elastic term.
Term 3 : Oscillatory viscoelastic term which decreases in amplitude with time.

Characterization of a Structural Adhesive in Automotive Environments



Characterization of a Structural Adhesive in Automotive Environments



Characterization of a Structural Adhesive in Automotive Environments

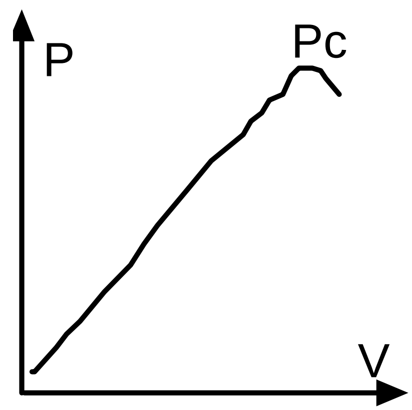
KIc Testing of Cast Epoxy

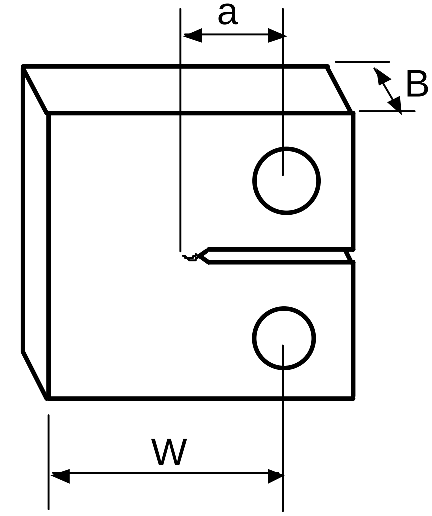
$$KIc = \frac{P_c f\left(\frac{a}{w}\right)}{B\sqrt{W}} \qquad [1]$$

$$GIc = \frac{K^2}{E'} \qquad [2]$$

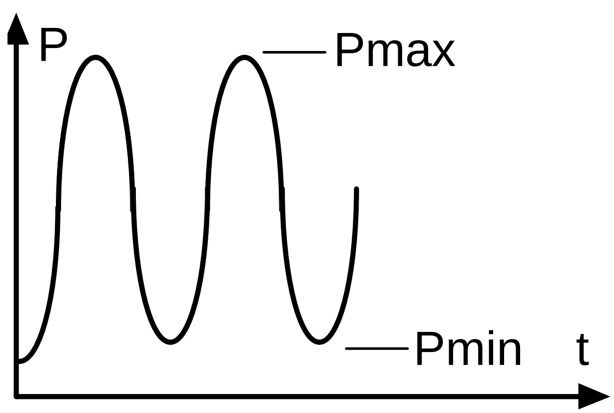
$$E' = E \quad plane\,stress$$

$$E' = \frac{E}{(1-\upsilon^2)} \quad plane\,strain$$





Pre-Cracking Procedure



$$\Delta K = \frac{\Delta P f\left(\frac{a}{w}\right)}{B\sqrt{W}} \qquad [1a]$$

$$\Delta P = P\max - P\min$$

$$K\max < 0.6KIc$$

Note: To avoid Kmax exceeding 0.6 KIc, it is necessary to "shed load" as the crack length increases.

Characterization of a Structural Adhesive in Automotive Environments

Pre-Cracking Procedure

Crack length can be determined each cycle from specimen compliance and material properties.

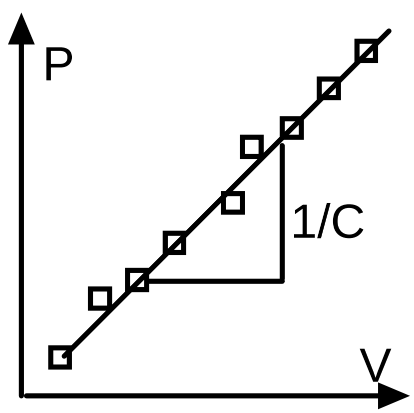
$$C = \frac{V}{P} = \frac{F\left(\frac{a}{w}\right)}{BE} \quad [3]$$

Solve [3] for (a/w) in terms of compliance:

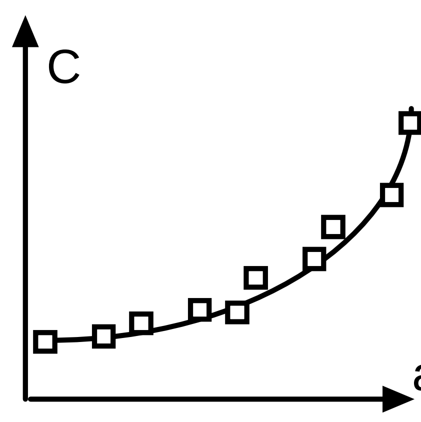
$$F\left(\frac{a}{w}\right) \Rightarrow \left(\frac{a}{w}\right) = F^{-1}\left(C = \frac{V}{P}\right) \qquad [4]$$

Graphically:

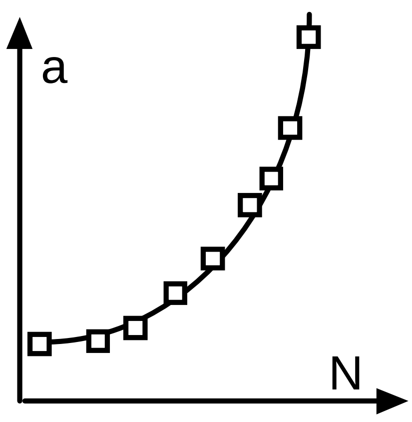
Compliance each cycle



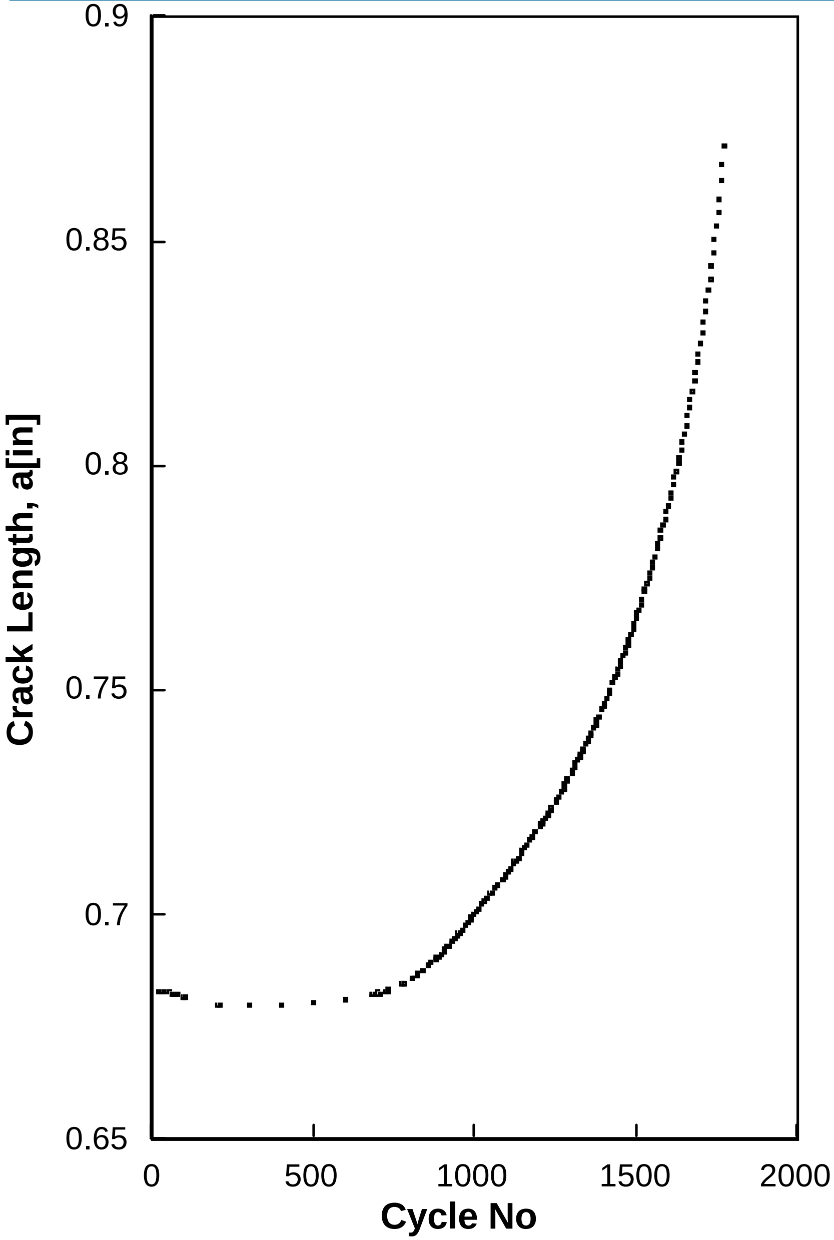
Compliance Calibration

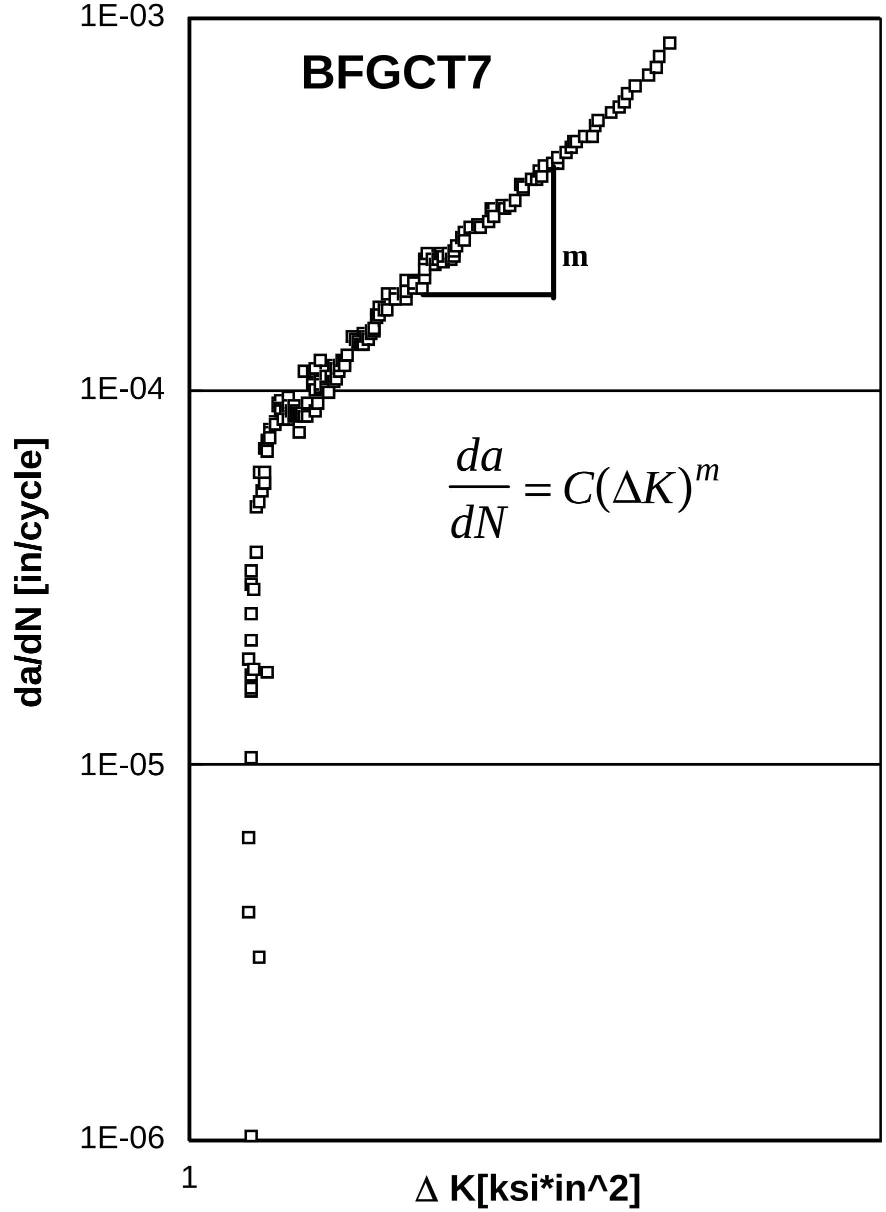


Crack Velocity



Characterization of a Structural Adhesive in Automotive Environments





Characterization of a Structural Adhesive in Automotive Environments

Compact Tension Fatigue Pre-Crack Fracture Surface