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PERKIN ELMER

DMA 7e

Theory

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DMA 7e Theory of Operation

Abstract

The Perkin-Elmer DMA 7e Dynamic Mechanical Analyzer theory, design and results are described. The measured signals, software calculations, data files and real time reporting signals are discussed. The functional components of the analyzer, along with the, design, theory, operation and specifications are described. The equations used to apply the force to the sample and the sample response is described. A general discussion of the fundamental principles of operation is provided. Standard reporting curves and their interpretation are described and typical results for a glass transition determination are discussed.

Introduction

Dynamic Mechanical Analysis (DMA) is the study of the time dependent mechanical behavior of materials. Mechanical properties of materials are determined in dynamic modes by applying an oscillating force and measuring the material's response. The results are interpreted using fundamental relationships such as Hooke's Law (for elastic behavior) and Newton's Law (for damping or viscous behavior). These results can be normalized for sample dimensions to obtain standard reporting values such as storage modulus and loss modulus.

Most materials exhibit both elastic and viscous properties and are referred to as viscoelastic materials. Dynamic Mechanical Analysis can fully characterize elastic, viscous, and viscoelastic properties as a function of temperature, time, frequency, or stress.

Experimental

The Perkin-Elmer 7 Series/UNIX Thermal Analysis Software is used to program isothermal, cooling, heating, frequency scan, and dynamic stress scan experiments using the Perkin-Elmer DMA 7e Dynamic Mechanical Analyzer as shown in Figure 1. The measured signals are sent from the DMA 7e to the software via the TAC interface. From these signals, the software calculates and saves the primary data in a data file. These and other values are also reported on the "status line" during set up and during the run.

Equipment

The DMA 7e is comprised of four functional components: motor, measuring system, detector, and environmental system, as shown in the cross-sectional diagram in Figure 2. The motor is a linear force motor that can produce 8,000 mN downward force and 6,500 mN upward force. Dynamic force can be applied at frequencies from 0.01 to 51 Hz. The force is applied to the sample through the central core rod which is suspended in a magnetic field. The force motor supports the weight of the probe and core rod, and applies the force to the sample. The vertical design of the instrument permits accurate control of the moving components and a very low compliance frame for applying force. The vertical design of the instrument also uses gravity to assist the operator in mounting samples.

The detector, a linear variable differential transformer (LVDT), measures the vertical deflection of the sample. The magnetic core of the LVDT maintains a constant horizontal distance from the coils through the 25 millimeter range. The measuring system holds the sample in place for testing and can accommodate a number of sample geometries and sample types[1,2].

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The environmental system includes the purge gas, cooling device, and furnace. The purge gas is supplied through the top of the measuring system and provides a uniform atmosphere for the sample. A number of cooling devices can be connected to the heat sink that surrounds the furnace. The furnace is of very low mass, closely coupled to the sample to enable precise temperature control. The heat sink is maintained at a relatively constant temperature and provides a uniform thermal environment for the furnace to heat or cool the sample.

Measurement

The sample is stressed by applying a sinusoidally varying **total force** (F_T) programmed in milliNewtons (mN). The total force applied to the sample is the sum of the **static force** (F_S) and **dynamic force** (F_D) with a chosen **frequency** in Hertz (Hz), as shown in Figure 3. The sample typically responds to the applied force with an oscillating displacement **amplitude** (y°), measured in micrometers (μm). The displacement amplitude corresponds to the average energy recovered in one cycle of oscillation in an elastic deformation. The sample also typically responds to the applied force with a **phase lag** (δ), reported in degrees (°). The phase lag corresponds to the average energy lost in one cycle of oscillation in a viscous deformation (Figure 4). Elastic and damping properties are calculated using fundamental relationships such as Hooke's and Newton's law.

Dynamic stress (σ^o), reported in Pascals (Pa), is calculated using the sample dimensions (form factors), amplitude, and the spring constant as shown in the table of equations. Each measuring system and geometry has a unique form factor containing sample dimensions described in Figure 5 and Figure 6. The **sensor temperature** is measured using a thermocouple, which is placed in close proximity to the sample.

Discussion

Purely elastic behavior, which is typical of springs, is described using **Hooke's law**. Hooke's Law states that when a force is applied to an elastic sample, the sample will exert an equal and opposite force that is proportional to the displacement times a constant ($F = - k_S x$). The **spring constant** (k_S) of a viscoelastic material excited by an oscillating force also depends upon the phase lag and inertial acceleration of the exciting force.

spring constant for a viscoelastic material = $k_S = \frac{F_T \cos\theta'}{y^\circ} + \omega^2 M$ (mN/mm)

where F_T is the total force applied to the sample, y° is the measured sample displacement **amplitude** in micrometers (μm), θ' (theta prime) is the **sample phase lag** in radians (rad), ω (omega) is the **angular frequency** in radians per second (rad/s) and M is the **mass** of the moving components of the analyzer (probe, core rod, clamps, and sample) in kilograms (kg).

Purely viscous behavior, which is typical of perfect fluids and often is represented by a dash-pot, is described by **Newton's Law**. Newton's law states that when a force is applied to a damping (fluid) sample, the sample will exert an equal and opposite force that is proportional to the displacement rate times a constant ($F = -Dv$). The displacement rate (v) is measured in millimeters per second (mm/s). **Sample damping** of a viscoelastic material excited by an oscillating force also depends upon the phase lag, frequency, and instrument damping. For the DMA 7e, both the spring constant and damping are automatically calculated by the software, displayed on the status line, and stored in each data file.

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damping for a viscoelastic material = $D_S = \frac{F_T \sin\theta'}{\omega\, y^o} - D_o$ (mN·s/mm)

where F_T is the total force applied to the sample, D_o is the instrument damping determined during calibration.

Results

Storage modulus (E'), reported in Pascals (Pa), is plotted versus temperature in Figure 7. Storage modulus is an indication of the ability of a material to store mechanical energy and is proportional to the sample spring constant. Storage modulus is used to characterize molecular relaxations. Storage modulus is high when molecular mobility is restricted, as is typical in cross-linked polymers and of polymers below the glass transition temperature (T_g).

When the movement of segments of a polymer chain is limited, the storage of mechanical energy is high. This is indicated by a high storage modulus (i.e., above 10^9Pa). When a polymer is heated above its T_g, an increase in free volume typically occurs. When free volume increases, molecular mobility increases and chain segments can gradually align with the applied force. When this happens in a sample, the storage modulus decreases.

Storage modulus is determined for each geometry using the sample dimensions, amplitude, and the spring constant as shown in the table of equations (Figure 5). The storage modulus is represented as a component of the total material behavior using the relationship

storage modulus = $E' = \frac{\sigma^o}{\varepsilon^o}\cos\delta$ (Pa)

where σ^o (sigma naught) is the **dynamic stress** amplitude, ε^o (epsilon naught) is the **dynamic strain** amplitude, and δ (delta) is the sample phase lag in degrees. (The dynamic strain amplitude is multiplied by 100 and reported as a percent strain (%).)

Loss modulus (E"), reported in Pascals (Pa), is plotted versus temperature in Figure 7. Loss modulus is an indication of the ability of a material to dissipate mechanical energy and is proportional to sample damping (viscous behavior). The loss modulus can be used to follow rheological changes (phase and flow) that take place during processing.

The shape of the loss modulus curve as a function of time or temperature can be correlated with viscosity curves on the same scale for most thermoset cures. Loss modulus is determined for each geometry using the sample dimensions, amplitude and the damping as shown in the table of equations (Figure 5). The loss modulus is represented as a component of the total material behavior using the relationship

loss modulus = $E'' = \frac{\sigma^o}{\varepsilon^o}\sin\delta$ (Pa).

Tangent delta (tan δ) is plotted versus temperature in Figure 7. Tangent delta is an indication of the overall mechanical properties of a material and is often referred to as the mechanical loss factor. It is an indicator of the relative amounts of energy stored and lost.

When the sample is dominated by elastic properties, tangent delta is low; when the sample is dominated by viscous properties, tangent delta is high. Tangent delta is exactly equal to one when both elastic and viscous properties are present in equal proportions. Tangent delta is

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used to characterize molecular relaxations and identify rheological transformations. It is defined by the relationship

$$\text{tangent delta} = \tan\delta = \frac{\omega D_S}{k_S} = \frac{E''}{E'}.$$

All other curves are calculated from the storage and loss modulus. For example, the **Complex modulus** (E*) reported in Pascal (Pa) is a representation of the bulk material behavior. The complex modulus, also called the DMA modulus, is a representation of the complete viscoelastic behavior of a material. The complex modulus contains both the elastic component (storage modulus) and the viscous component (loss modulus). The complex modulus is defined by the relationship

$$\text{complex modulus} = E^* = \sqrt{E'^2 + E''^2} \text{ (Pa)}.$$

Typical Applications

The DMA 7e can be used to characterize the effects of thermal and mechanical history including aging, annealing and orientation. The DMA 7e is most often used to characterize thermomechanical behavior of polymers. This includes the identification of the melt, alpha, beta, gamma and secondary dispersion transitions. The DMA 7e is sensitive to the effects of blending, grafting and copolymerization, The DMA 7e can be used to characterize molecular relaxations of polymers as they are altered by the effect of additives, tackifiers, oils, waxes, fillers, pigments and other modifiers.

Identification of the glass transition (T_g) is one of the more common uses of the DMA 7e. At temperatures below the T_g, a polymer typically behaves like a glassy material. At higher temperatures, the polymer typically behaves like a viscoelastic material. The T_g is used to identify the temperature range of the glassy to viscoelastic transition. This information is used to predict final product performance. The T_g can be used as an indicator of the degree of cure of a thermoset, dimensional stability, specific strength and modulus.

Quality assurance and quality control analysis are performed using the DMA 7e. Composite materials can be quickly and accurately analyzed for modulus. Materials with a modulus from $1x10^3$ Pa to $3x10^{11}$ Pa (1 kPa to 300 GPa) can be characterized with the DMA 7e. Modulus is sensitive to the degree of orientation, filler content, crystallinity, degree of cure, staging, post cure, and other processing effects. The storage modulus is often used as an indicator of physical properties such as brittle point, impact properties, creep, strength and dimensional stability.

Physical properties of thin films and fibers can also be characterized using the DMA 7e. Processing parameters often give rise to varying physical properties in the final product. The physical properties often result from changes in molecular structure and thermal history.

Isothermal cure and dynamic cure scans are performed on a wide variety of curing materials using the DMA 7e. Materials can be analyzed from a liquid to a solid state in a single experiment using the cup and plate geometry. Characterization and optimization of the curing process can result in faster curing cycles, lower internal stresses and higher degrees of cure.

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Stress/strain analysis of films and fibers are performed using the DMA 7e and the extension measuring system. Typical stress/strain analysis includes the identification of the linear region, proportional limit, yield point, drawing and ultimate strength.

Conclusion

Dynamic Mechanical Analysis can fully characterize elastic, viscous, and viscoelastic properties of a material as a function of temperature, time, frequency, and stress. Results from DMA analysis are interpreted using fundamental relationships such as Hooke's Law (for elastic behavior) and Newton's Law (for damping or viscous behavior). These results can be normalized for sample dimensions to obtain standard reporting values such as storage and loss modulus.

References

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5 Flory, P.J. *Principles of Polymer Chemistry*; Cornell University: Ithaca, N Y, 1986.

6 Turi, E. *Thermal Characterization of Polymeric Materials*: Academic: New York, 1981.

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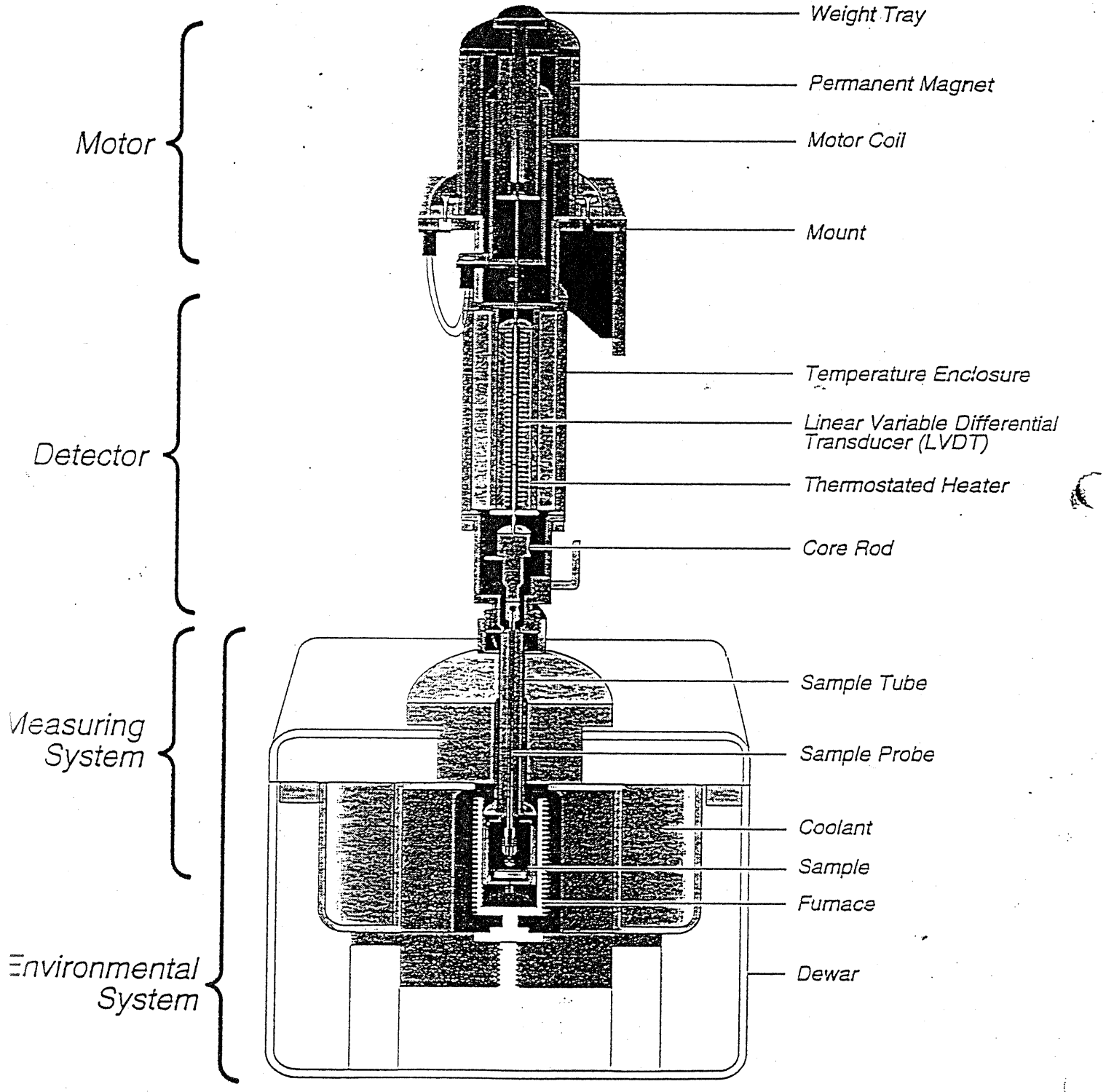
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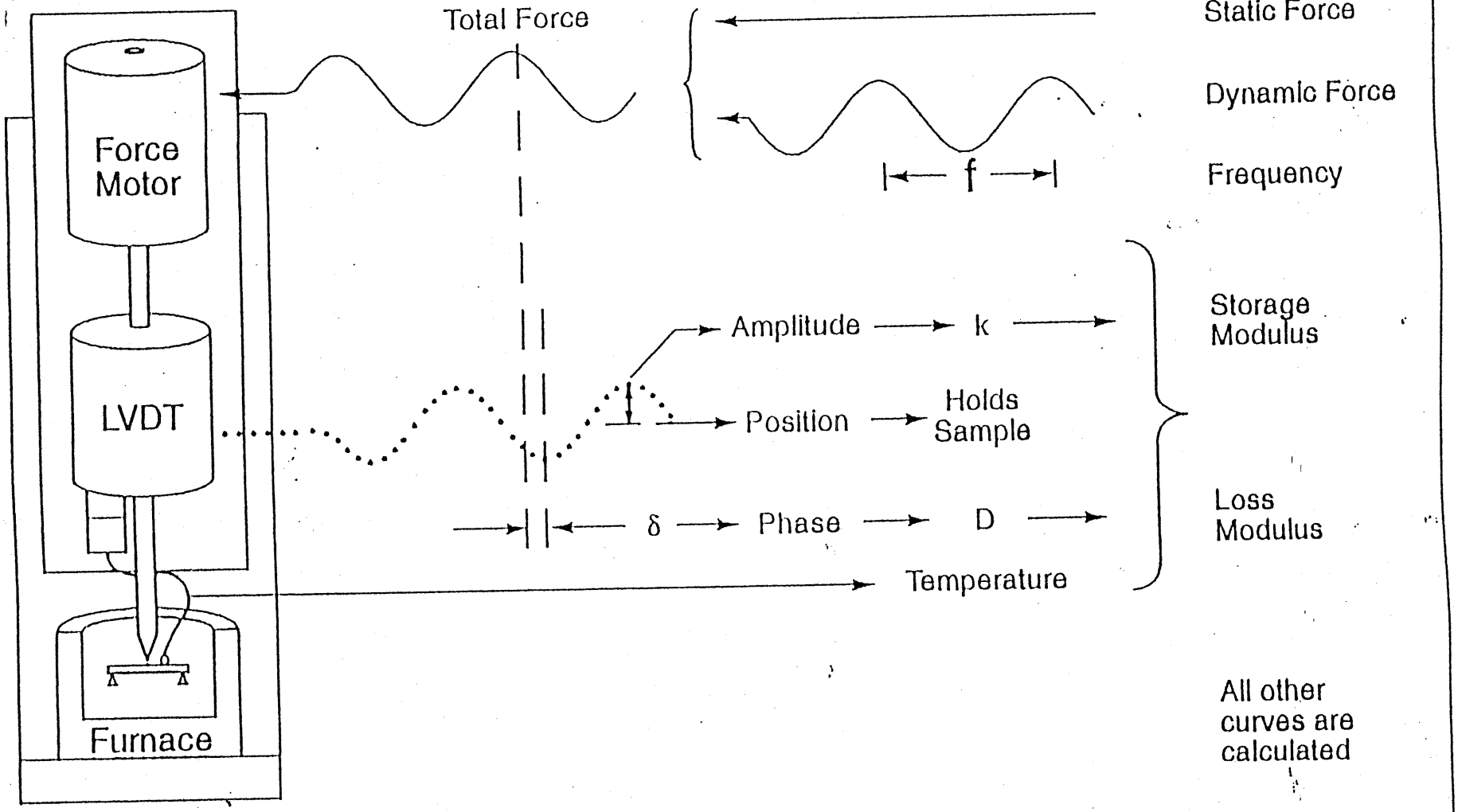
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DMA 7e Operation

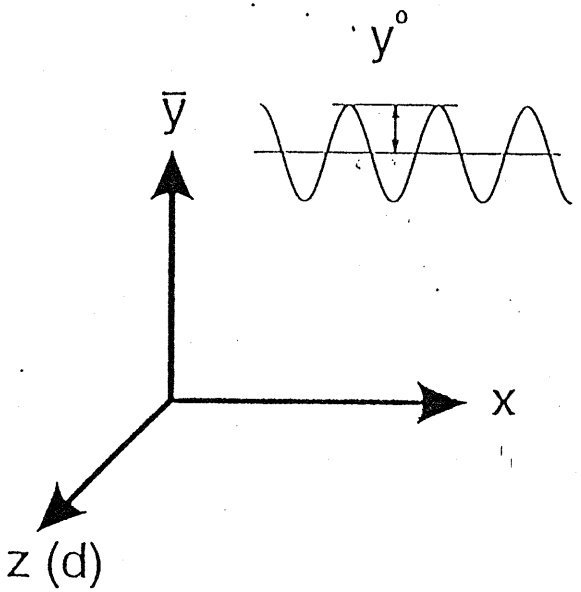


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DMA 7e Dynamic Equations and Form Factors

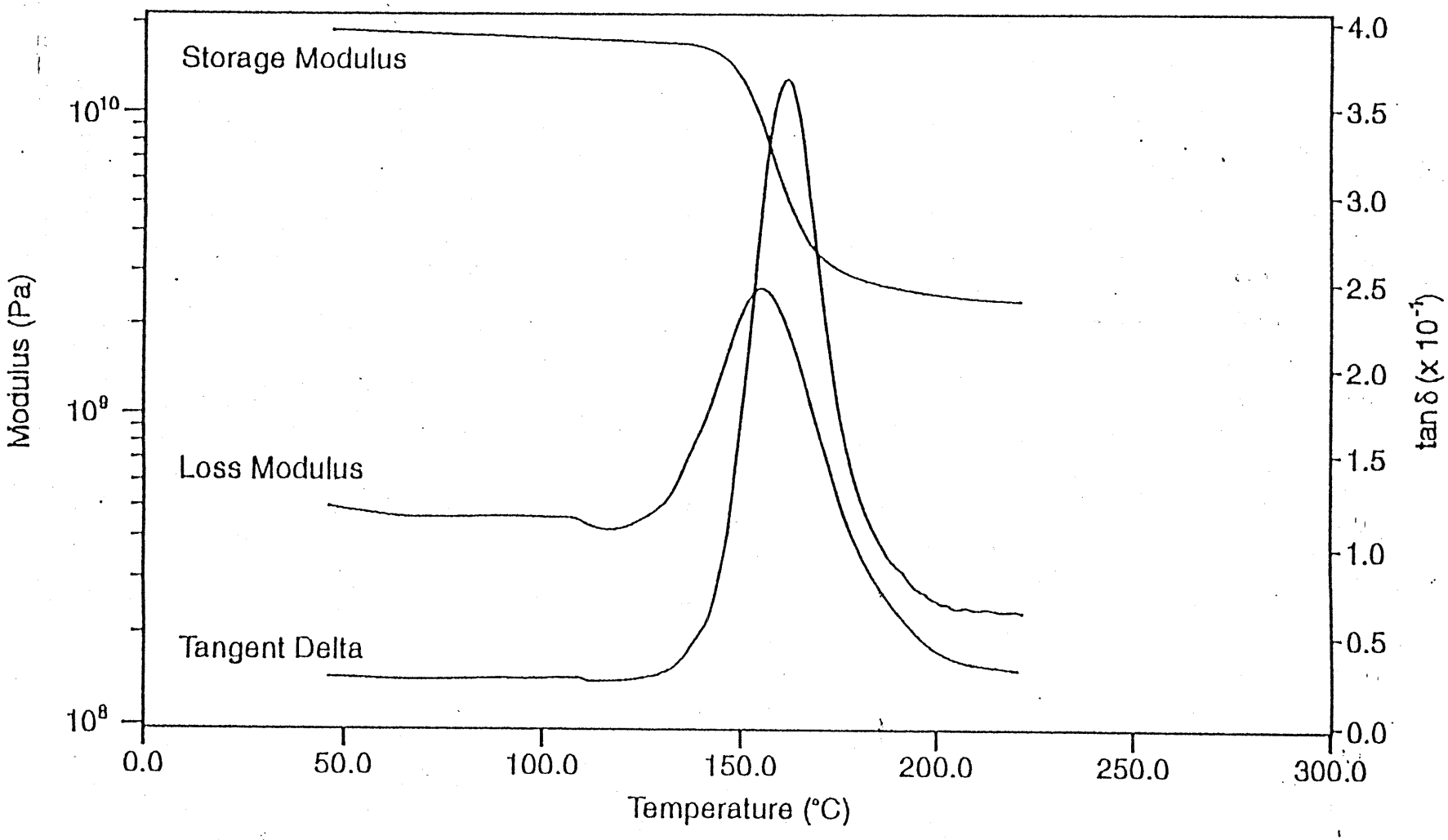
Sample spring constant, k_s (nM/mm)
Sample damping, D_s (mN • s/mm)
Sample width, x (mm)
Sample depth, z, (mm)
Sample diameter, d (mm)
Sample height, y (mm)
Average sample height, $\bar{y}$ (mm)
Average sample amplitude, y^o (μm)
Storage modulus = $\sigma/\varepsilon \cos \delta$
Loss modulus = $\sigma/\varepsilon \sin \delta$
Sample stress, σ (Pa)
Sample strain, ε (%)
Frequency, ω (rad/sec)
Geometry form factor (circled values)



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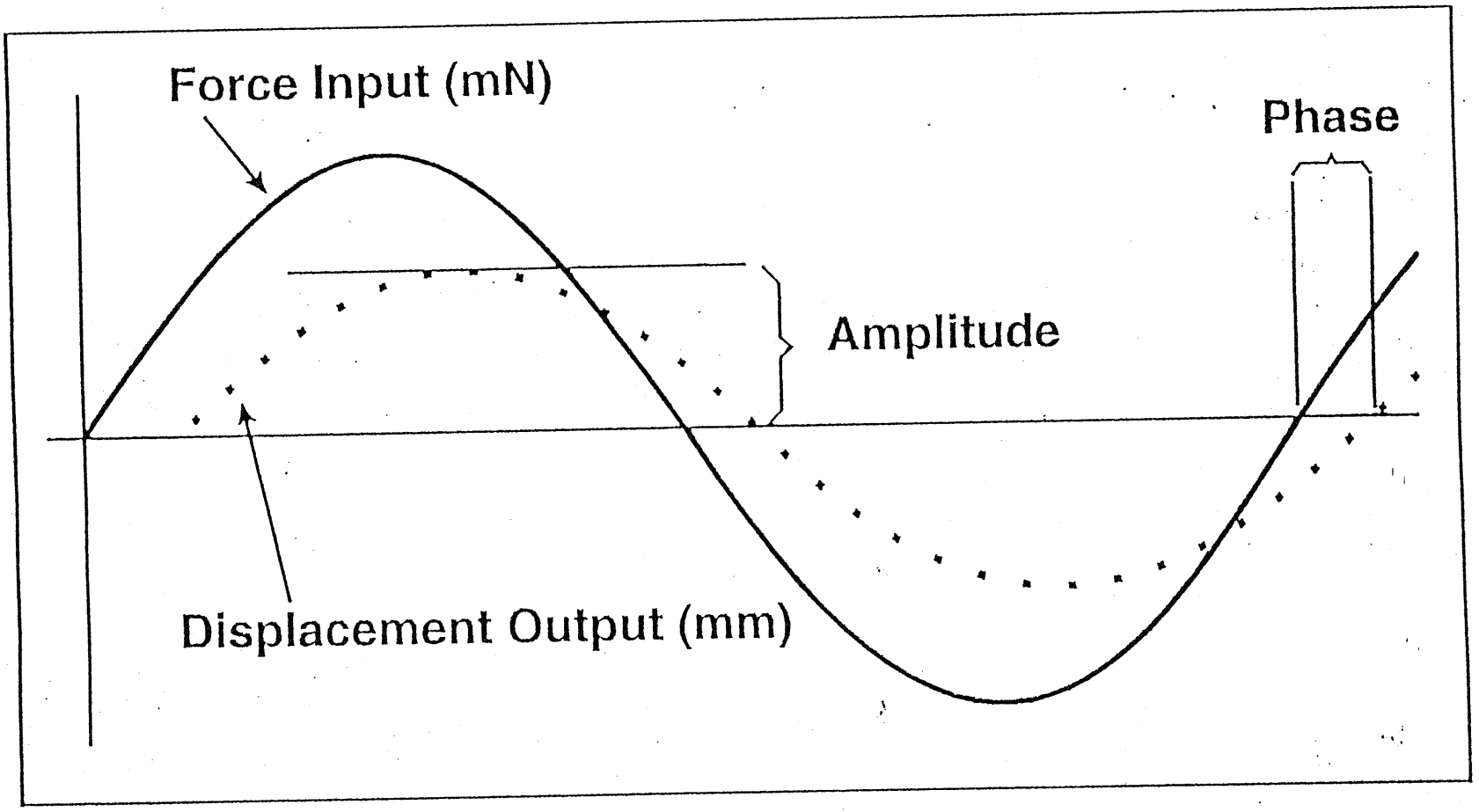
Epoxy-Glass Composite



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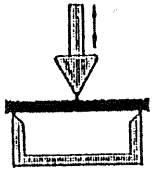
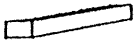
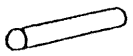
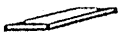
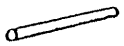
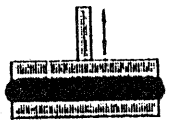
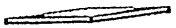
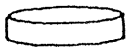
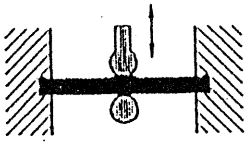
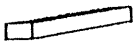
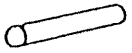
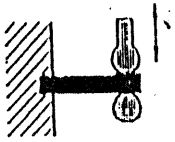
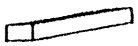
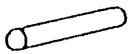
DMA Force and Displacement



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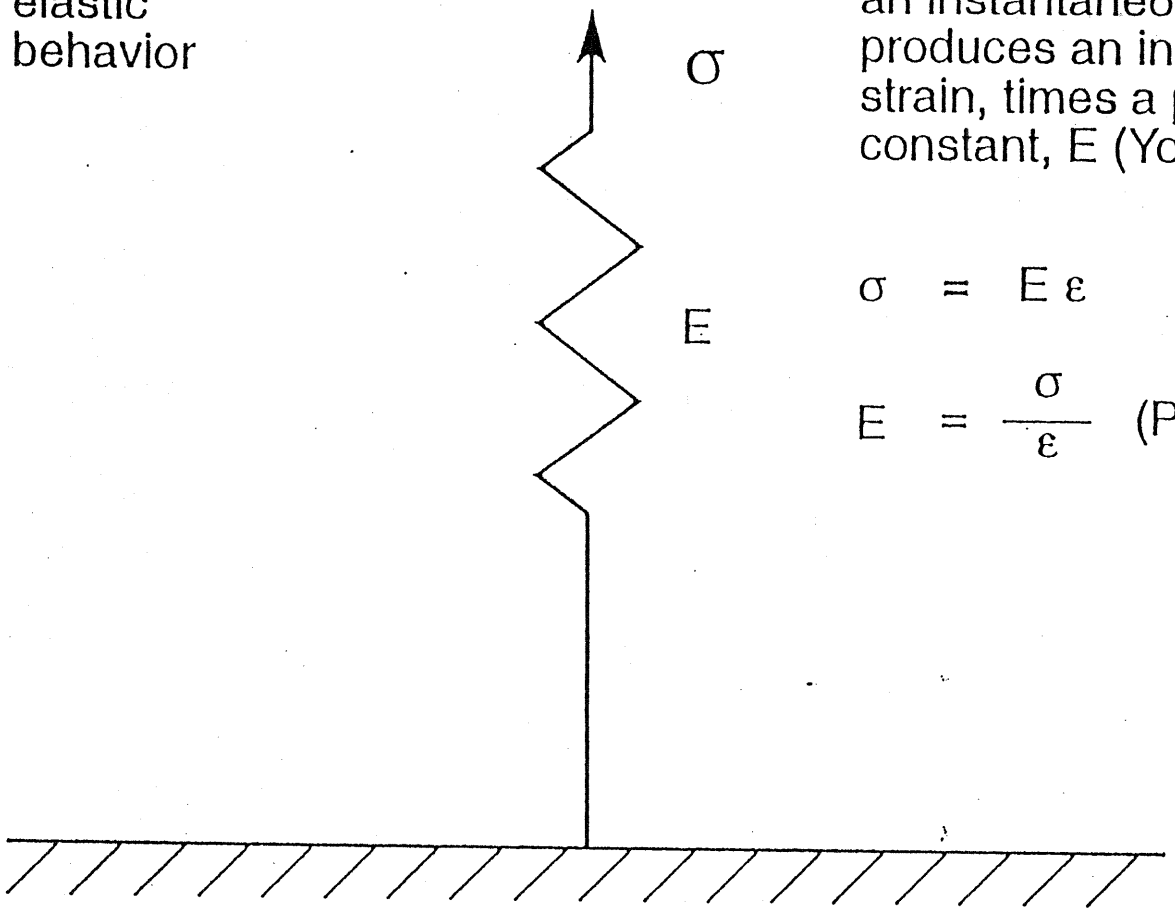
DMA 7e Dynamic Equations and Form Factors

Measuring System		Geometry		Storage Modulus	Loss Modulus	Stress	Strain
Flexure		Rectangle		$\frac{k_s x^3}{4 z y^3}$	$\frac{\omega D_s x^3}{4 z y^3}$	$\frac{3 k_s y^o x}{2 z y^2}$	$\frac{6 y^o y}{x^2}$
		Cylinder		$\frac{4 k_s x^3}{3 \pi d^4}$	$\frac{4 \omega D_s x^3}{3 \pi d^4}$	$\frac{8 k_s y^o x}{\pi d^3}$	$\frac{6 y^o d}{x^2}$
Tension		Film		$\frac{k_s \bar{y}}{x z}$	$\frac{\omega D_s \bar{y}}{x z}$	$\frac{k_s y^o}{x z}$	$\frac{y^o}{\bar{y}}$
		Fiber		$\frac{k_s \bar{y}}{\pi d^2}$	$\frac{4 \omega D_s \bar{y}}{\pi d^2}$	$\frac{4 k_s y^o}{\pi d^2}$	$\frac{y^o}{\bar{y}}$
Compression		Rectangle		$\frac{k_s \bar{y}}{x z}$	$\frac{\omega D_s \bar{y}}{x z}$	$\frac{k_s y^o}{x z}$	$\frac{y^o}{\bar{y}}$
		Disk		$\frac{k_s \bar{y}}{\pi d^2}$	$\frac{4 \omega D_s \bar{y}}{\pi d^2}$	$\frac{4 k_s y^o}{\pi d^2}$	$\frac{y^o}{\bar{y}}$
Dual Cantilever		Rectangle		$\frac{k_s x^3}{16 z y^3}$	$\frac{\omega D_s x^3}{16 z y^3}$	$\frac{3 k_s y^o x}{4 z y^2}$	$\frac{12 y^o y}{x^2}$
		Cylinder		$\frac{k_s x^3}{3 \pi d^4}$	$\frac{\omega D_s x^3}{3 \pi d^4}$	$\frac{4 k_s y^o x}{\pi d^3}$	$\frac{12 y^o d}{x^2}$
Single Cantilever		Rectangle		$\frac{4 k_s x^3}{z y^3}$	$\frac{4 \omega D_s x^3}{z y^3}$	$\frac{6 k_s y^o x}{z y^2}$	$\frac{3 y^o y}{2 x^2}$
		Cylinder		$\frac{64 k_s x^3}{3 \pi d^4}$	$\frac{64 \omega D_s x^3}{3 \pi d^4}$	$\frac{32 k_s y^o x}{\pi d^3}$	$\frac{3 y^o d}{2 x^2}$

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Spring Unit Model

Describes elastic behavior



Hooke's Law states that an instantaneous stress produces an instantaneous strain, times a proportionality constant, E (Young's Modulus)

$$\sigma = E\,\varepsilon \quad \text{or}$$

$$E = \frac{\sigma}{\varepsilon} \quad (\text{Pa})$$

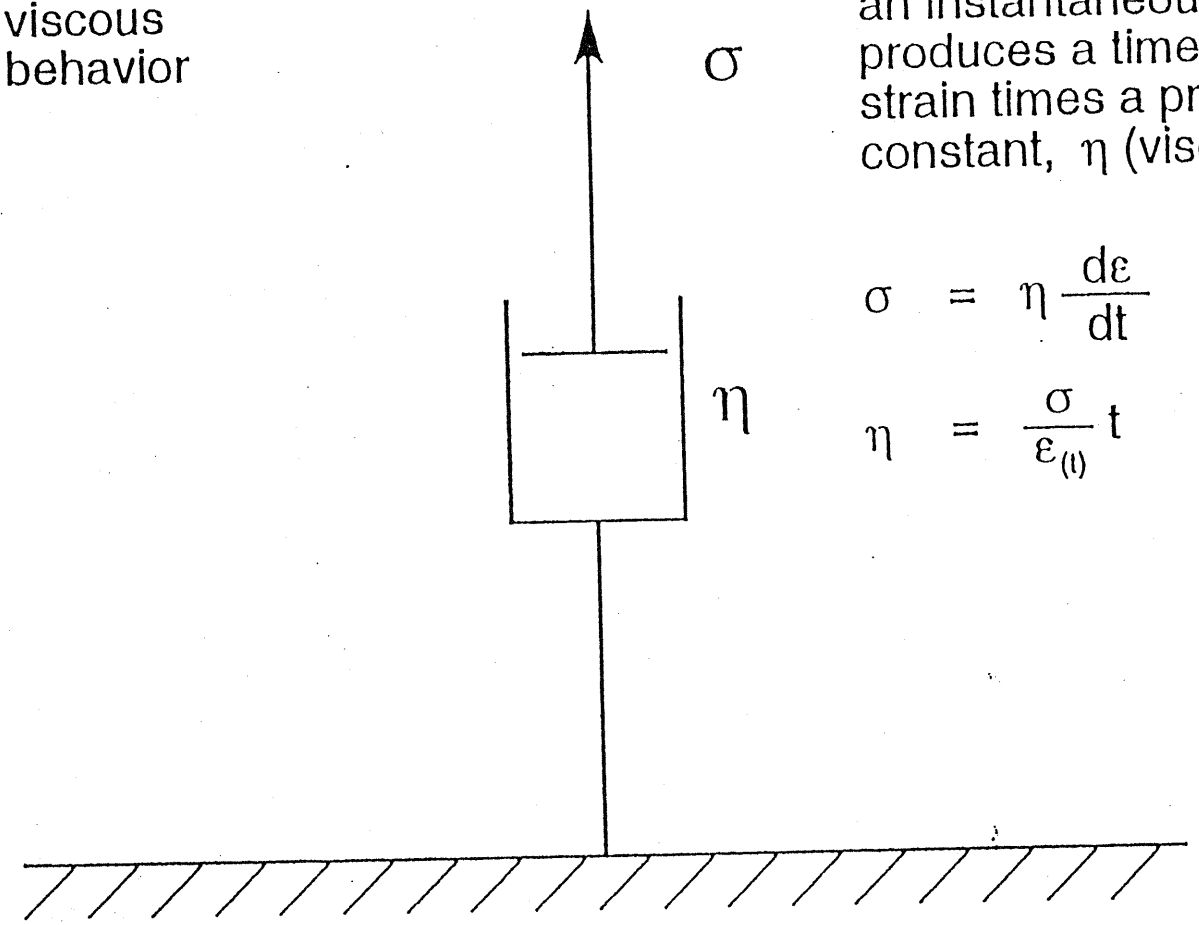
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Aklonis, MacKnight 1983

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Dashpot Unit Model

Describes viscous behavior



Newton's Law states that an instantaneous stress (σ) produces a time dependant strain times a proportionality constant, η (viscosity)

$$\sigma = \eta \frac{d\varepsilon}{dt} \quad \text{or}$$

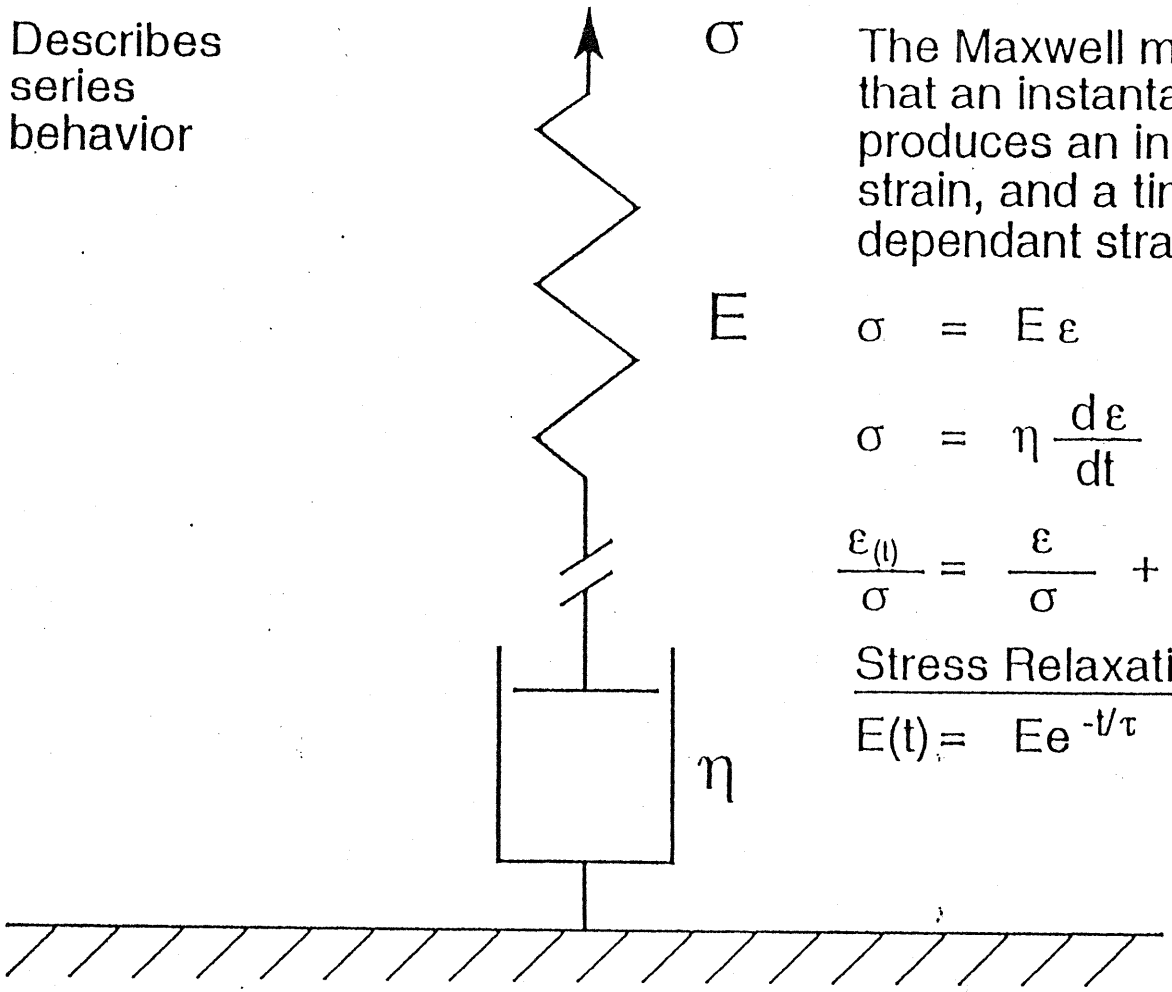
$$\eta = \frac{\sigma}{\varepsilon_{(l)}} t \quad (Pa \cdot s)$$

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Maxwell Unit Model (Element)

Describes series behavior



The Maxwell model states that an instantaneous stress produces an instantaneous strain, and a time dependant strain ($\varepsilon_{(l)}$)

$$\sigma = E\,\varepsilon \quad \text{and}$$

$$\sigma = \eta \frac{d\varepsilon}{dt} \quad \text{combine to give}$$

$$\frac{\varepsilon_{(l)}}{\sigma} = \frac{\varepsilon}{\sigma} + \frac{t}{\eta}$$

Stress Relaxation

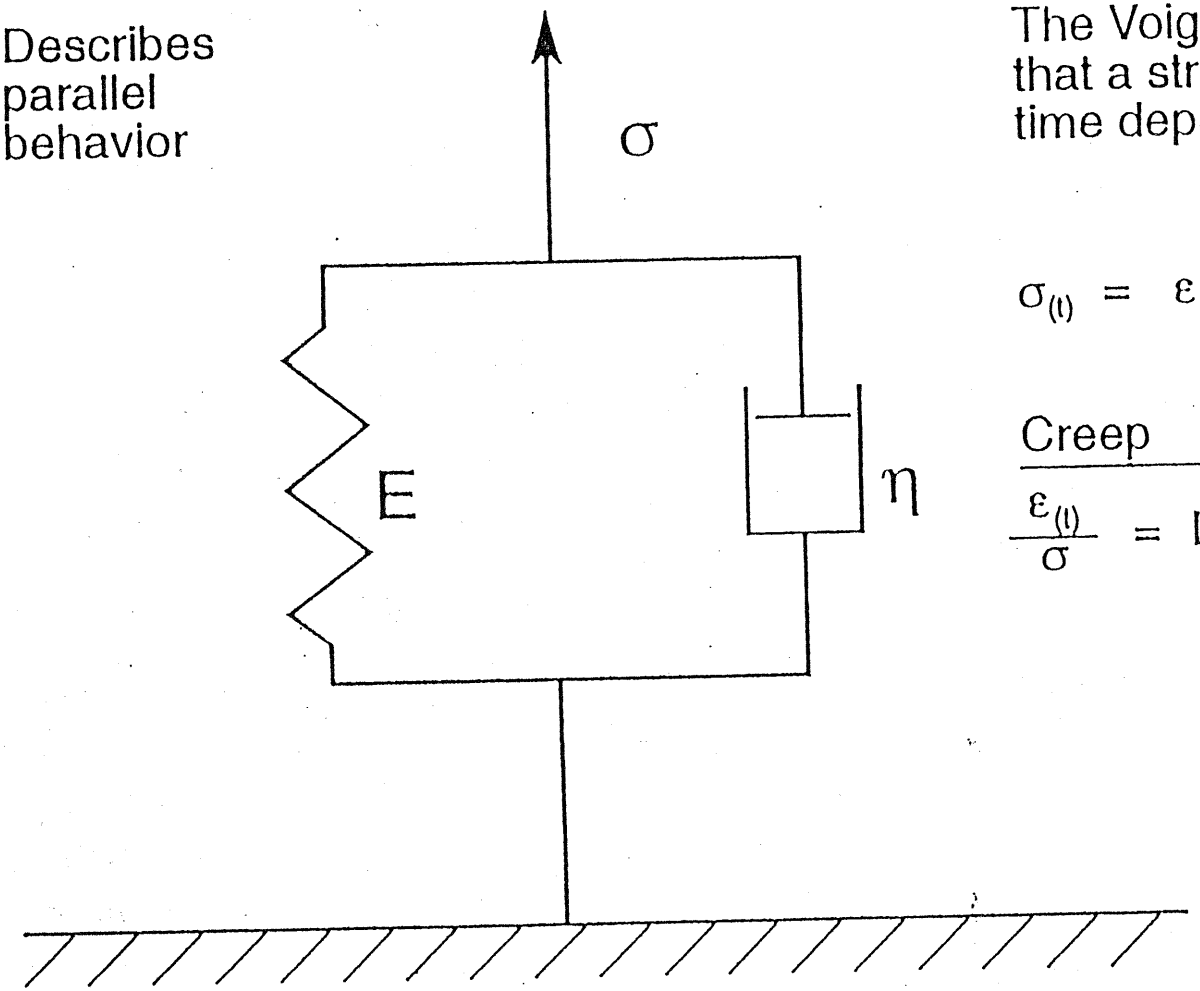
$$E(t) = Ee^{-t/\tau} \quad (Pa)$$

4085C03

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Voight Unit Model

Describes parallel behavior



The Voight model states that a stress produces a time dependent strain ($\varepsilon_{(t)}$)

$$\sigma_{(t)} = \varepsilon_{(t)} E + \eta \frac{d\varepsilon}{dt}(t)$$

Creep

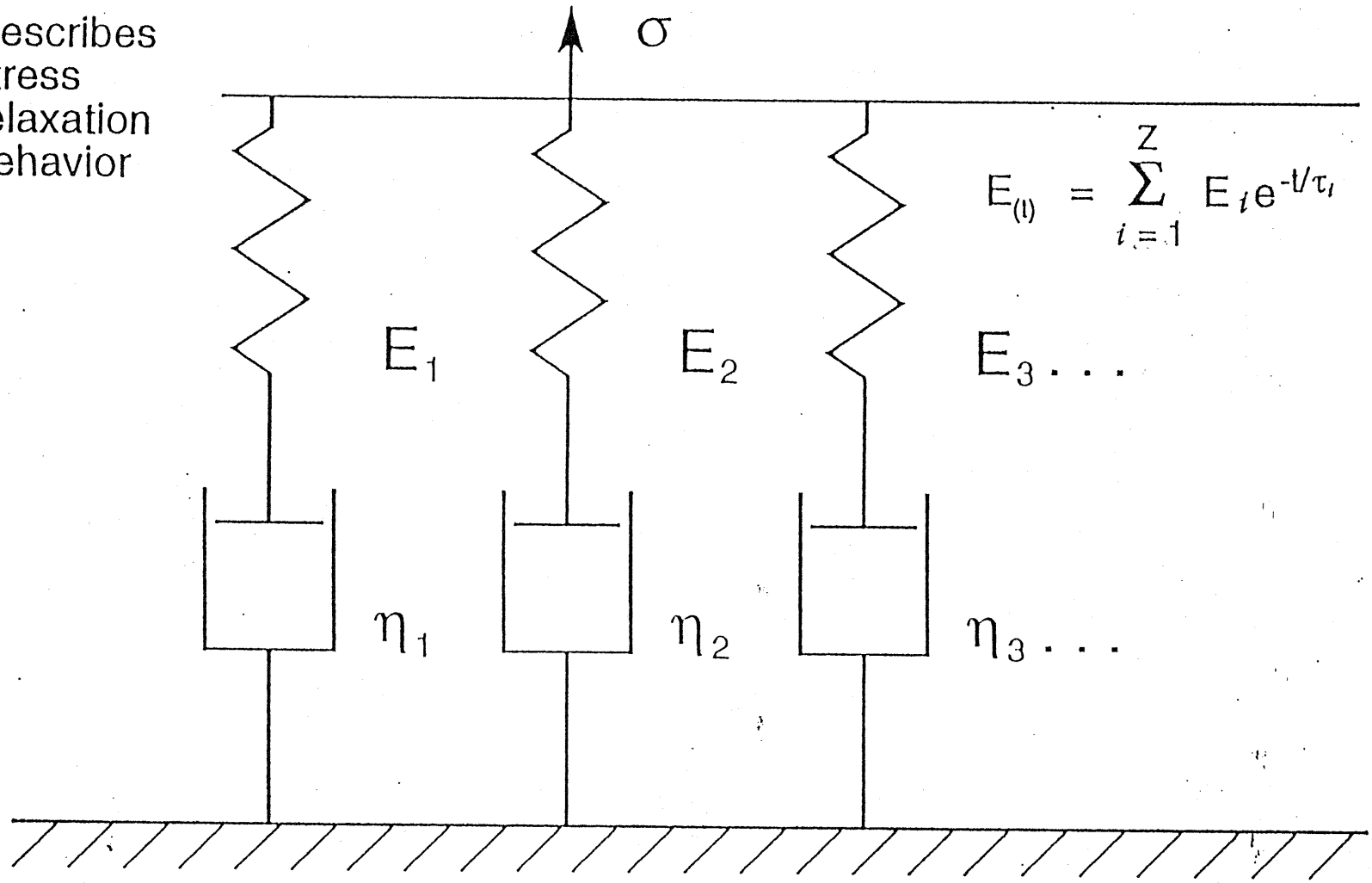
$$\frac{\varepsilon_{(t)}}{\sigma} = D\left(1 - e^{-t/\tau}\right) \qquad (Pa^{-1})$$

4085C04

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Maxwell - Weichert Model

Describes stress relaxation behavior



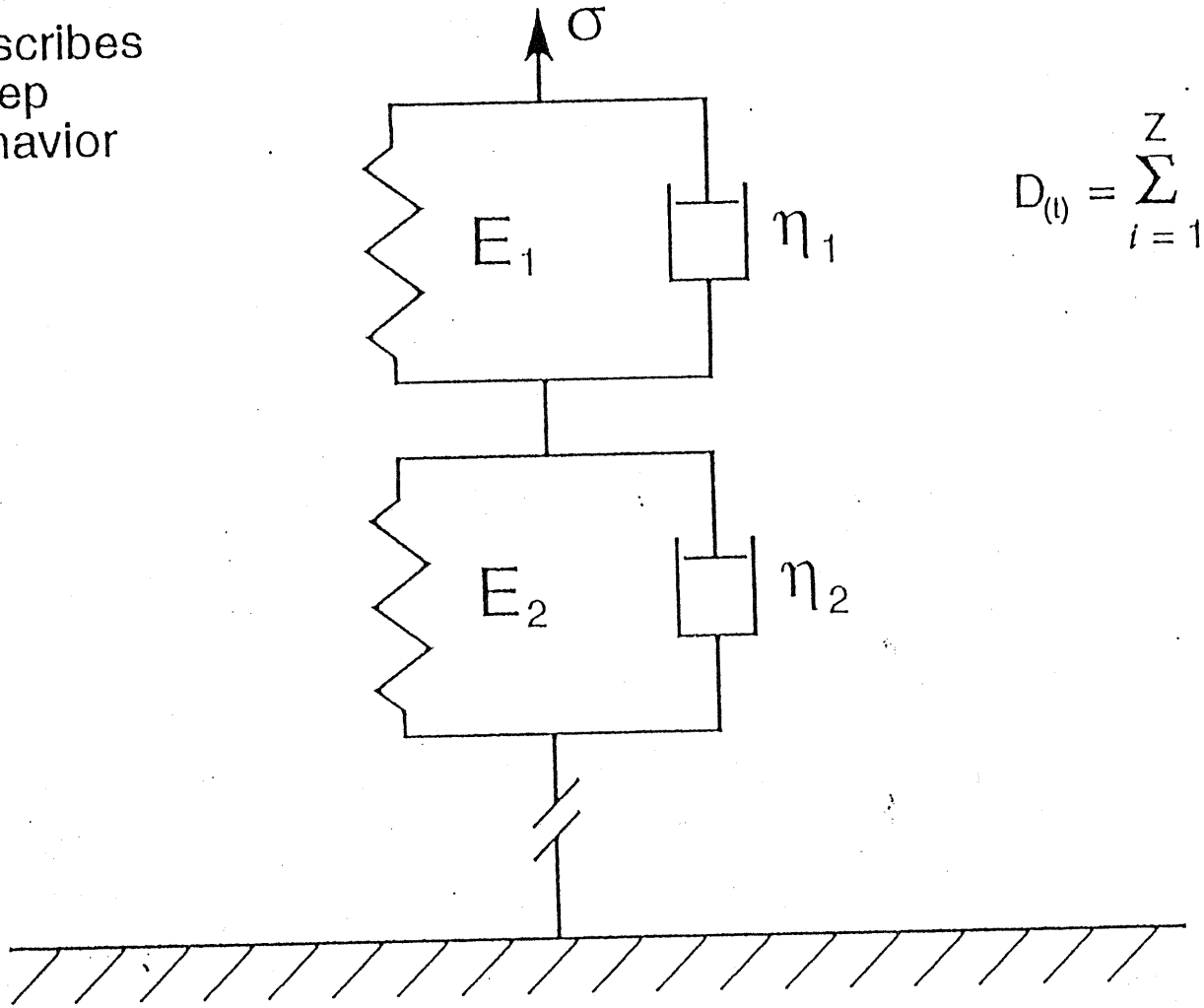
$$E_{(t)} = \sum_{i=1}^{Z} E_i e^{-t/\tau_i}$$

4085C05

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Voight - Kelvin Model

Describes creep behavior



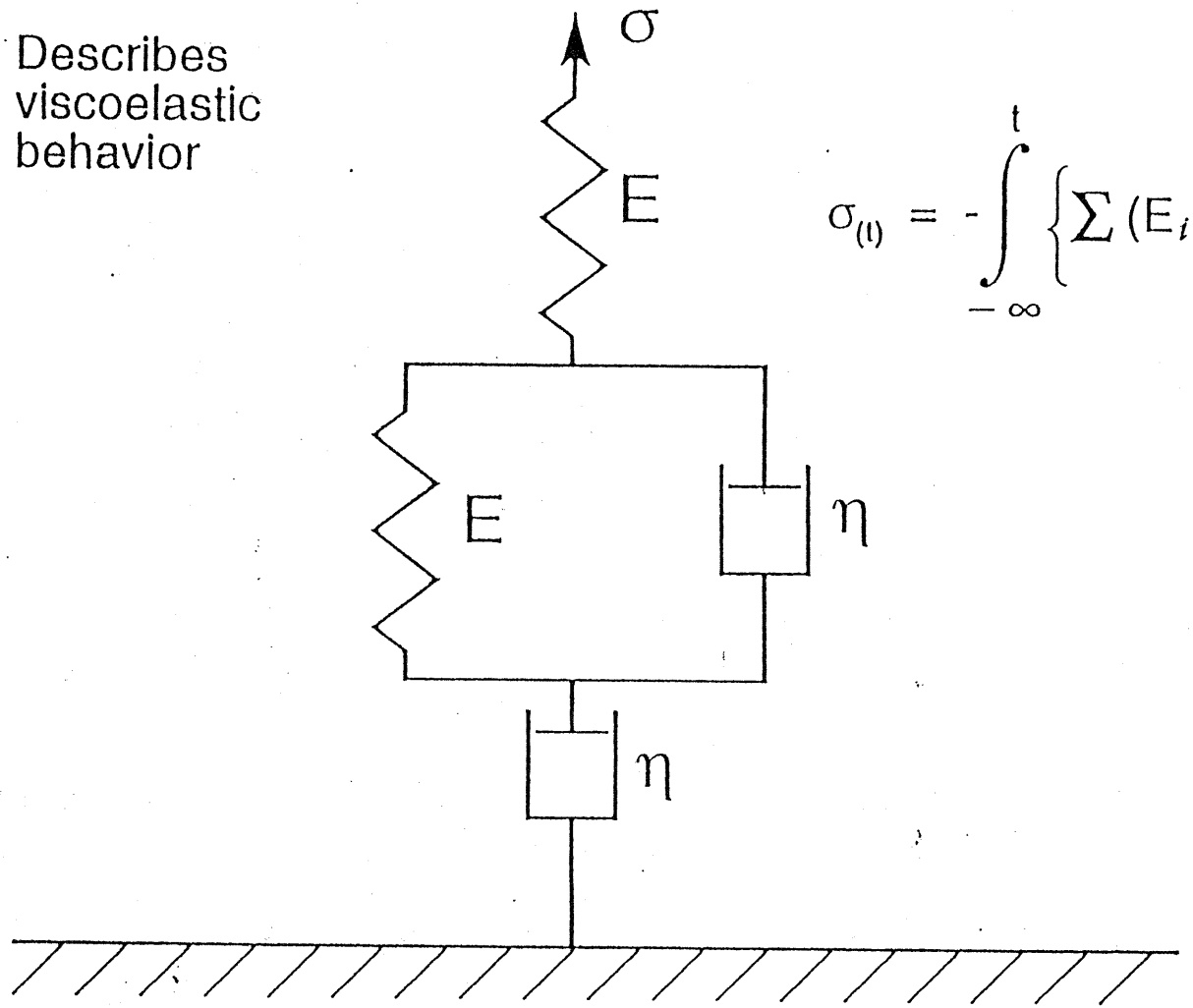
$$D_{(t)} = \sum_{i=1}^{Z} D_i (1 - e^{-t/\tau_i})$$

4085C06

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Viscoelastic Unit Model

Describes viscoelastic behavior



$$\sigma_{(t)} = -\int_{-\infty}^{t} \left\{ \sum (E_i / \tau_i)\, e^{-\frac{(t - t')}{\tau_i}} \right\} t_{(t,\, t')}\, dt$$

4085C07

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Young's Modulus

The Ratio of Stress to Strain

or

Storage Modulus

The Ratio of Stress to Strain

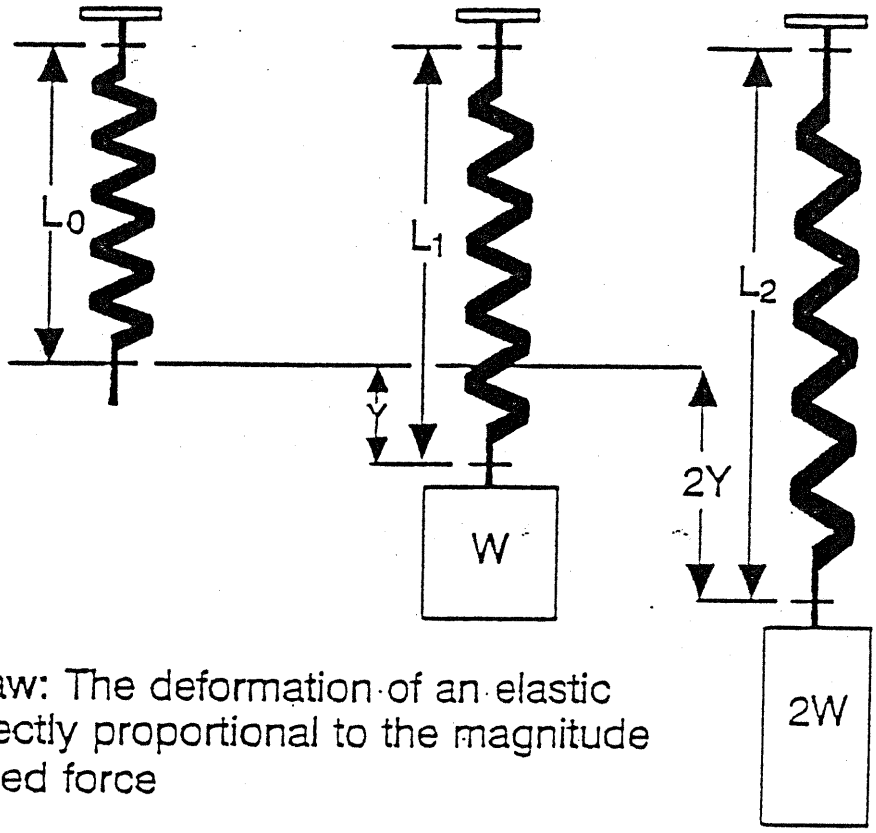
or

Elasticity

The Ratio of Stress to Strain

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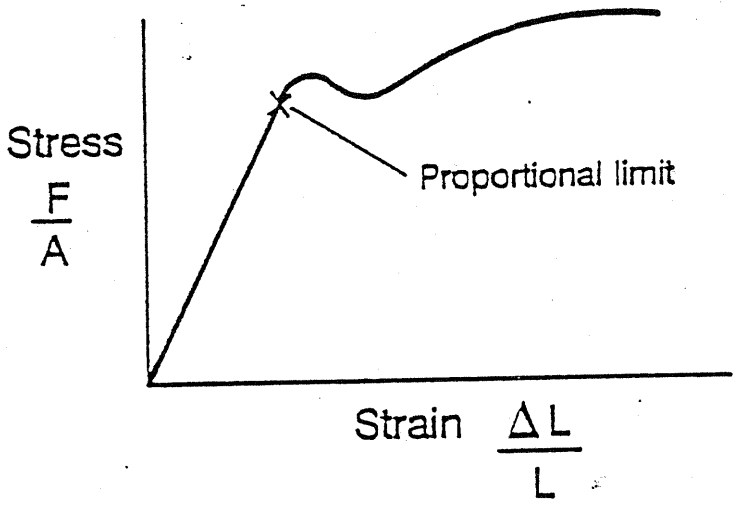
Elasticity



Hooke's Law: The deformation of an elastic body is directly proportional to the magnitude of the applied force

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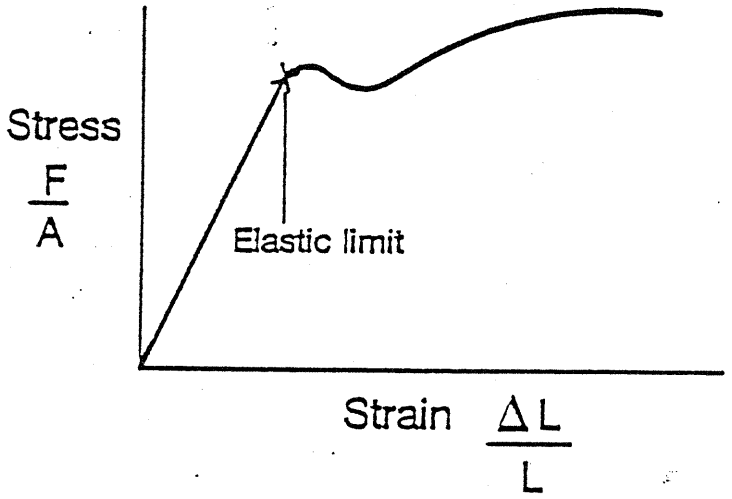
Modulus of Elasticity



The Proportional Limit is the point on a stress-strain curve at which it begins to deviate from the straight-line relationship between stress and strain.

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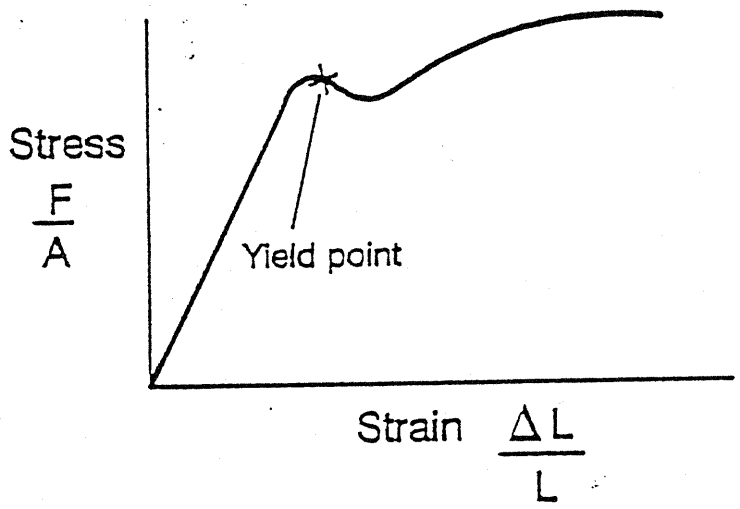
Modulus of Elasticity



The Elastic Limit is the maximum stress a sample may be subjected to and still return to its original length upon release of the load.

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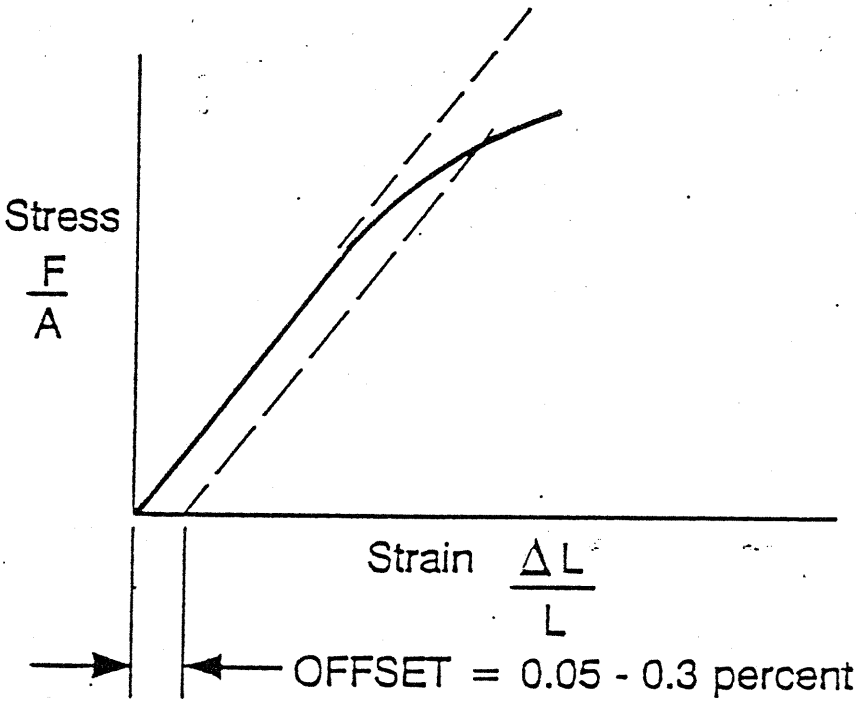
Modulus of Elasticity



The Yield Point is a point on the stress-strain curve at which there is a sudden increase in strain without a corresponding increase in stress.

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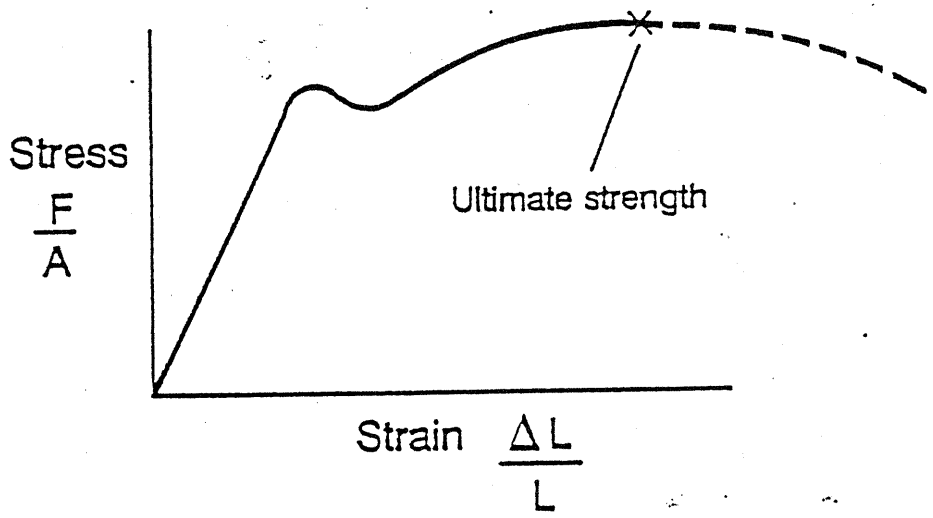
Modulus of Elasticity



The yield strength is the stress which will induce a specified permanent set, usually 0.05 to 0.3 percent which is equivalent to a strain of 0.0005 to 0.003. The yield strength is useful for materials which have no yield point

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Modulus of Elasticity

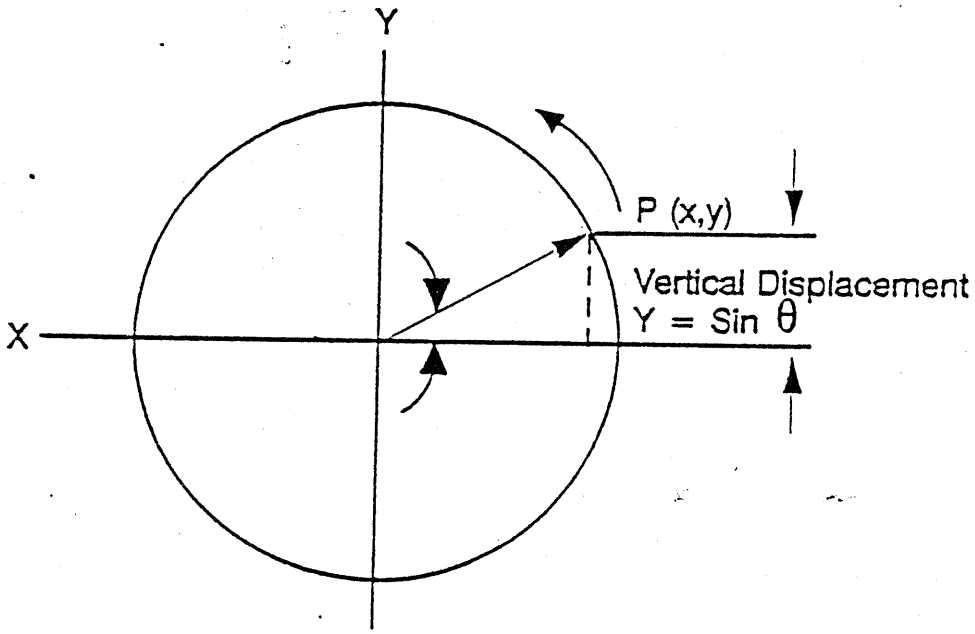


The ultimate strength is the maximum stress value obtained from a stress-strain curve.

The ultimate strength is the maximum stress, based on the original area, which is developed in the material before rupture.

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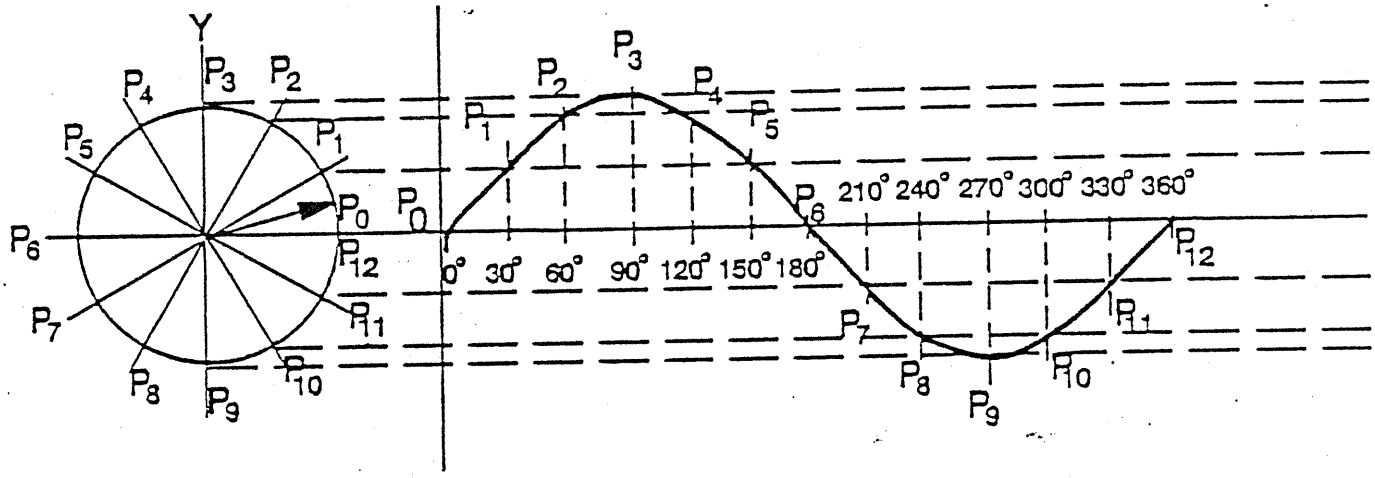
Rotating Vectors - the Sine Wave



$$\text{Sin } \theta = \frac{\text{Opposite}}{\text{Hypotenuse}}$$

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Sine Wave

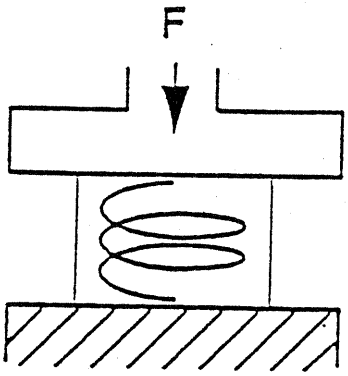


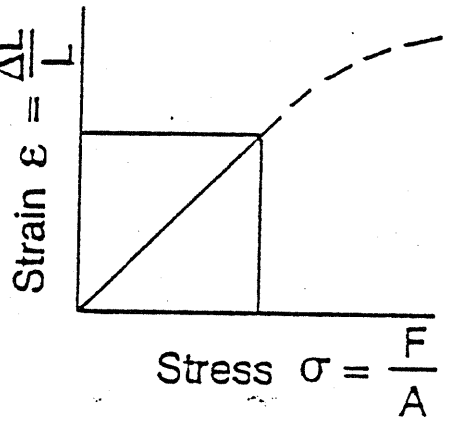
$$90^\circ = \frac{\pi}{2} \text{ Radians}$$

$$180^\circ = \pi \text{ Radians}$$

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Response of a Perfectly Elastic Material to a Constant Force

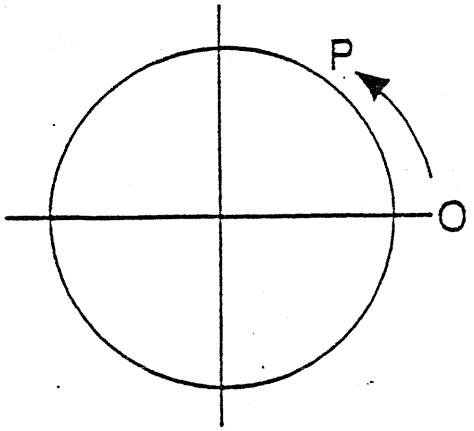




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Definitions

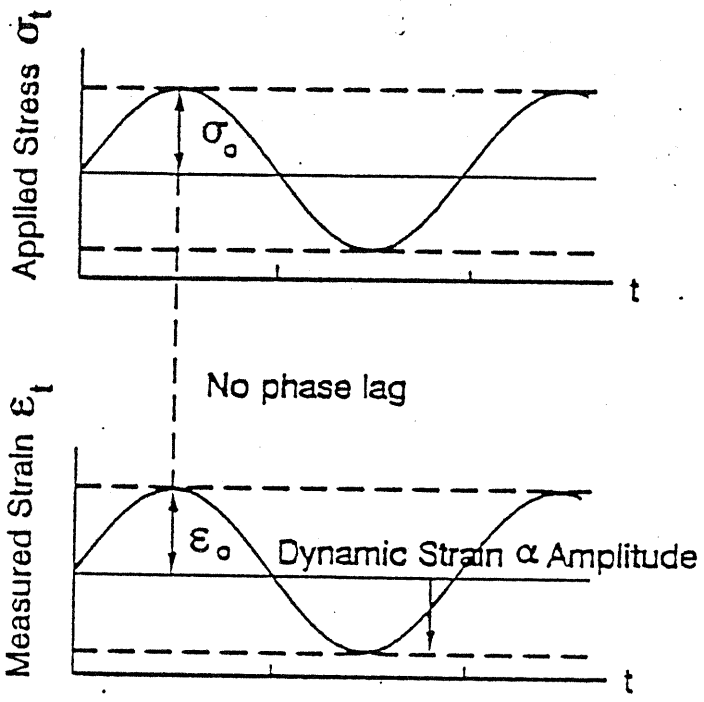
- A body which is in periodic motion transverses a series of positions according to a fixed pattern. The series of positions which it assumes is called the cycle.



- The time required to complete one cycle, i.e., assume all positions and velocities, is called the time period.
- Frequency (ω) is the number of cycles completed in a given time period.

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Response of a Perfectly Elastic Material to a Sinusoidal Stress of Frequency ω



$$\sigma_t = \sigma_o \sin \omega t$$

$$\varepsilon_t = \varepsilon_o \cos \omega t$$

Storage Modulus:

$$E' = \frac{\sigma_o}{\varepsilon_o} = \frac{\text{Dynamic Stress}}{\text{Dynamic Strain}}$$

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Definition

- Velocity is the ratio of displacement to time

$$V = \text{displacement} / \text{time}$$

examples: $V = \text{mile} / \text{hour}$

or

$V = \text{meters} / \text{second}$

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Definition

- Acceleration is the time rate of change of velocity

$$a = \frac{V - V_o}{t}$$

example: an automobile from rest can reach 60 miles/hr in 11 seconds

$$a = \frac{60 \text{ miles/hr}}{11 \text{ sec}}$$

$$a = \frac{88 \text{ ft/sec}}{11 \text{ sec}}$$

$$a = 8 \text{ ft/sec}^2$$

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Definition

- A Newton is that force which produces an acceleration of one meter per second per second in a mass of one kilogram

  1 Newton = 0.2248 Lbs. force

- A Pascal is the ratio of force to area measured in newtons per square meters

  $$Pa = {}^{N}/_{M^2}$$

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Definition

- Viscosity is the ability of a material to flow due to an applied stress.
- Viscous materials deform and do not return to their original shape.

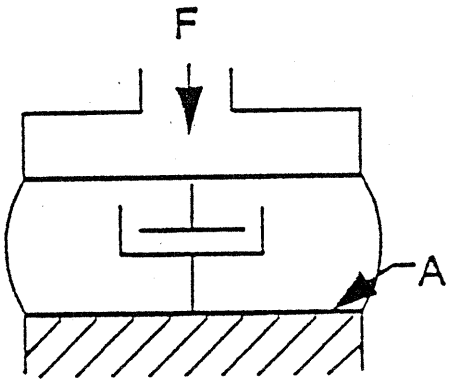
Viscosity is measured in terms of:

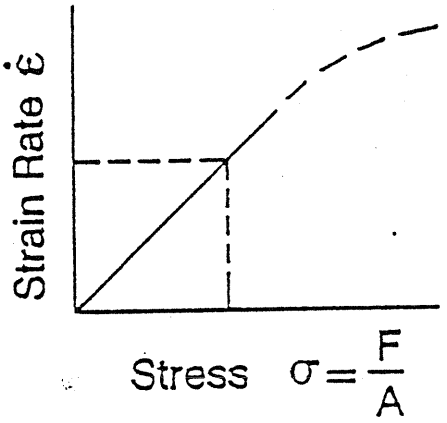
η = Newton - seconds / M^2

η = Pa · S

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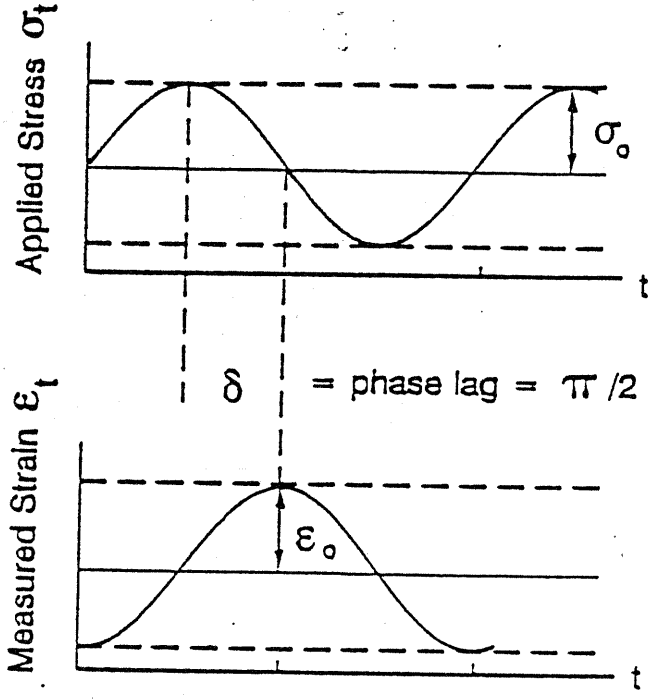
Response of a Perfect Newtonian Fluid to a Constant Force or Stress





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Response of a Newtonian Fluid to a Sinusoidal Stress of Frequency ω



$\sigma_t = \sigma_o \sin \omega t$

Strain and stress out of phase

$\varepsilon_t = \varepsilon_o \cos \omega t$

Loss Modulus:

$$E'' = \frac{\sigma_o}{\varepsilon_o}$$

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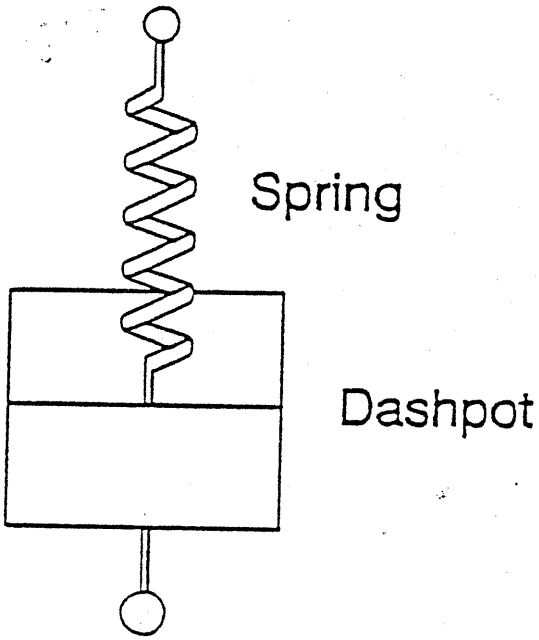
Definition

- Viscoelasticity is used to describe materials that exhibit both elastic and viscous properties

  i.e.: Thermoplastics between T_g and T_m
  or
  uncured/partially cured rubber

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Maxwell Element

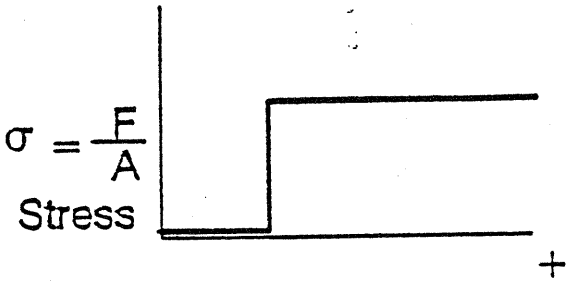


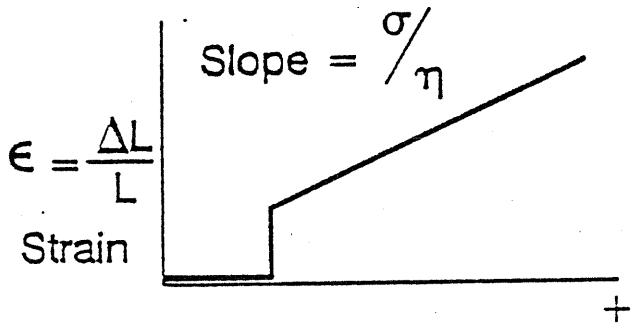
(Series)

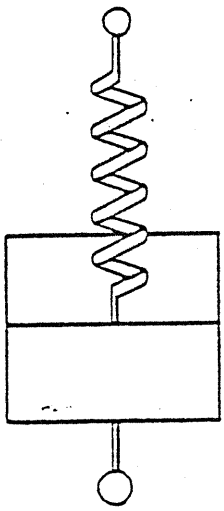
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Maxwell Element

Step function of stress and strain repsonse





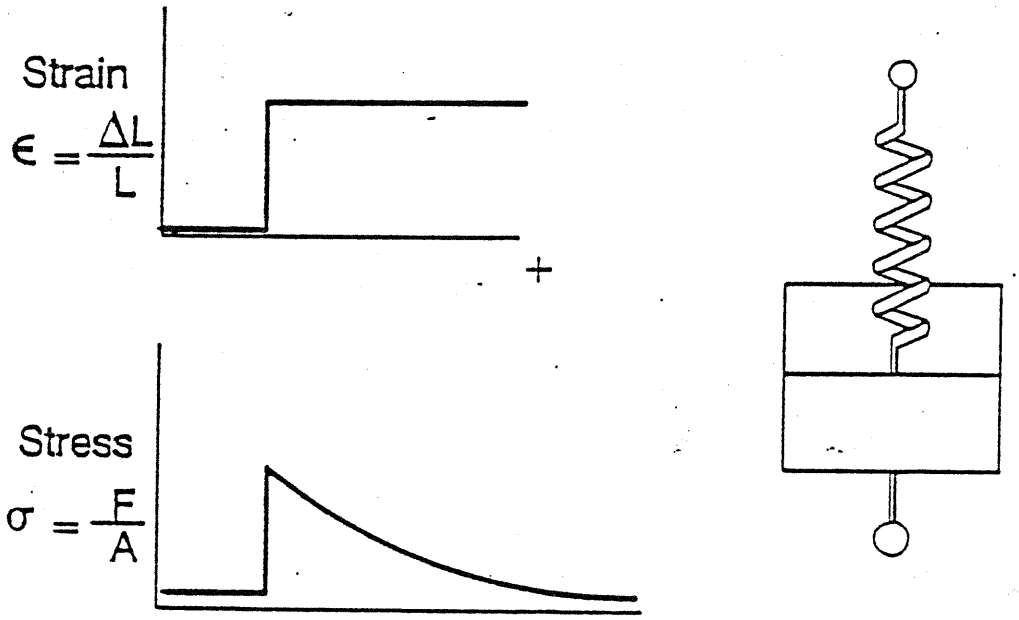


η = viscosity

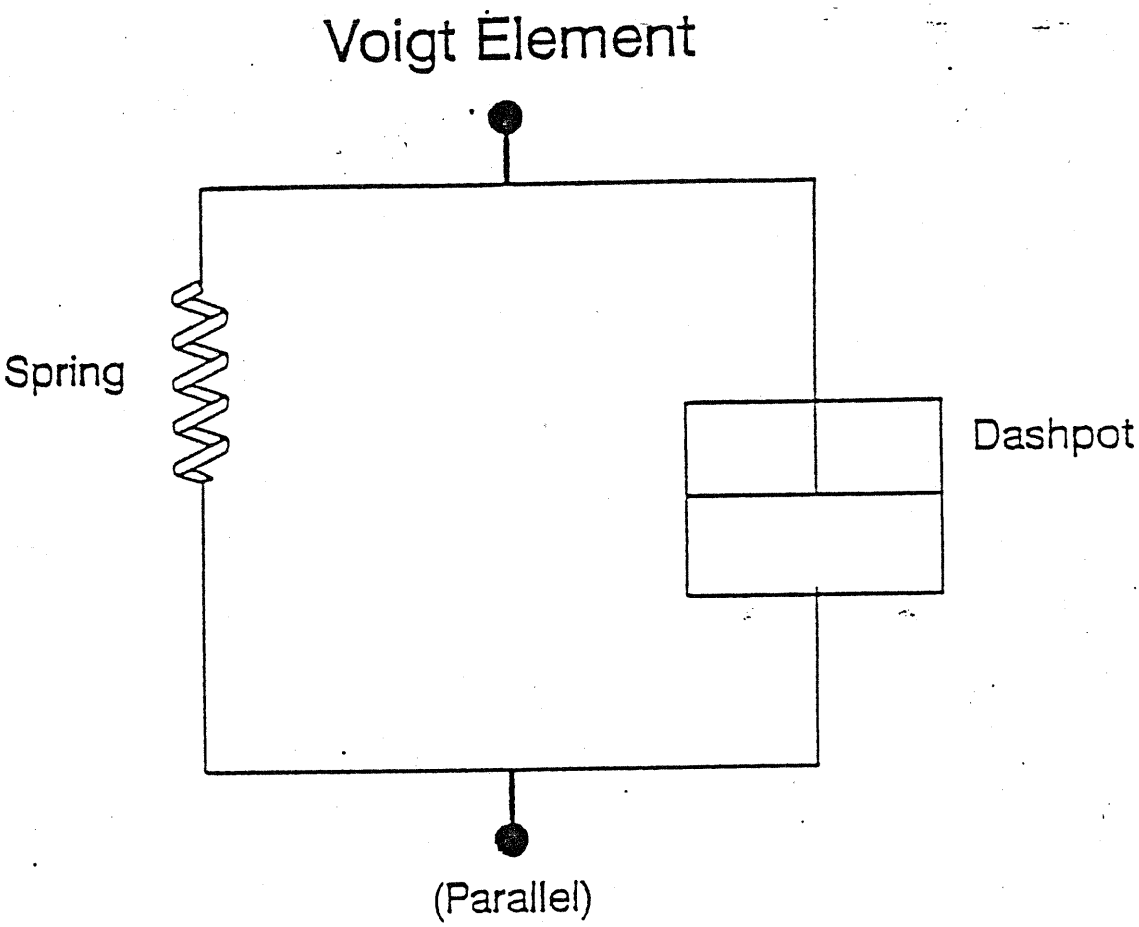
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Maxwell Element

Step Function of Strain and Stress Response



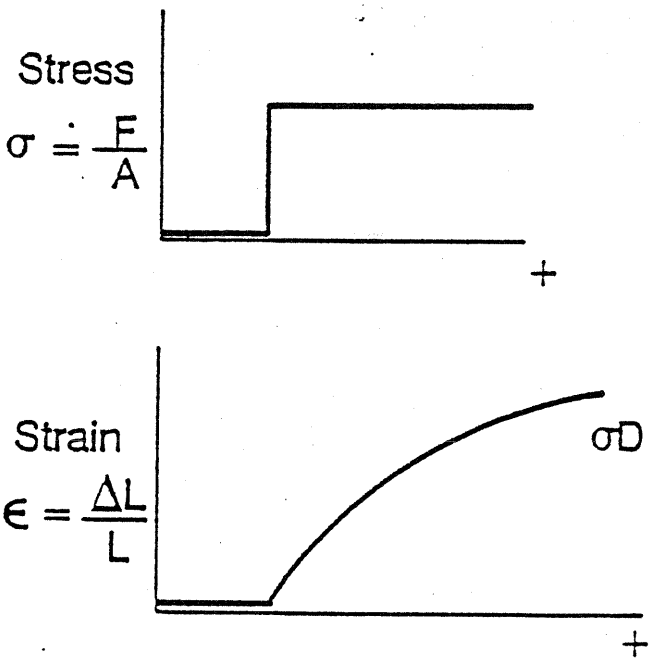
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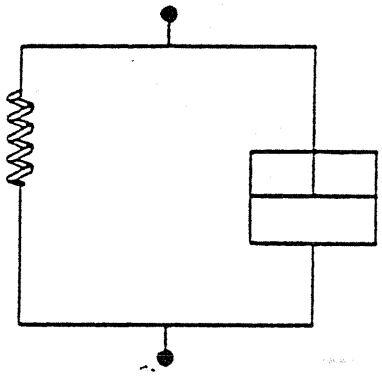


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Maxwell Element

Step Function of Stress and Strain Response

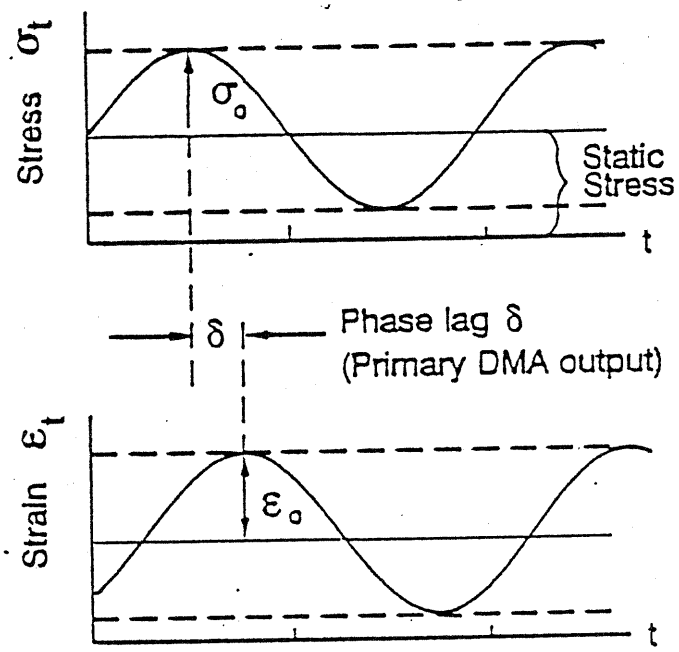




D = compliance of the spring

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Response of a Viscoelastic Material to a Sinusoidal Stress of Frequency ω



$$\sigma_t = \sigma_o \sin \omega t$$

$$\varepsilon_t = \varepsilon_o \sin(\omega t - \delta)$$

$\varepsilon_o \alpha$ Amplitude
(Primary DMA output)

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PERKIN ELMER

DMA 7e

Measuring Systems

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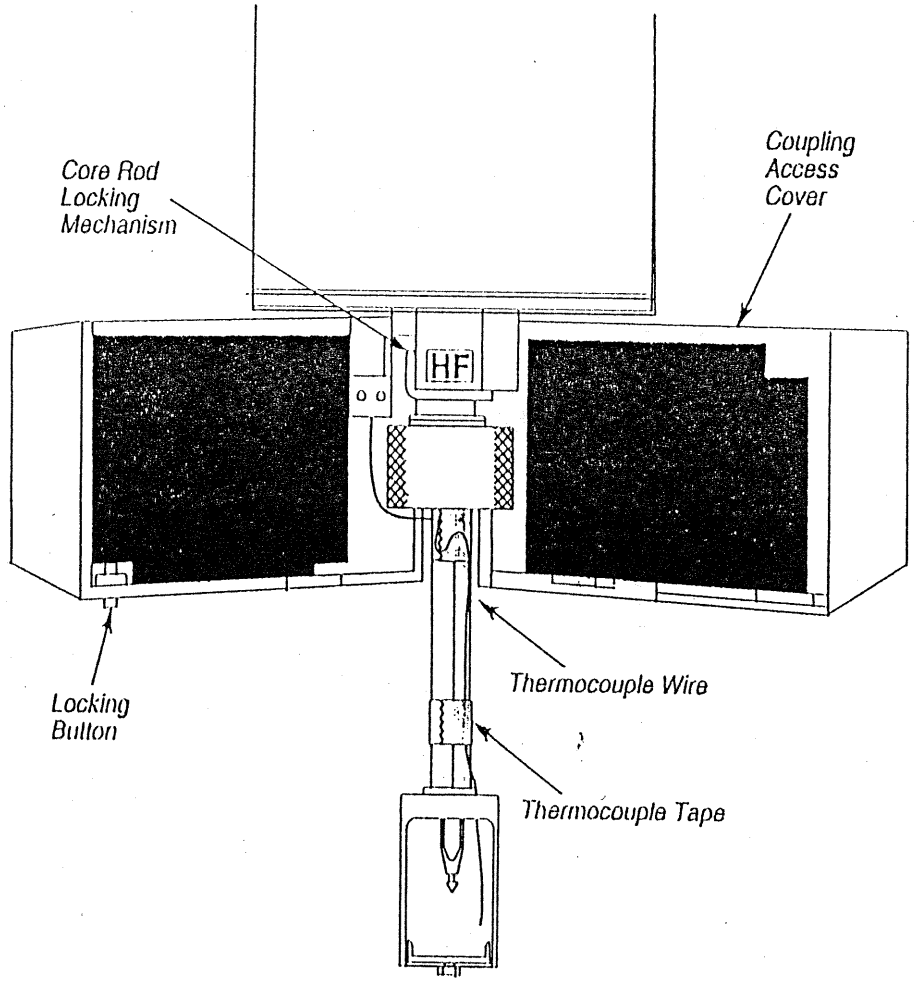
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DMA 7e
Measuring Systems

- 3-Point / 4-Point Bending
  - Stainless steel
  - Quartz
- Parallel Plate
  - Stainless steel
  - Quartz
- Cantilever
  - Stainless steel
- Extension
  - Stainless steel
  - Quartz
- Dilatometry
  - Quartz

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DMA 7e
Measuring System Installation



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DMA 7 Measuring Systems

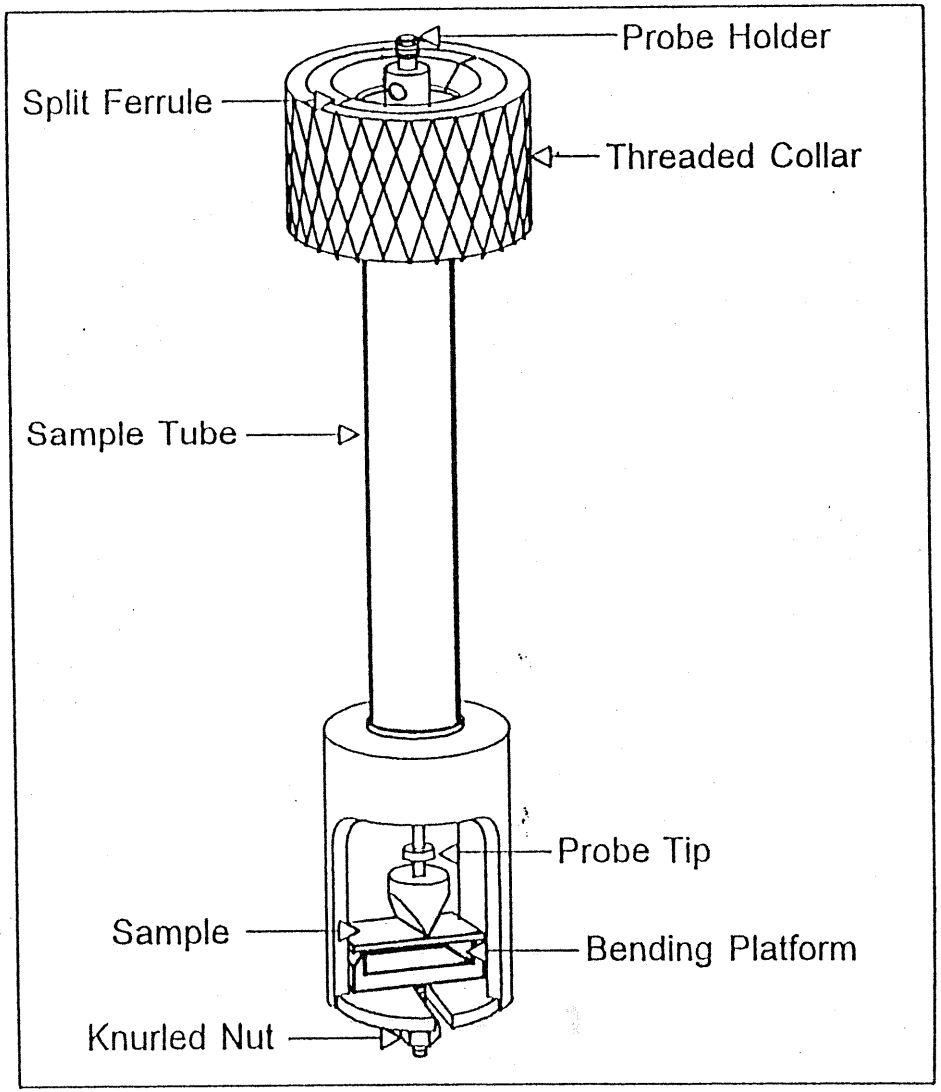
Stainless Steel & Quartz

Accomodate most sample types & geometries

Easy to install, clean & change

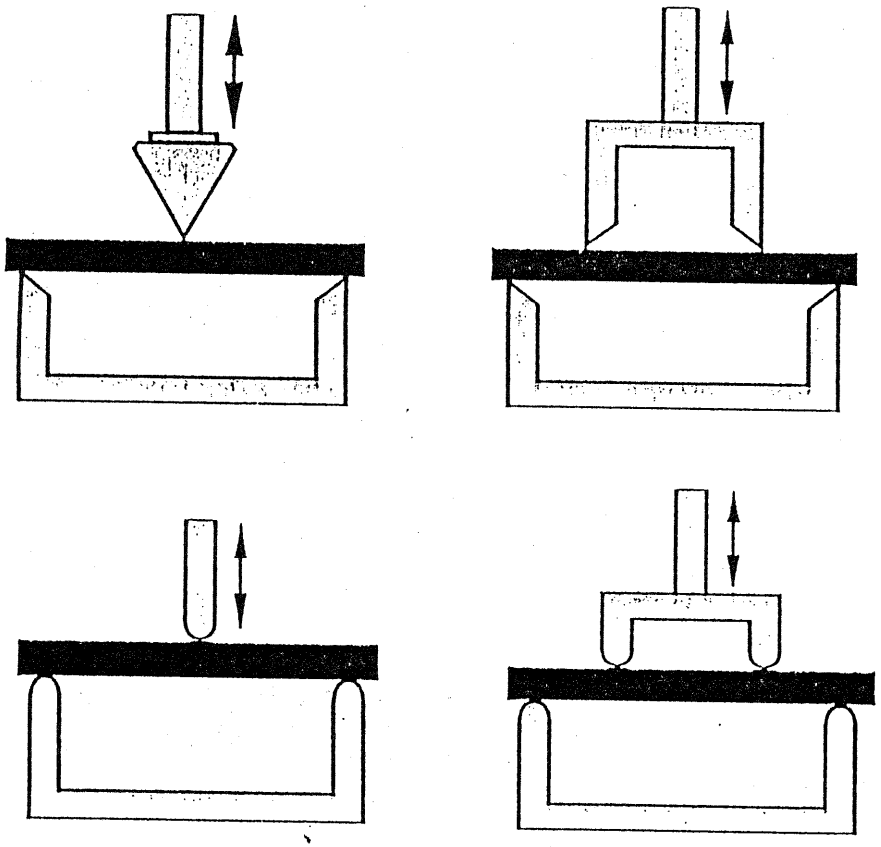
User customization

Rugged / long life



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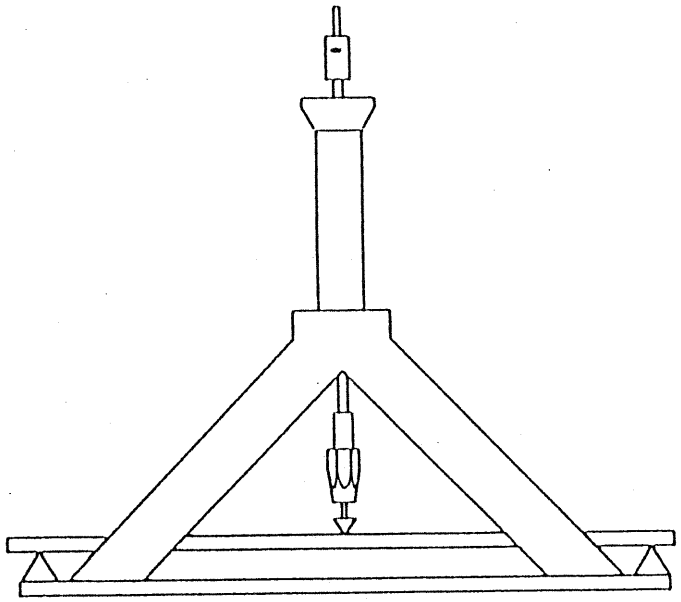
DMA 7e
3-Point Bending Measuring System



- Stainless steel & quartz
- Standard or rounded
- 3 & 4-Point Bending
- For high modulus solid materials (10^5 to 10^{12} Pa)
- Tips & base are easy to remove and install
- Handles samples from 5 to 20mm
- Can be customized

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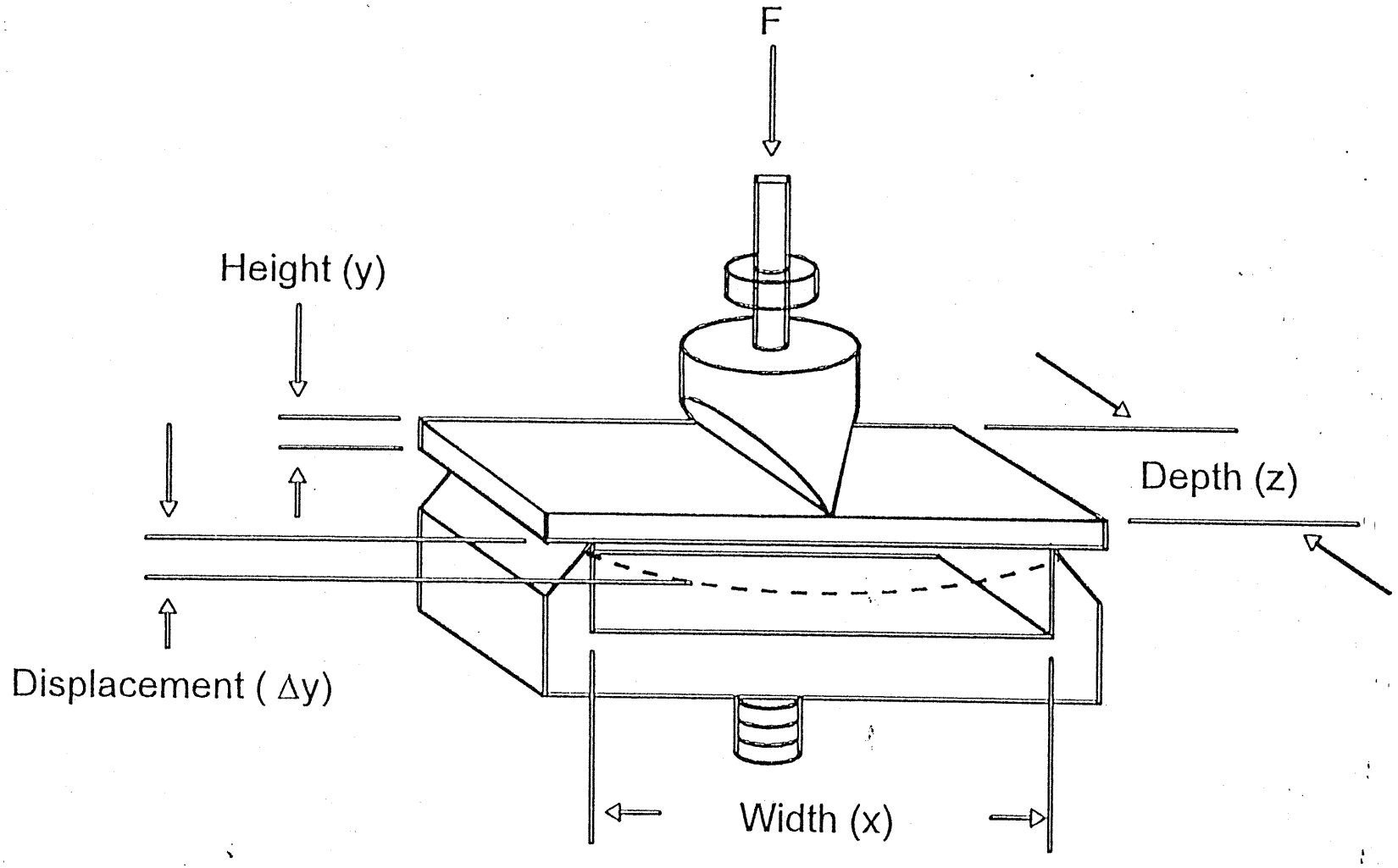
DMA 7e
Large 3-Point Bending Measuring System



- Stainless steel
- Standard or rounded
- 3 & 4-Point Bending
- For very high modulus solid materials (10^5 Pa and greater)
- Handles samples from 65 to 165mm (ambient)
- Compatible with ASTM Method D790
- Can be customized

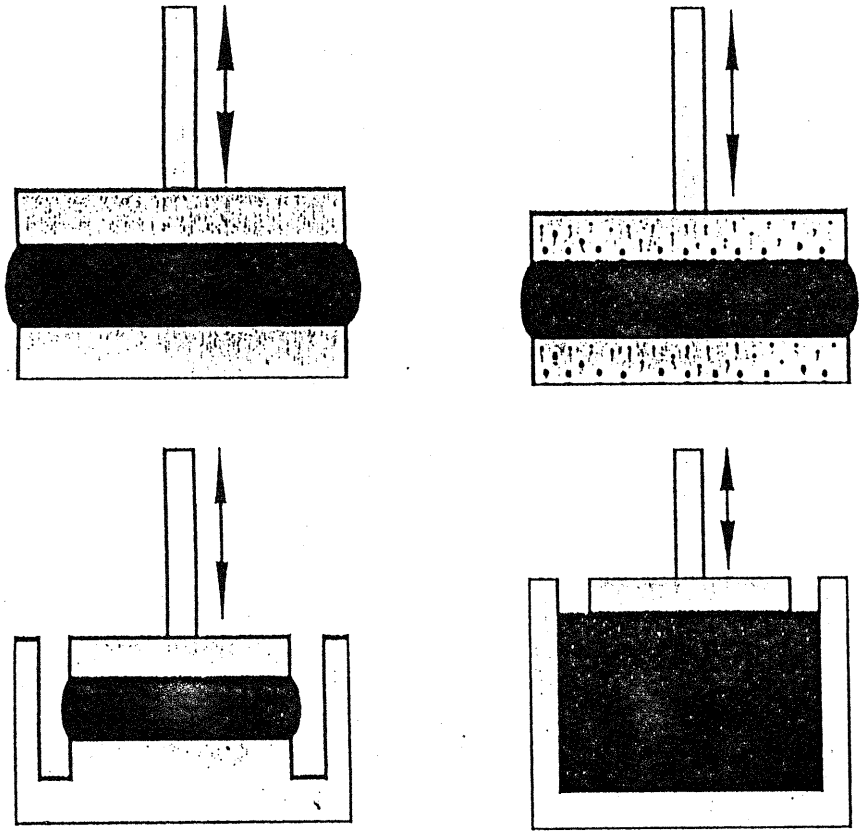
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Three-Point Bending Dimensions



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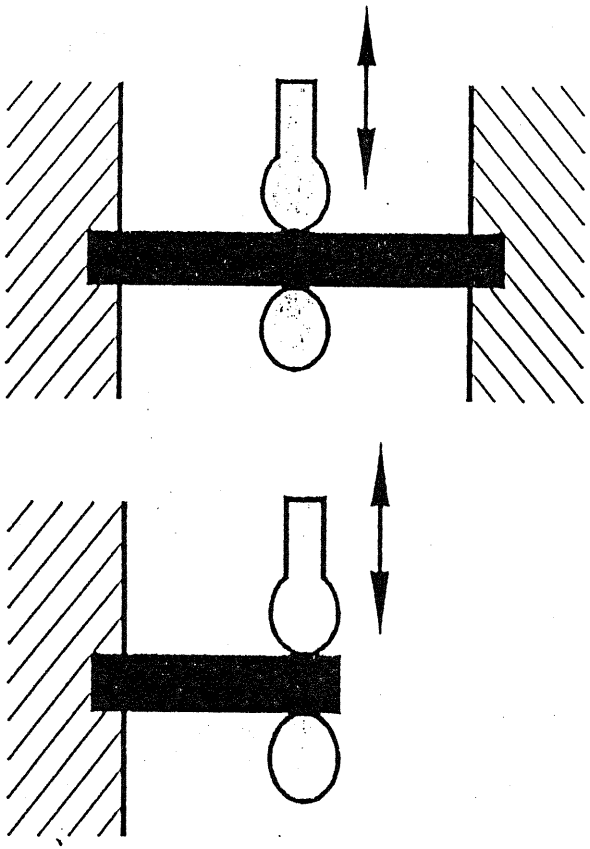
DMA 7e
Parallel Plate Measuring System



- Stainless steel & quartz
- Flat plates, sintered plates, plates with tray, cup and plate
- For semisolids or solids in modulus range (10^3 to 10^6 Pa)
- Tips & base are easy to remove and install
- Can be customized
- Sintered plates for samples with oil/additives
- Plate with tray prevents spillover into furnace
- Cup & plate for liquid samples

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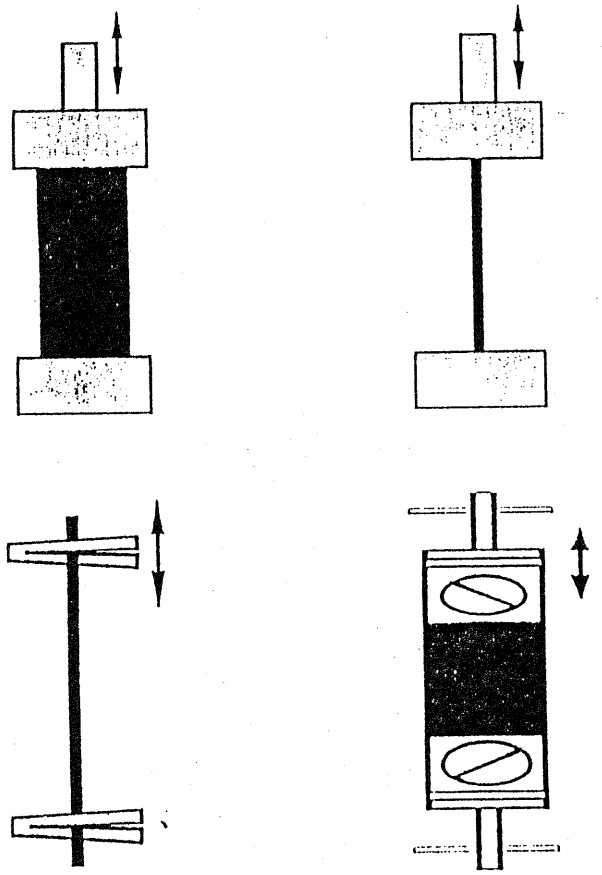
DMA 7e
Cantilever Measuring System



- Single and dual cantilever
- High and low modulus clamps
- Used for materials which soften and require support
- Modulus range:
  - Dual Cantilever 10^4 to 10^9 Pa
  - Single Cantilever 10^4 to 10^6 Pa

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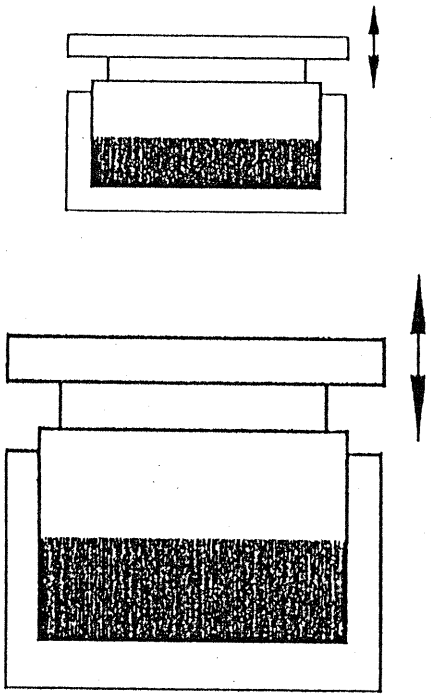
DMA 7e
Extension Measuring System



- Stainless steel & quartz
- Many different clamp types
- For film, fiber, hair, etc in modulus range:
  - Film 10^3 to 10^{10} Pa
  - Fiber 10^3 to 10^{12} Pa
- Samples can be loaded externally
- Can be customized

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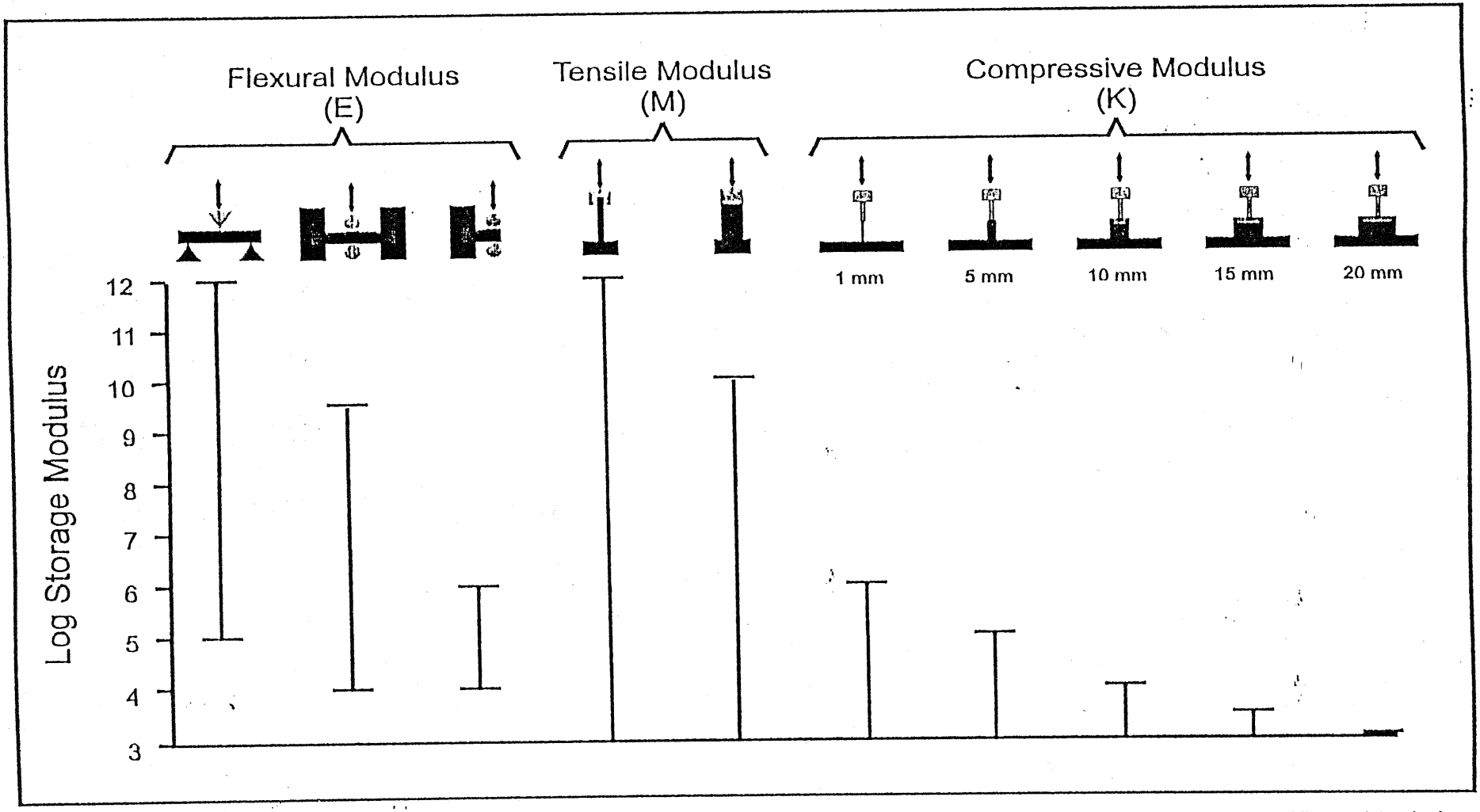
DMA 7e
Dilatometer Accessories



- Quartz
- Two different sizes available:
  - Small (0.1cc volume)
  - Large (1.0cc volume)
- Used to measure volumetric expansion / contraction
- Many sample types can be used (i.e. liquid, solid, etc)

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DMA 7e
Measuring System Range



B. Twombly, **PERKIN ELMER** *Thermal Analysis*

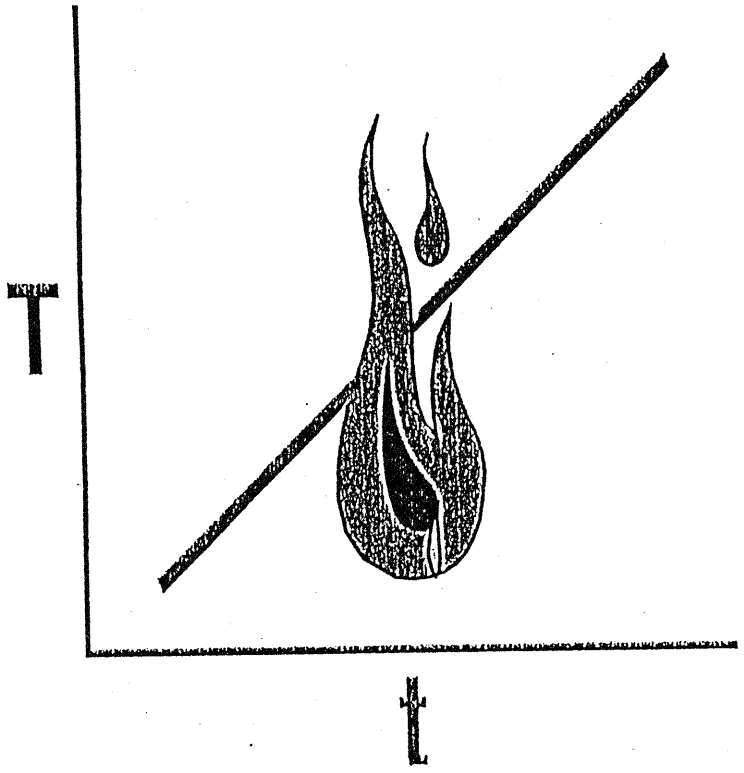
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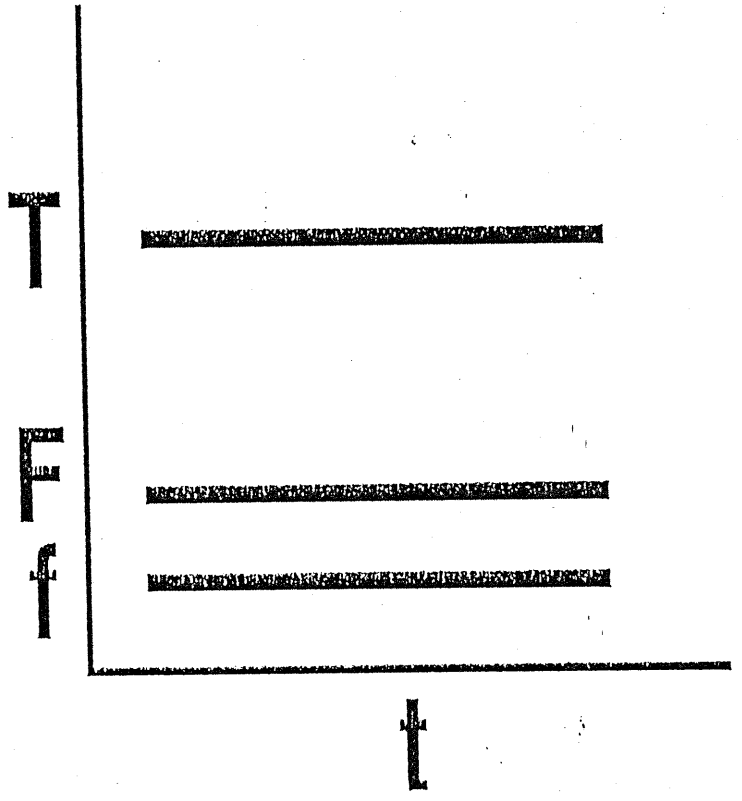
DMA 7e
Operating Modes

- Temperature/Time Scan
- Frequency Scan
- Dynamic Stress Scan
- Static Stress Scan (Creep Ramp)
- Creep-Recovery
- Constant Force (TMA)
- Constant Position (Constant Length)

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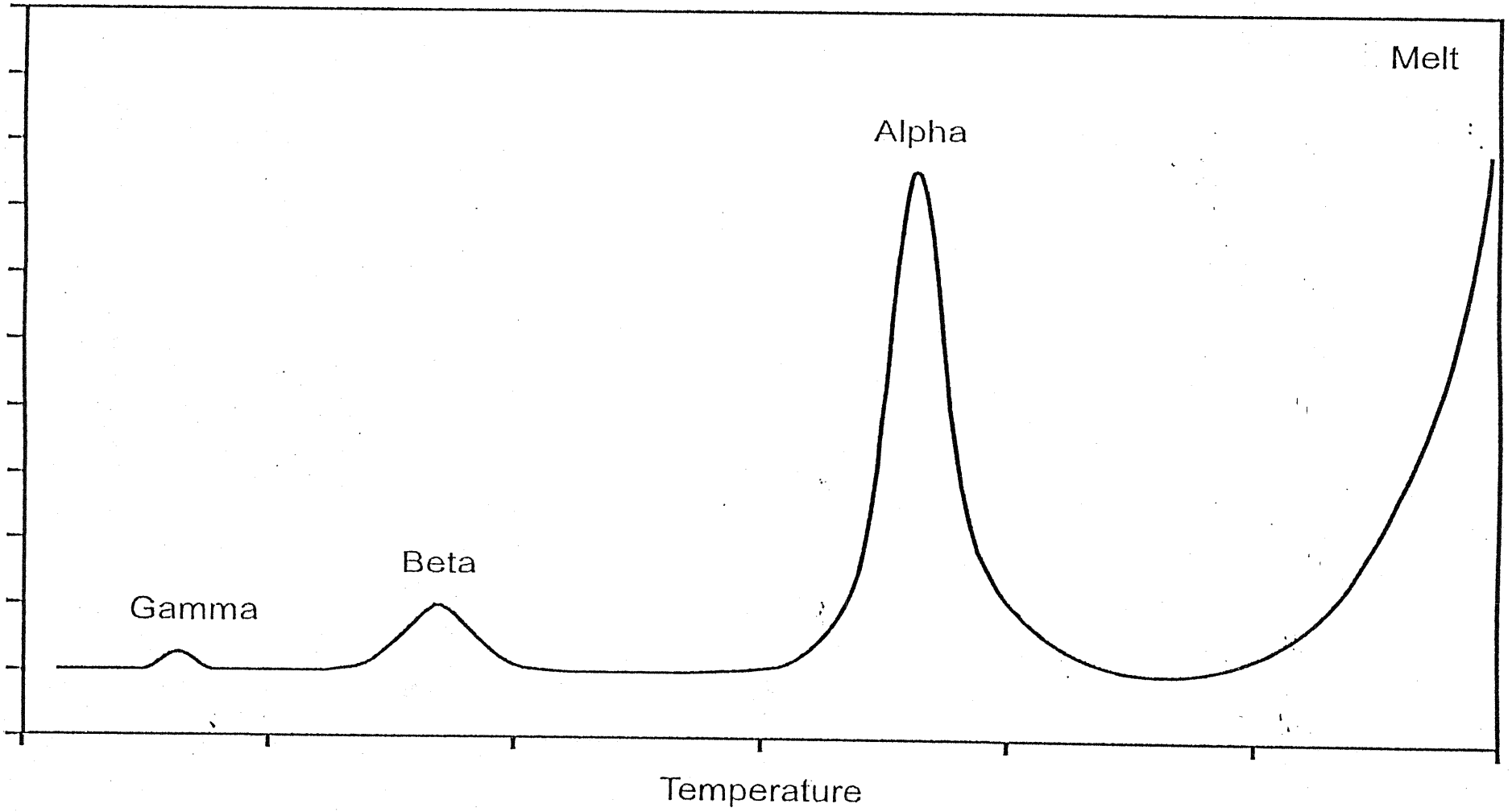
DMA 7e
Temperature/Time Operating Mode





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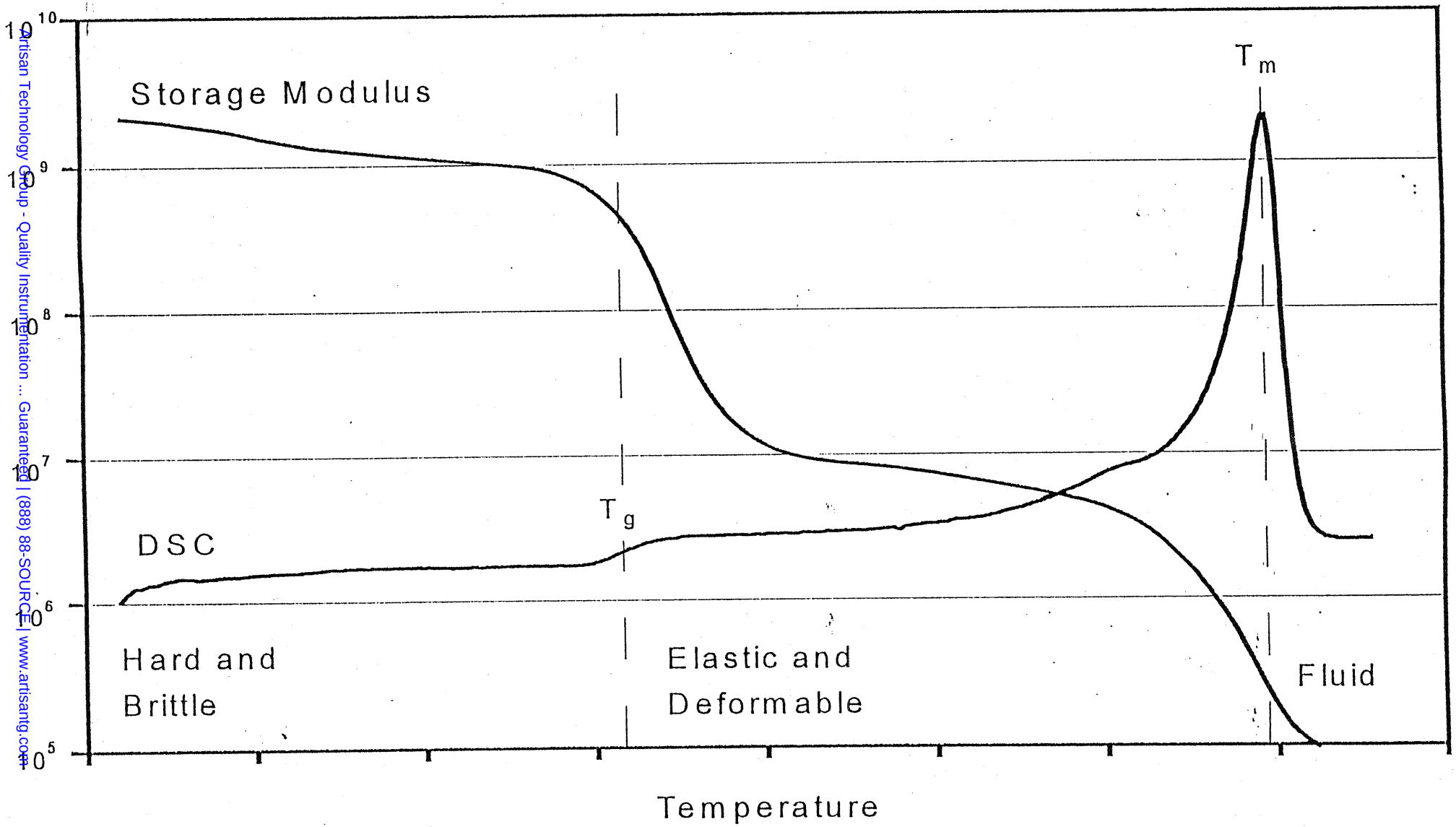
DMA 7e
Tangent Delta



B. Twombly, Perkin-Elmer

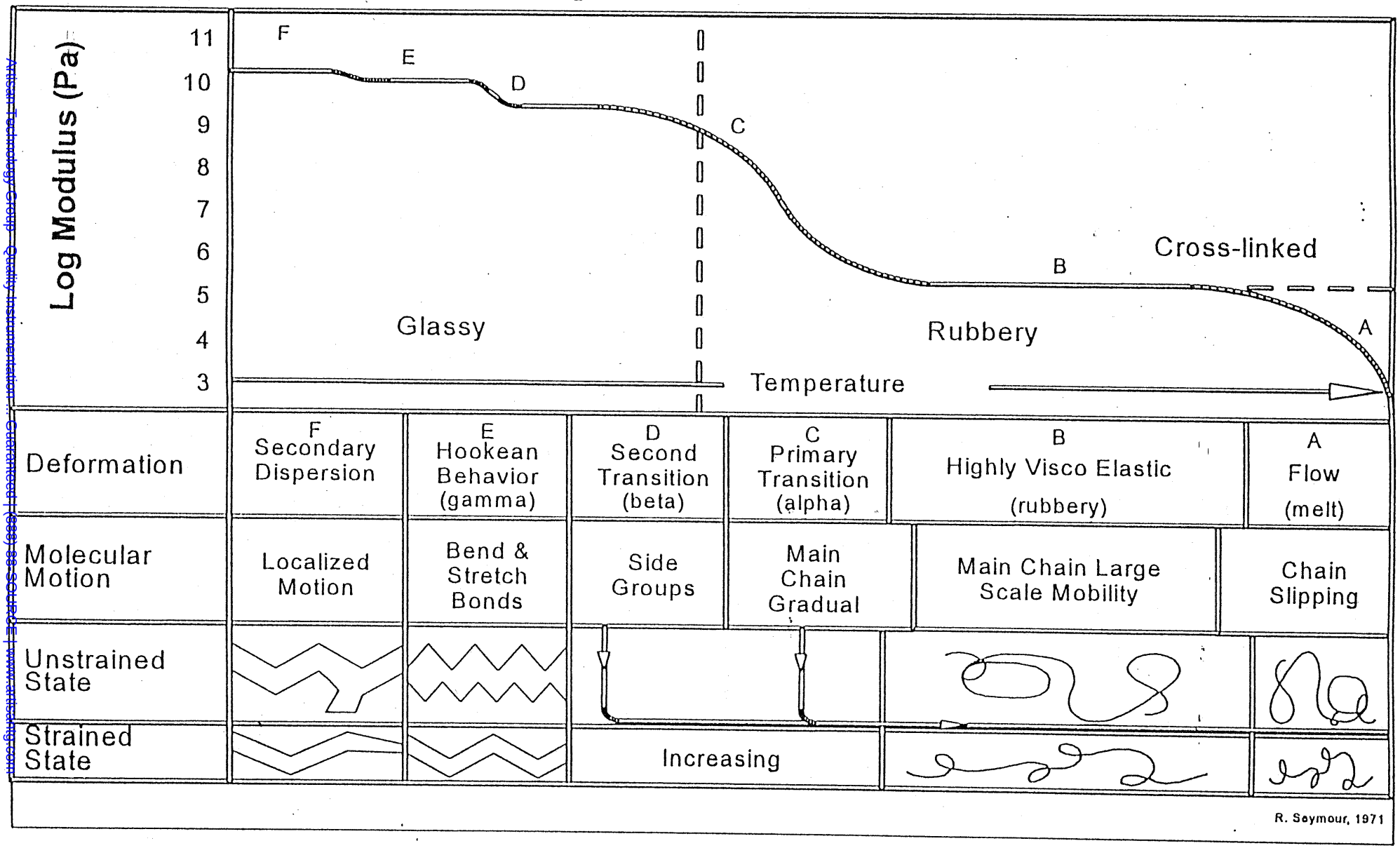
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DMA 7e
Storage Modulus



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DMA 7e
Temperature Scan

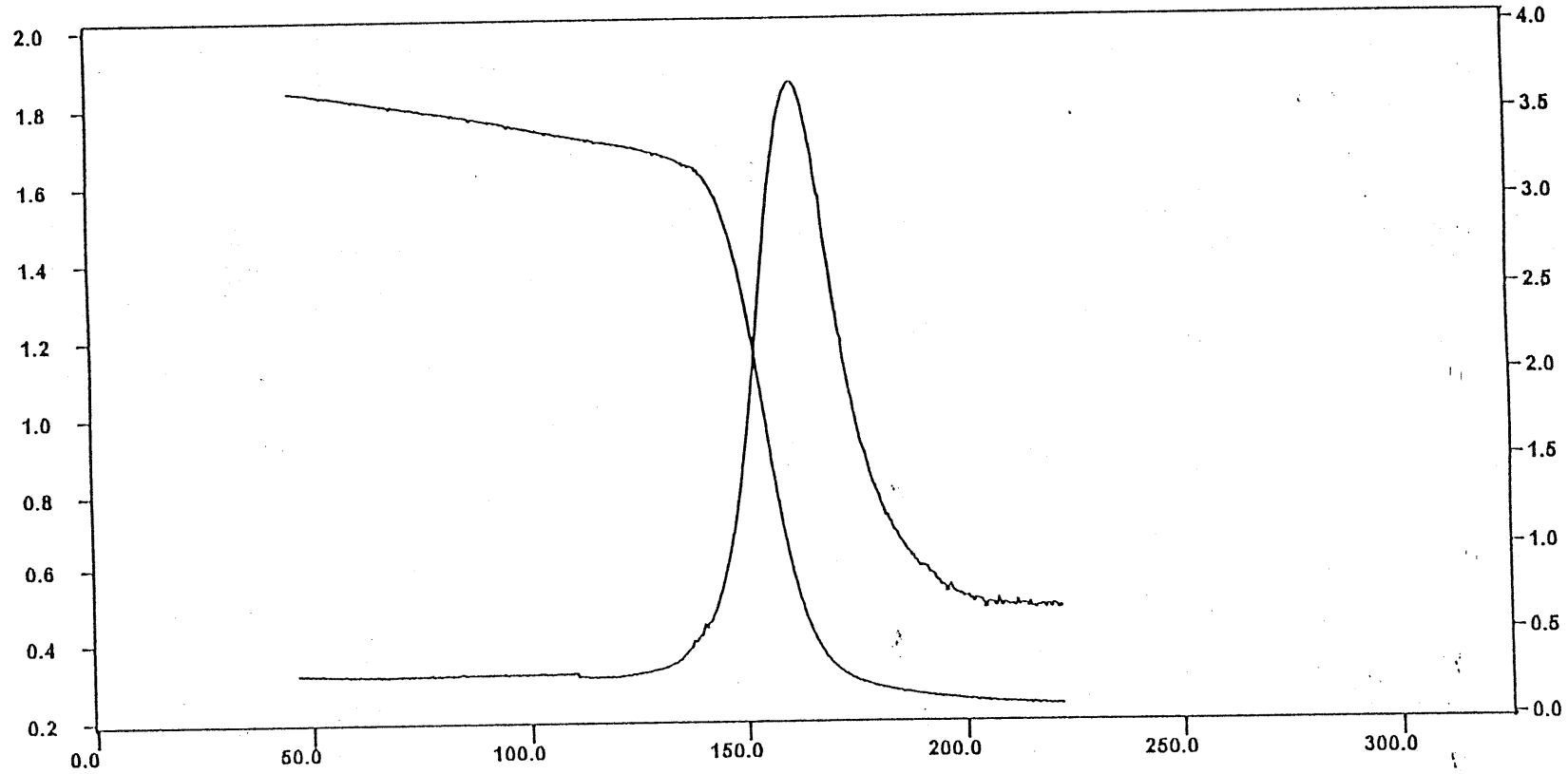


DMA 7e
Temperature Scan Mode

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Curve 1: DMA Temp/Time Scan In 3 Point Bending
File Info: temp5 Tue Feb 13 19:27:31 1990
Frequency: 10.00 Hz Strain: 0.050%
GLASS-EPOXY COMPOSITE Tension: 120.000%

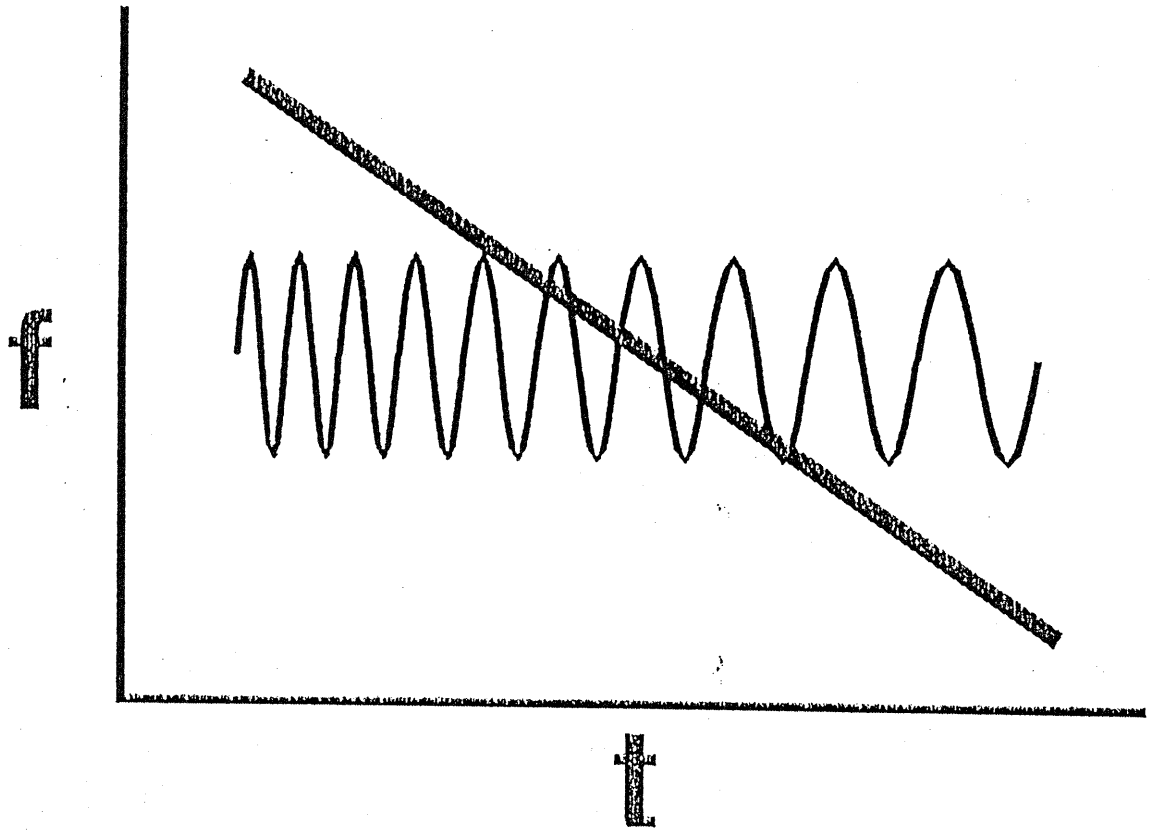
GLASS-EPOXY COMPOSITE



TEMP1: 40.0 C
TEMP2: 226.0 C
TIME1: 0.0 min
RATE1: 2.0 C/min

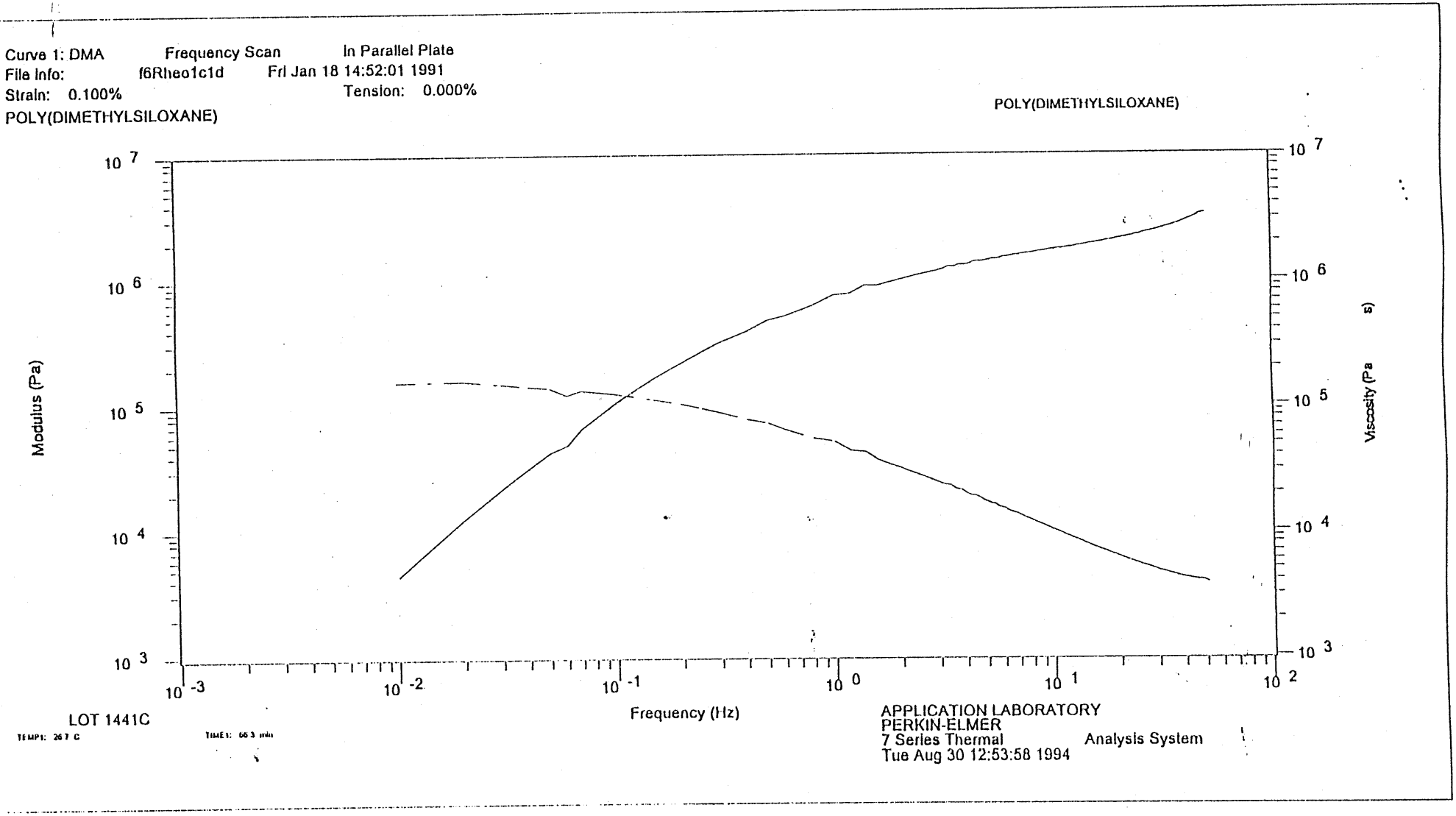
APPLICATION LABORAORY
PERKIN-ELMER
7 Series Thermal Analysis System
Tue Aug 30 12:49:11 1994

DMA 7e
Frequency Scan Mode



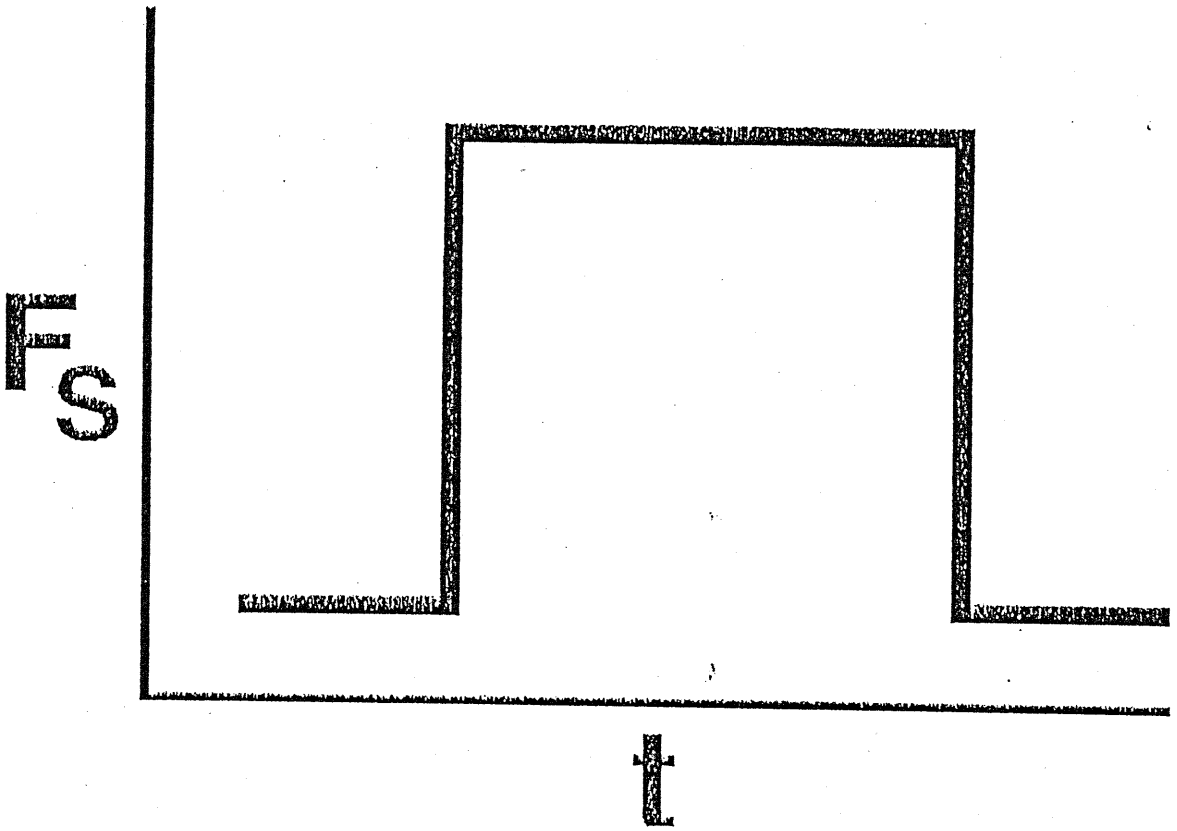
Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

DMA 7e
Frequency Scan Mode



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DMA 7e
Creep-Recovery Mode

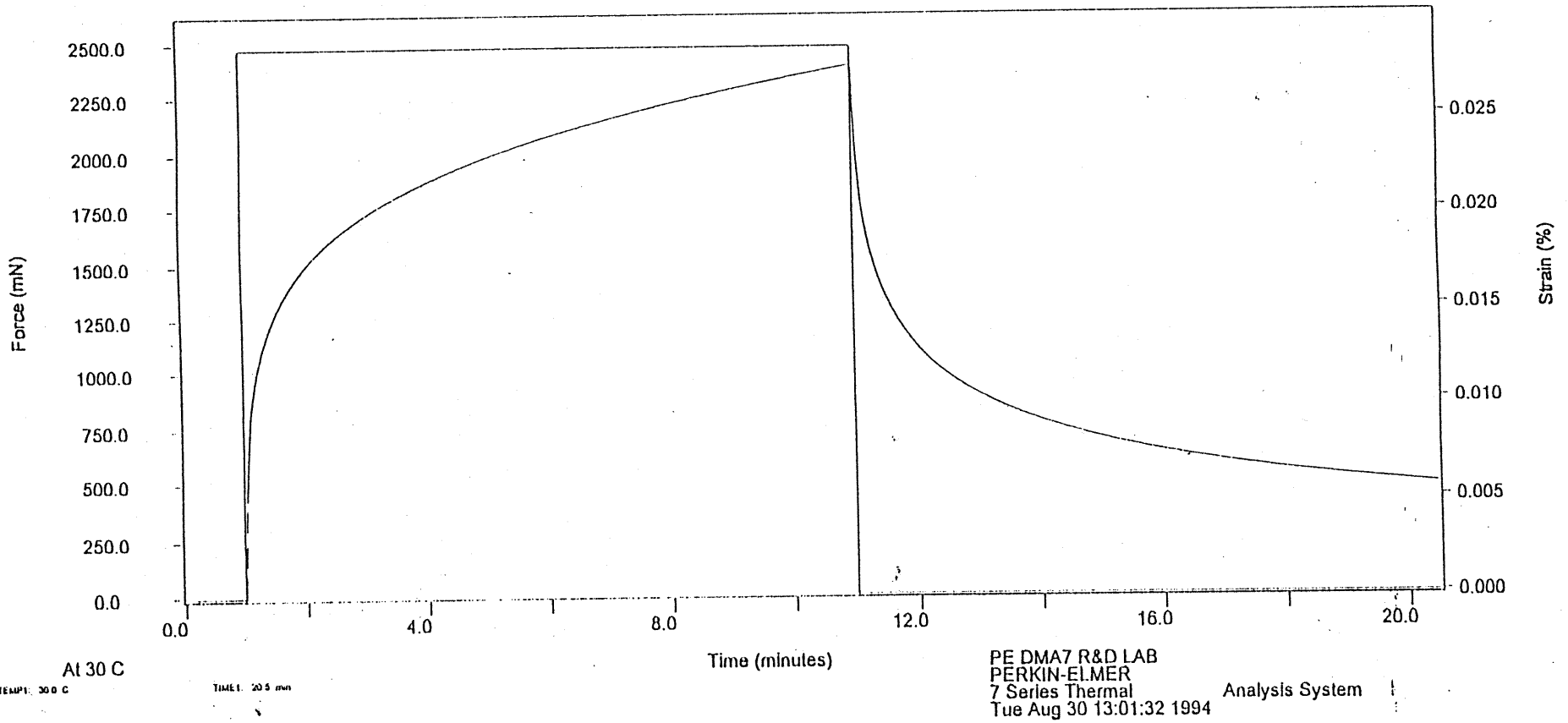


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DMA 7e
Creep Recovery

Curve 1: DMA Creep Recovery in 3 Point Bending
File info: creppvc1 Mon Jul 11 23:54:56 1988
Sample Height: 3.061 mm Creep Stress: 2.00e+06Pa
POLY(VINYL CHLORIDE)

Recovery Stress: 1.00e+01Pa
POLY(VINYL CHLORIDE)

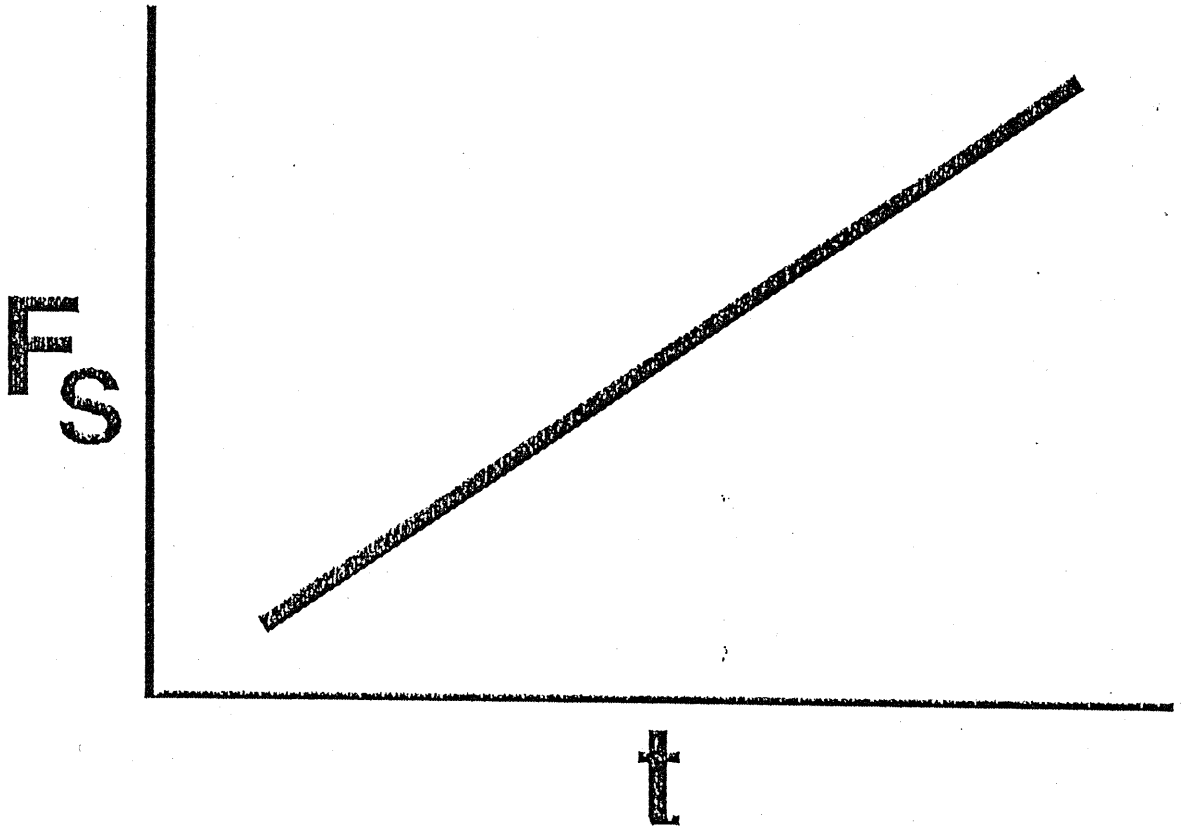


At 30 C
TEMP1: 30.0 C
TIME1: 20.5 mn

PE DMA7 R&D LAB
PERKIN-ELMER
7 Series Thermal Analysis System
Tue Aug 30 13:01:32 1994

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DMA 7e
Creep Ramp Mode

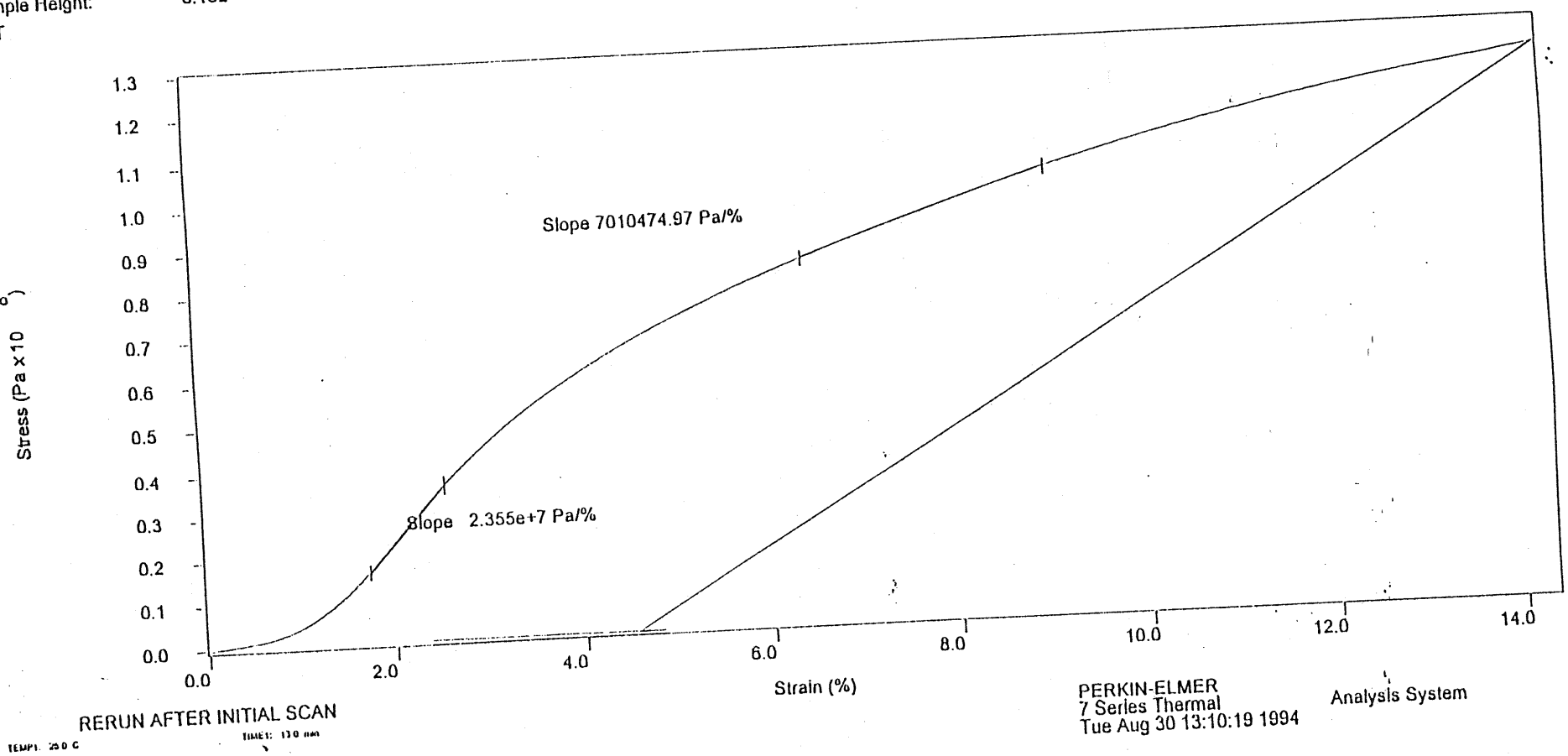


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DMA 7e
Creep Ramp

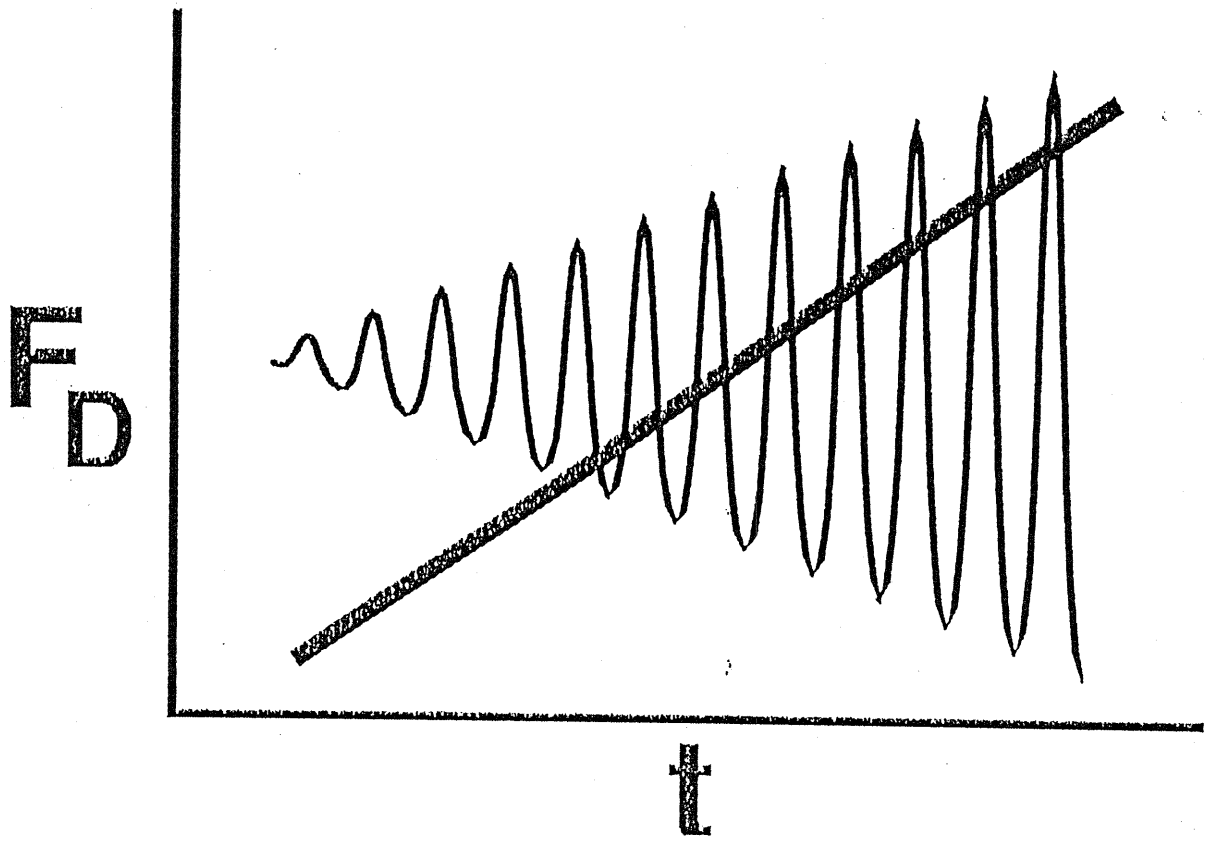
Curve 1: DMA Creep Recovery in Extension
File Info: PET002 Thu Mar 10 13:30:57 1994
Sample Height: 6.162 mm Creep Stress: 6428.0mN
PET

Recovery Stress: 1.0mN



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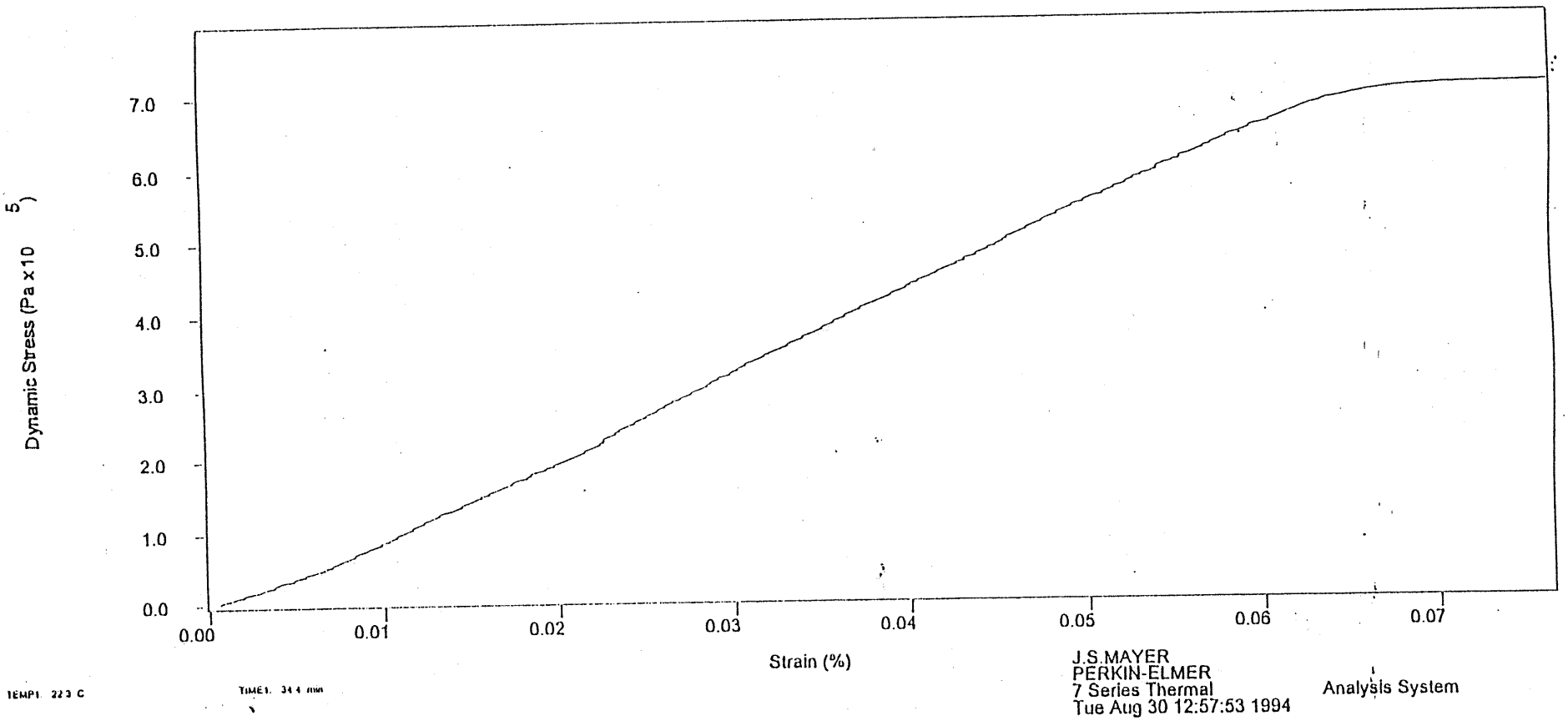
DMA 7e
Dynamic Stress Scan Mode



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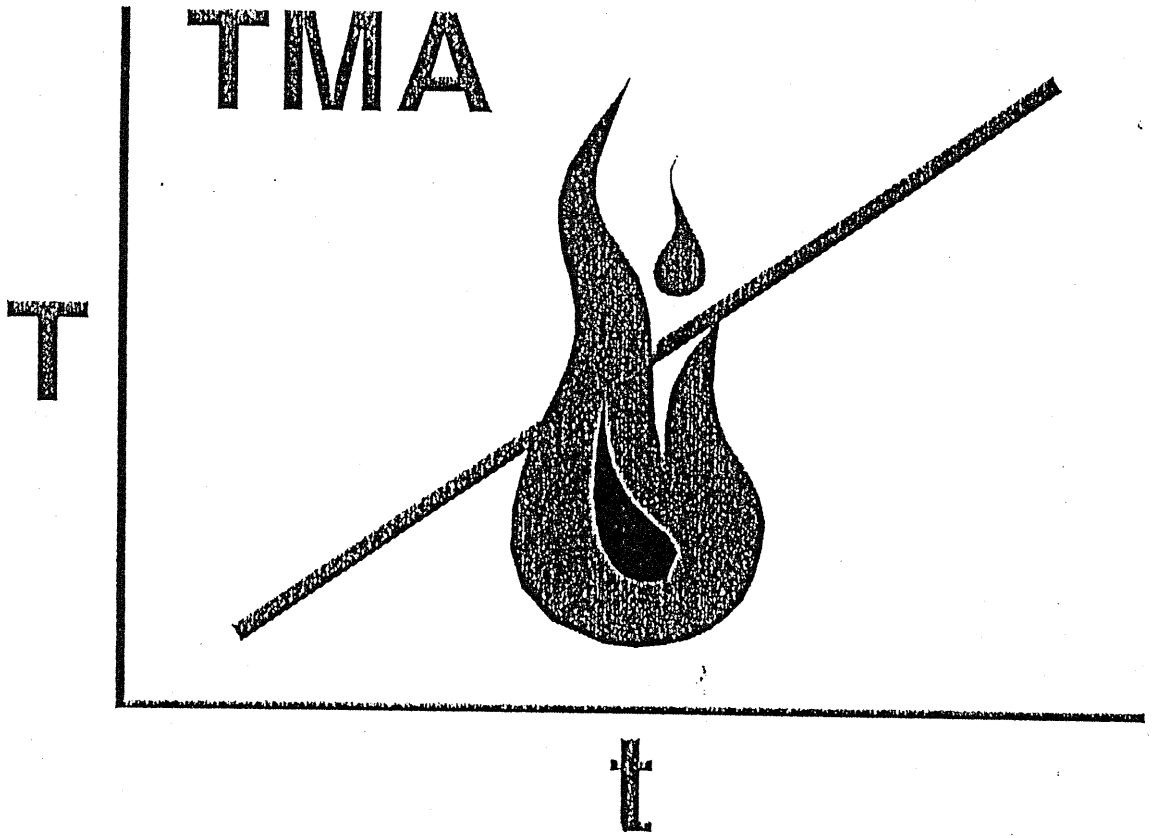
DMA 7e
Stress Scan

Curve 1: DMA AC Stress Scan In 3 Point Bending
File Info: sscramg2 Sat Sep 16 19:38:40 1989
Frequency: 1.00 Hz Stress Rate: 10.0mN/min
CERAMIC STRESS SCAN SLO Tension: 110.000%



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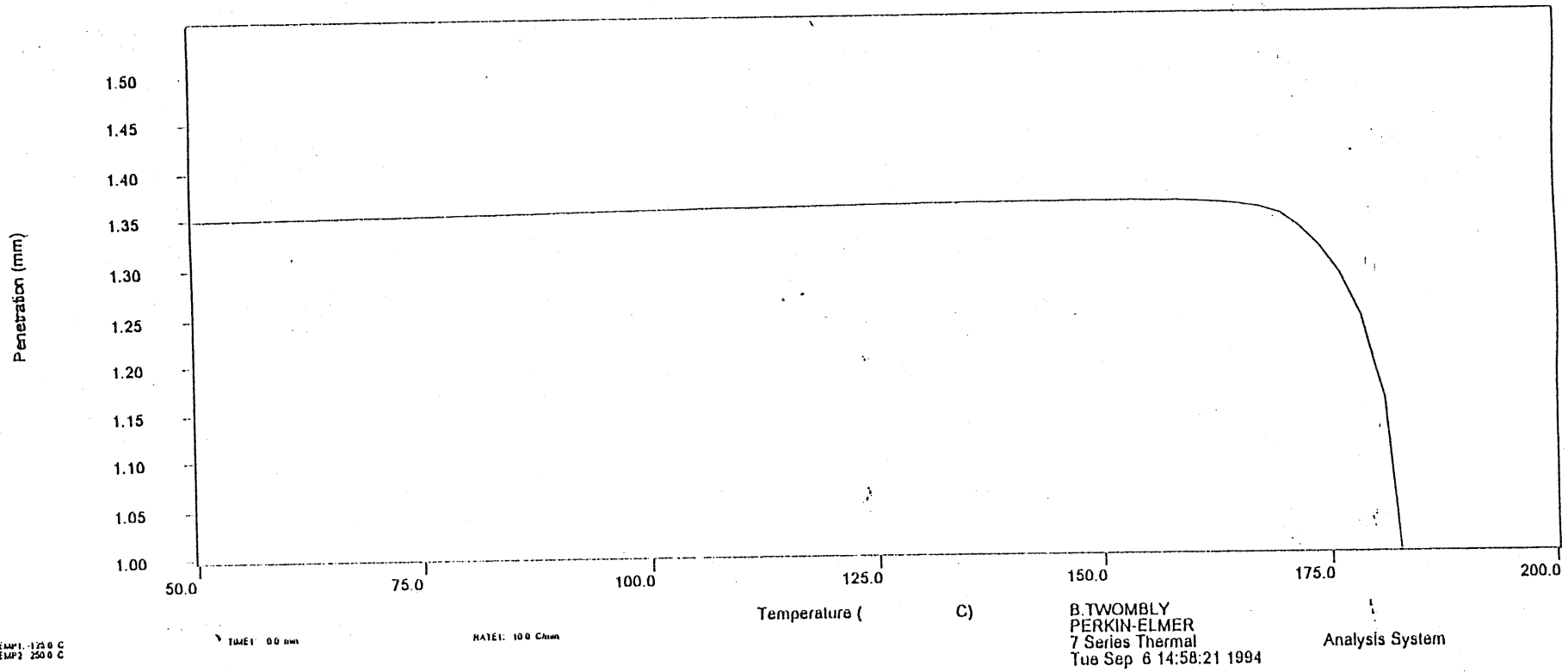
DMA 7e
Constant Force (TMA) Mode



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DMA 7e
Constant Force (TMA)

Curve 1: TMA In Penetration
File Info: T5exx1 Wed Oct 4 16:18:08 1989
Sample Height: 1.354 mm
POLYPROPYLENE BLEND



TEMP1: 125.0 C
TEMP2 250.0 C
TIME1 0.0 min
RATE1: 10.0 C/min

B.TWOMBLY
PERKIN-ELMER
7 Series Thermal
Tue Sep 6 14:58:21 1994
Analysis System

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DMA 7e
Motor Controls

- **Force Control** — Maintains a constant force on the sample
- **Stress Control** — Maintains a constant stress on the sample
- **Position Control** — Maintains a constant sample position
- **Tension Control** — Maintains a constant relationship between static and dynamic force
- **Amplitude Control** — Maintains a constant sample amplitude
- **Strain Control** — Maintains a constant sample strain

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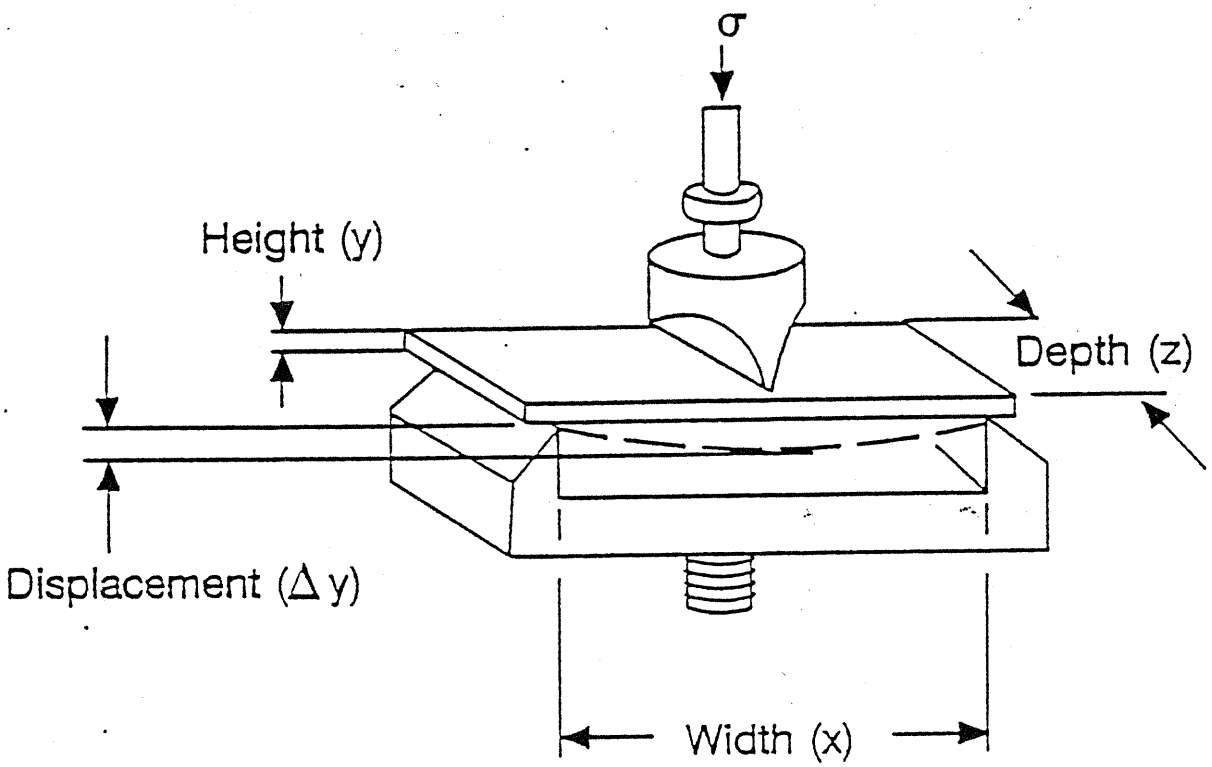
DMA 7e
Software

- Direct instrument control
- Single or multiramp programs
- Automatic calibration routines (using traceable standards)
- Real time data display
- Automatic balance tare and read weight
- MACRO programming
- ASCII or HPGL File Capture
- Instrument *Status Lines*
- Numerous calculation programs
- Curve optimization
- Curve comparison
- Subtraction and multiple derivative curves
- Convenient archive routines
- Advanced software programs

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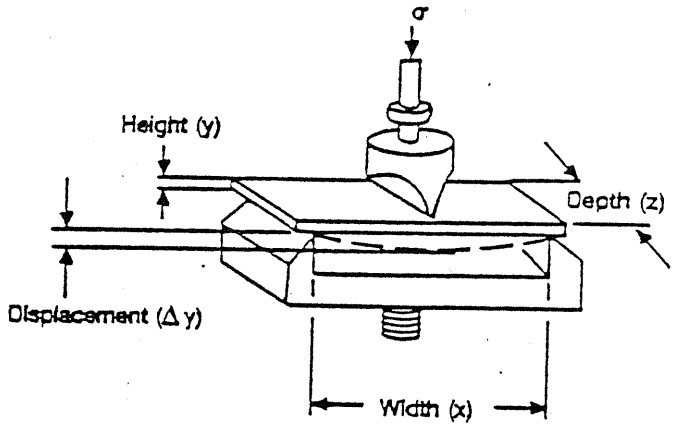
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Three-Point Bending Dimensions



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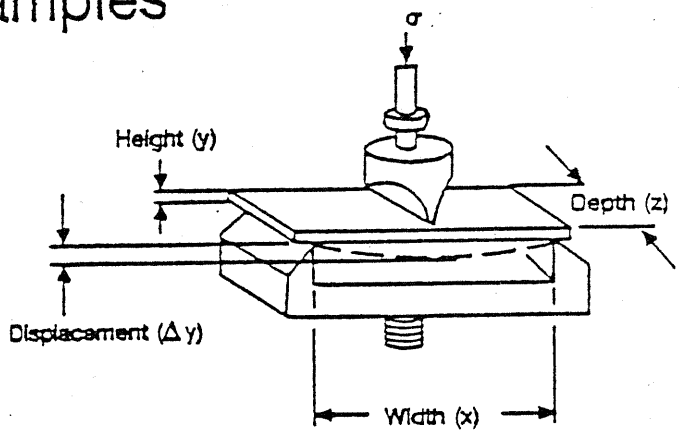
Three-Point Bending Samples



- Sample loading/aligning
  - Knife edges parallel to probe
  - Sample perpendicular to platform
- Bending platform size
  - 15 to 20 mm for high modulus ($> 10^8$ Pa)
  - 5 to 10 mm for low modulus (10^6 to 10^8 Pa)

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Three-Point Bending Samples

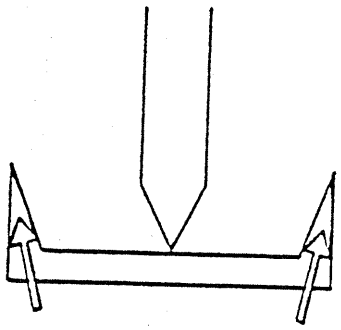


- Sample size
  - Typically 2 X 2.5 X 22 mm
  - Larger for soft materials
- Static and dynamic stress
  - Static stress should be 10 to 25% greater than the dynamic stress (i.e., Autotension value of 110 to 125)
  - Dynamic stress should typically give a strain of 0.005 to 1.5%

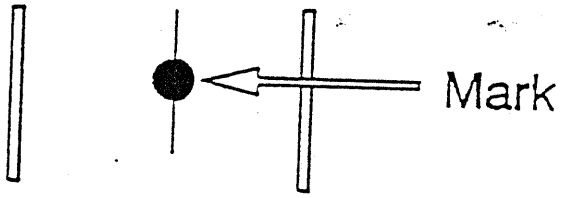
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Three-Point Bending
Sample Mounting

Adjust position of knife edges to center probe at mark



Knife Edges

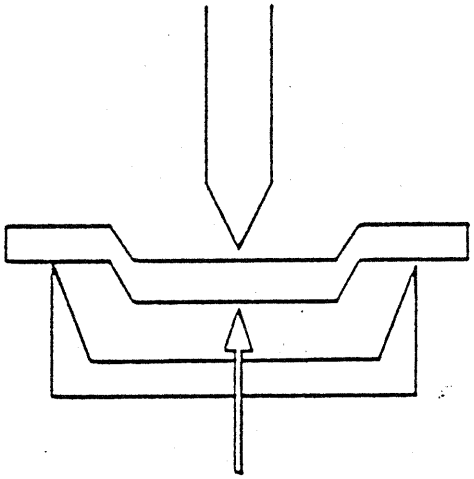


Knife edge parallel with probe edge

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Three-Point Bending
Sample Mounting

Place zero block between knife edges press read zero key

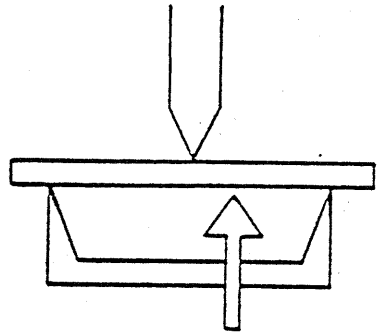


Zero block

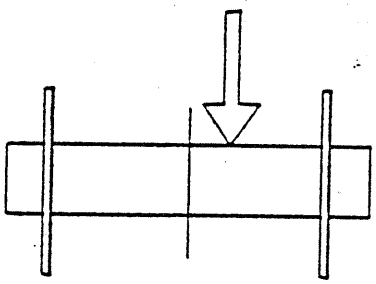
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Three-Point Bending
Sample Mounting

Replace zero block with sample as below read sample height



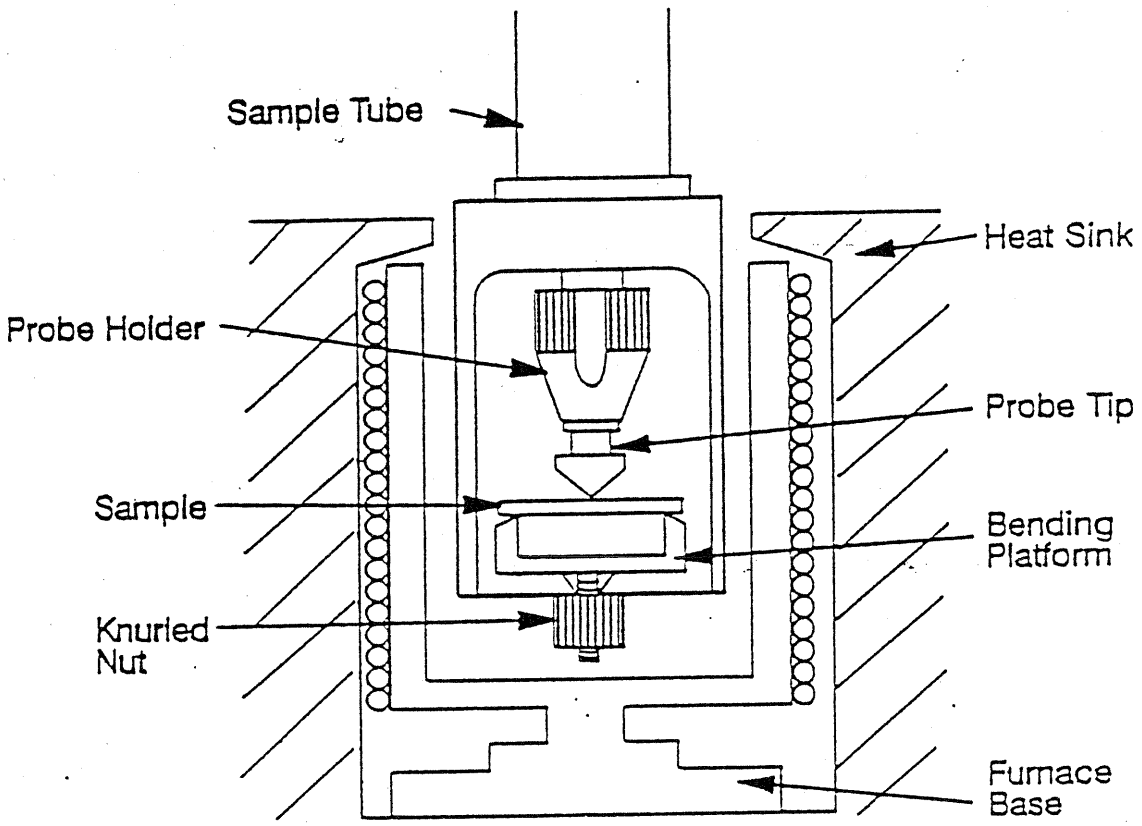
Sample



Sample perpendicular with knife edges

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Three-Point Bending



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DMA LABORATORY
Experiment #2

3 Point Bending
Temp/Time scan
PE REFERENCE E

OBJECTIVE: This laboratory experiment will allow the student to become more familiar with:

- Set-up for making a run
- Sample handling
- Making a run
- Data handling/manipulation

DIRECTIONS: Read the entire question or instruction before responding.

1. What coolant is being used? _____

2. What Purge Gas is being used? _____

3. What Purge Rate is being used? _____ cc/min

4. Why are you using that purge rate?

5. Roughly measure the knife edge spacing of 3 point bending bottom fixture. Circle that dimension below.

   5mm 10mm 15mm 20mm

6. Measure the PE REFERENCE E Sample

Sample depth = _____

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7. Install the appropriate probe tip.

8. Install the base (15mm plate knife edge spacing) and align to upper blade to the base so that the knife edges are parallel.

9. Set up and run a 3 point bending experiment as follows:

- Exit
  - Change mode to Temp/time Scan
  - Exit

- Run system
  - Set-up and run
    - Modify parameters
      - Measuring System 3 point bending
      - Sample I.D.=_____
      - Comment = _____
      - Operator I.D.=_____
      - Sample Geometry - Rectangle

- On the DMA
  - Probe up
  - Install Auto zero block
  - Probe down

- Modify Parameters
  - Sample zero (read after Y displacement is displayed and stable via the insert key)

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9. continued

- On the DMA
  - Raise probe and remove Auto zero block
  - Install sample, square to the knife edges
  - Lower probe

- Modify Parameters
  - Sample height (read after Y displacement is displayed and stable via the insert key)
  - Type in width = 15mm
  - Type in Depth
  - Exit

- On the DMA
  - Raise the furnace (check coolant)

- FUNCTION KEY "GO TO TEMP" of 50°C (observe DMA indicator lights)

- Modify Parameters
  - STATIC STRESS = 1.1 e6 Pa
  - DYNAMIC STRESS = 1.0 e6 Pa
  - Frequency = 1 Hz
  - Static Control = Tension
  - Static Set Point = 110%
  - Dynamic Control = Force
  - Dynamic Setpoint -
  - Temperature Program
    - Temp 1 = 50°
    - Time 1 = ____
    - Rate 1 = 5.0°C/minute
    - Temp 2 = 250°C
    - End Condition = Go To Load

Note: use the Page Down Key to go to the next page of this method.

- Display Parameters
  - Auto-Optimize = ON
  - X Scale = Temperature

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9. continued.

- X Min = _____
- X Max = _____
- Y1 Min = 1.0 e6 Pa
- Y1 Max = 3.0 e10 Pa
- Y2 Min = 0.0
- Y2 Max = 0.5
- Data/Atmosphere Parameters
  - Data Delay = _____
  - Data Size = 500
  - Purge Gas A = Helium
  - Purge Gas B =
  - Valve 1 time = 0.0 min
  - Valve 2 time = 0.0 mi
  - Exit

10. View the status line

	Original Values	Doubled Stress
Record Time		
Temp		
Freq		
S. Stress		
D. Stress		
Strain		
Height		
Amplitude		
Phase		

Note: Depress the Control and PLUS (+) Key simultaneously to change to the next status line.

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Original Values Double Stress

Time _____

F_s _____

F_d _____

F_t _____

F_o _____

Height _____

Amplitude _____

Phase _____

Note: Depress the Control and Plus (+) Key simultaneously to change to the next status line.

Time _____

K_s _____

D_s _____

T_b _____

Height _____

Amplitude _____

Phase _____

Note: Depress the control and Plus (+) Key simultaneously to change to the next status line.

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Note: Depress the control and Plus (+) Key simultaneously to change to the next status line.

11. Depress the static control function key.

12. Via modify parameters increase the static and dynamic stresses until the amplitude on the status line exceeds 5.0μ.

13. Wait for the DMA to equilibrate (view method key) with no controls on.

14. Change the Y1 axis so that the modulus cursor is on scale.

15. Record: Height _____
    Amplitude _____
    Phase_____

16. Begin the run with no controls on.

17. During the rum, change the display if required or desired.

18. Observe the DMA Task Block at the top of the screen (Red Block).
    What is displayed there? _____

19. During the run, change the display if required or desired.

20. Observe the DMA Task Block at the top of the screen (Red Block).
    What is displayed there? _____

21. At 200°C record the status line

    Record: Time = _____
    Temp = _____
    Freq = _____
    S. Stress = _____
    D. Stress =____

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Strain =_____
Height = _____
Amplitude = ____
Phase = _____

22. Just after the run is over, observe the indicator lights on the DMA.

23. Save the DATA.

24. Calculate the onset of the Modulus

25. Calculate the Peak of Tan Delta

26. Calculate the Peak of the Loss Modulus

27. Create a table of the Loss Modulus from 100° to 200°

28. Recall another 3 point bending curve from the DMA directory and create a table of the modulus curve from 100° to 200°

29. Compare the 2 data sets.

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DMA LABORATORY
Experiment #3A
3 Point Bending

Stress Scan
Thick Pencil Lead

Objective: This laboratory experiment will allow the student to become more familiar with :

- Set-up for making a run
- Sample handling
- Creating a method
- Making a run
- Data handling/manipulation

Directions: Read the entire question or instructions before responding.

1. Check the purge gas and rate. RECORD: RATE = _____

   GAS = _____

2. Check the coolant.

3. The sample will be pencil lead. Measure the diameter of the sample and record _____ mm.

4. Put sample aside for now.

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5. Set-up and run a 3 point bending experiment as follows:

- o Change mode to stress scan
  - o Exit
  - o Run system
  - o Set up and run
  - o Modify Parameter

  - o Sample ID = _____

  - o Comment = _____

  - o Operator ID = _____

  - o File Name = _____

  - o Measuring system = 3 point bending

- o Sample Geometry = Cylinder
  - o Enter all necessary info

6. What is another method of measuring the sample diameter?

- o On the DMA
  - o Load Sample
  - o Raise furnace

- o DMA 7 Parameters
  - o Static Force = 12 mN
  - o Frequency = 1.00 Hz

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6. continued

- DMA Program
  - Hold for = _____
  - Initial Force = 10 mN
  - Force Rate = 10 mN/min
  - Final Force = Max Allowed
  - Enc Condition = Go To Load
  - Static Control = Tension
  - Static Setpoint = 120%
  - Function key GOTO TEMP of = 30°C

  Display Parameters
  - Auto Optimize = ON
  - X Scale = Dynamic Force
  - Minimum = _____
  - Maximum = _____
  - Y1 Minimum = 0
  - Y1 Maximum = _____
  - Y2 Minimum = _____
  - Y2 Minimum = _____
  - Y2 Maximum = _____

  Data/Atmosphere Parameters
  - Data Delay = 0 min
  - Data Size = 2000 pts
  - Purge Gas A = Helium
  - Purge Gas B =
  - Valve 1 Time = 0.0
  - Valve 2 Time = 0.0
  - Exit

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7. continued.

Record:

TEMP	STATIC STRESS	DYNAMIC STRESS	STRAIN	AMPLITUDE	PHASE

Initial Force:

Adjusted Force:

8. Wait until DMA has equilibrated. (View all status lines).

9. Start Run. (Static control should be ON).

10. During this run, on the reverse side of this paper, list all the data catagories of all the status lines.

11. During this run, change to another task and recall another data file.
Analyze this data as you choose.

12. Return to the current run.

13. After the run is complete, save the file.

14. Manipulate the curves on the screen.
To achieve this order:
X Axis = Force
Y1 Axis = Dynamic Stress
Y2 Axis = Strain
Curve #3 = Phase Angle

15. Optimize data so that the phase angle curve resolution of its Y axis is at least one decade.

16. Plot this curve arrangement.

17. Identify the minimum dynamic stress

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15. Optimize data so that the phase angle curve resolution of its Y axis is at least one decade.

16. Plot this curve arrangement.

17. Identify the minimum dynamic stress

18. Identify the minimum visco-elastic limit.

19. Identify the maximum visco-elastic limit.

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DMA LABORATORY
Experiment #3B
3 Point Bending

Stress Scan
Thin Pencil Lead

Objective: This laboratory experiment will allow the student to become more familiar with :

- Set-up for making a run
- Sample handling
- Creating a method
- Making a run
- Data handling/manipulation

Directions: Read the entire question or instructions before responding.

1. Check the purge gas and rate. RECORD RATE: _____

   GAS: _____

2. Check the coolant.

3. The sample will be pencil lead. Measure the diameter of the sample and record _____ mm.

4. Put sample aside for now.

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5. Set-up and run a 3 point bending experiment as follows:

   - o Change mode to stress scan
     - o Exit
     - o Run system
     - o Set up and run
     - o Modify Parameter

     - o Sample ID = _____

     - o Comment = _____

     - o Operator ID = _____

     - o File Name = _____

     - o Measuring system = 3 point bending

   - o Sample Geometry = Cylinder
   - o Enter all necessary info

6. What is another method of measuring the sample diameter?

   - o On the DMA
     - o Load Sample
     - o Raise furnace

   - o DMA 7 Parameters
     - o Static Force = 12 mN
     - o Frequency = 1.00 Hz

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6. continued

- DMA Program
  - Hold for = _____
  - Initial Force = 10 mN
  - Force Rate = 10 mN/min
  - Final Force = Max Allowed
  - Enc Condition = Go To Load
  - Static Control = Tension
  - Static Setpoint = 120%
  - Function key GOTO TEMP of = 30°C

  Display Parameters
  - Auto Optimize = ON
  - X Scale = Dynamic Force
  - Minimum = _____
  - Maximum = _____
  - Y1 Minimum = 0
  - Y1 Maximum = _____
  - Y2 Minimum = _____
  - Y2 Minimum = _____
  - Y2 Maximum = _____

  Data/Atmosphere Parameters
  - Data Delay = 0 min
  - Data Size = 2000 pts
  - Purge Gas A = Helium
  - Purge Gas B =
  - Valve 1 Time = 0.0
  - Valve 2 Time = 0.0
  - Exit

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7. View the status line via the "View Method" hard key. Increase the Initial Force and Static Force under "Modify Parameter" so that ΔY increases above 1.0 u with the tension control on.

   Record:

TEMP	STATIC STRESS	DYNAMIC STRESS	STRAIN	AMPLITUDE	PHASE

Initial Force:

Adjusted Force:

8. Wait until DMA has equilibrated. (View ALL status lines).

9. Start Run. (Tension control should be ON).

10. During this run, on the reverse side of this paper, list all the data catagories of the status lines.

11. During this run, change to another task and recall another data file.
    Analyze this data as you choose.

12. Return to the current run.

13. After the run is complete, save the file.

14. Manipulate the curves on the screen.
    To achieve this order:
    X Axis = Force
    Y1 Axis = Dynamic Stress
    Y2 Axis = Strain
    Curve #3 = Phase Angle

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15. Optimize data so that the phase angle curve resolution of its Y axis is at least one decade.

16. Plot this curve arrangement.

17. Identify the minimum dynamic stress

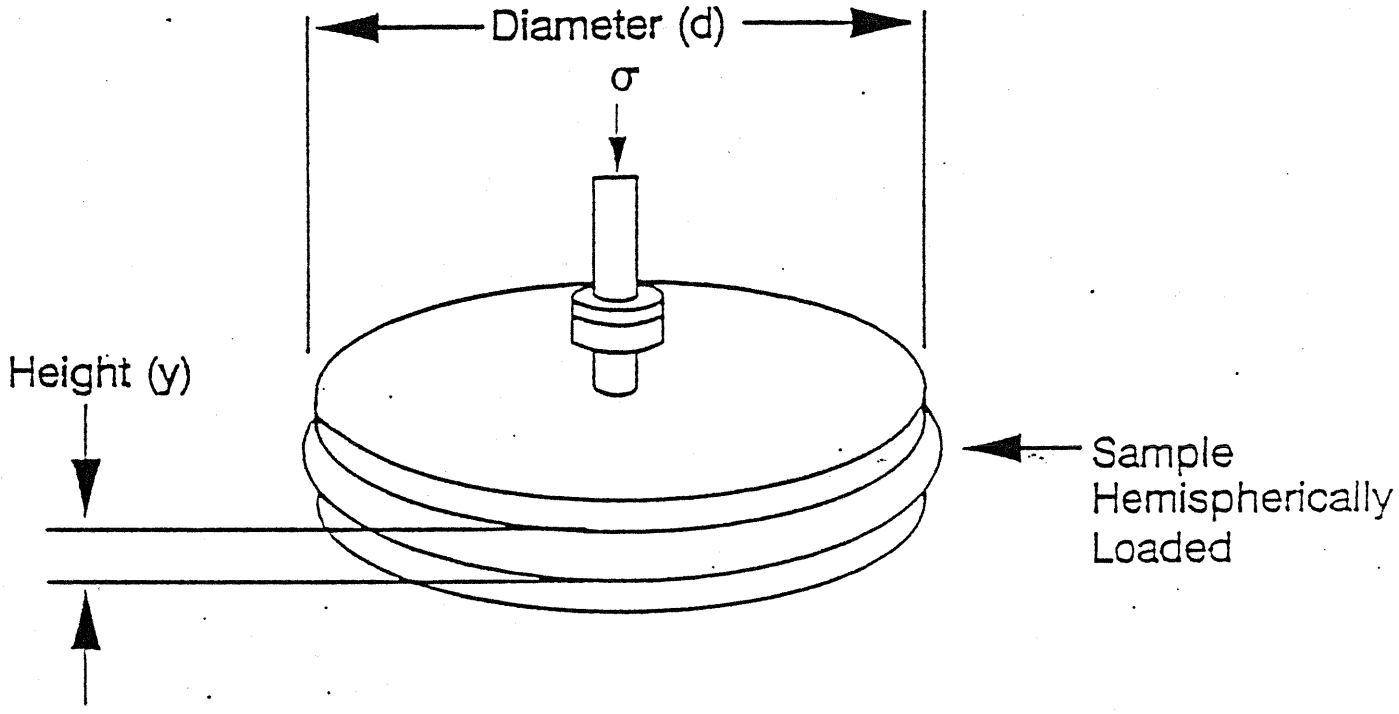
18. Identify the minimum visco-elastic limit.

19. Identify the maximum visco-elastic limit.

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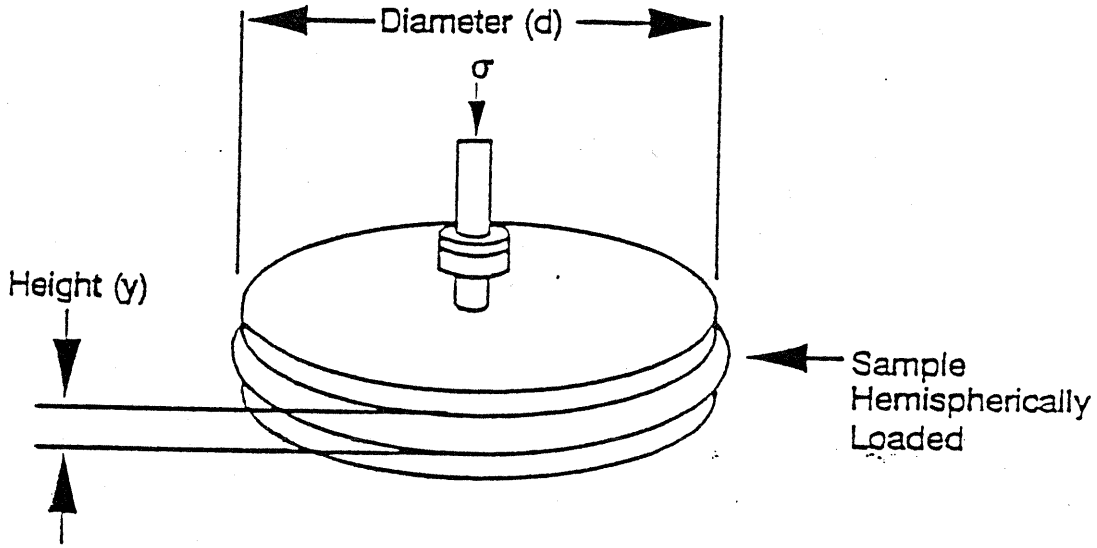
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Parallel Plate Dimensions



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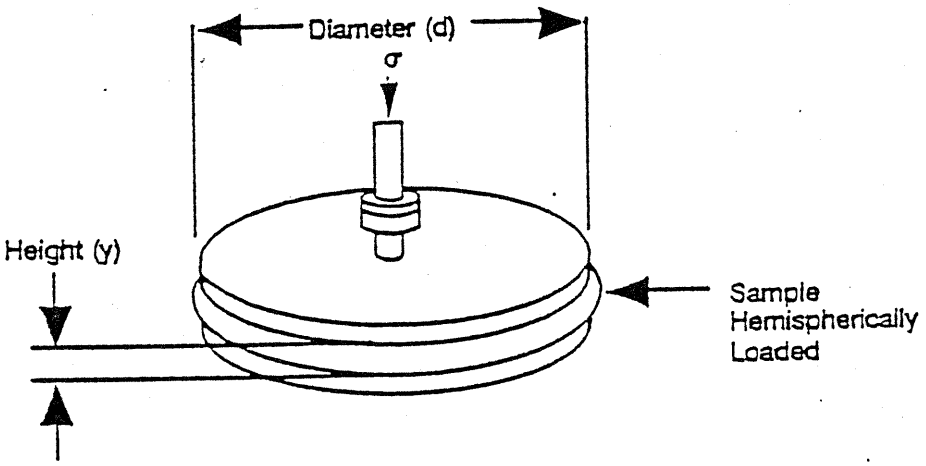
Parallel Plate Samples



- Soft samples
  - Those which cannot otherwise be held; e.g., silicone
- Samples above T_g
  - Ability to scan over wide temperature in a single run

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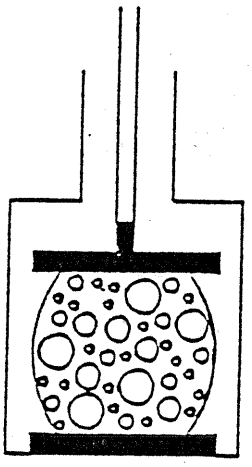
Parallel Plate Samples



- Materials going through dramatic changes
  - Two to three decades in Modulus; e.g., epoxy cure
- High Creep, High Temperature
  - Samples that may be difficult to hold; e.g., PTFE
- Pellets
  - No sample preparation necessary

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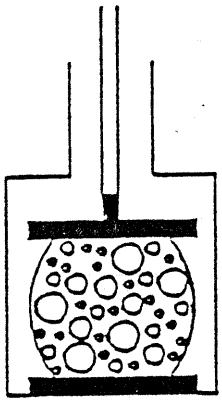
Parallel Plate
Samples



- Sample Placement / Plate Alignment
  - Center on bottom plate

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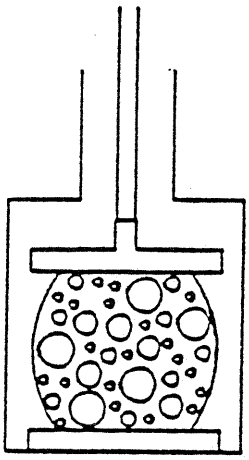
Parallel Plate
Samples



- What size plate to use
  - Plate should match sample size
  - If sample flows (PDMS), plate should match sample after equilibrium
  - For smaller samples, top plate can be smaller than bottom plate

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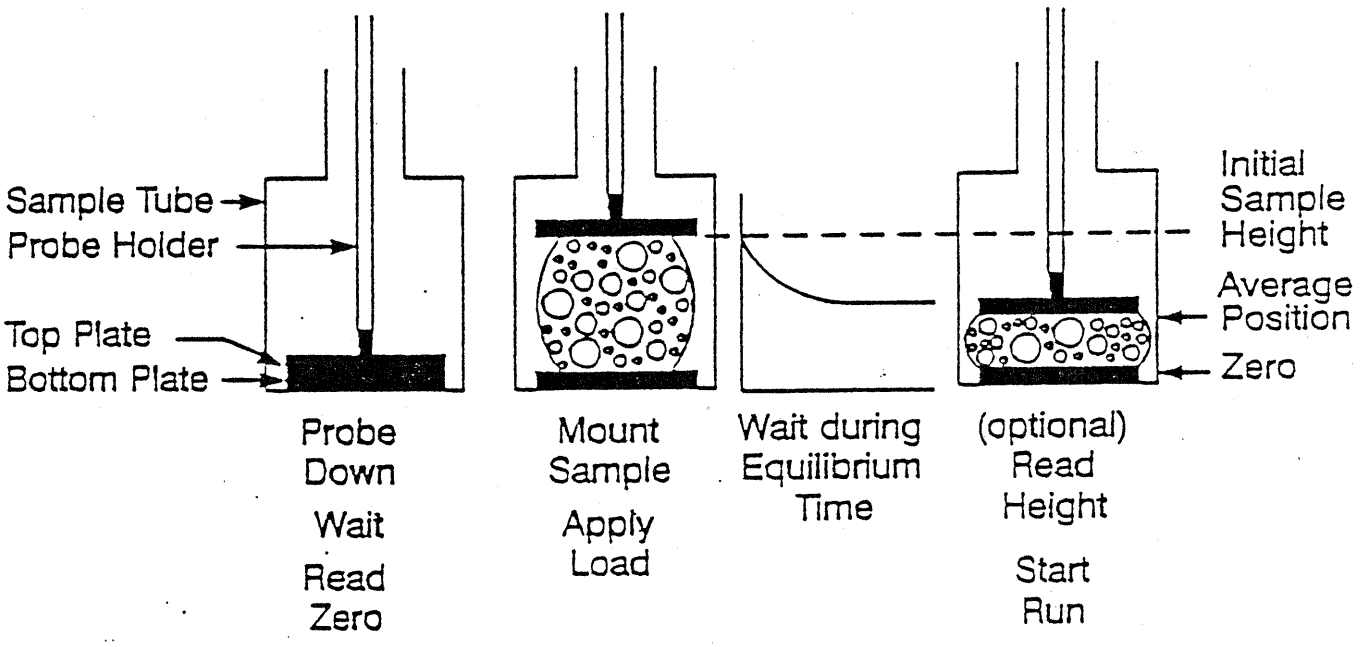
Parallel Plate
Samples



- How much sample to use
  - Sufficient to create 1 to 2 mm gap
  - Match (upper) plate size and avoid spilling over the plate edges

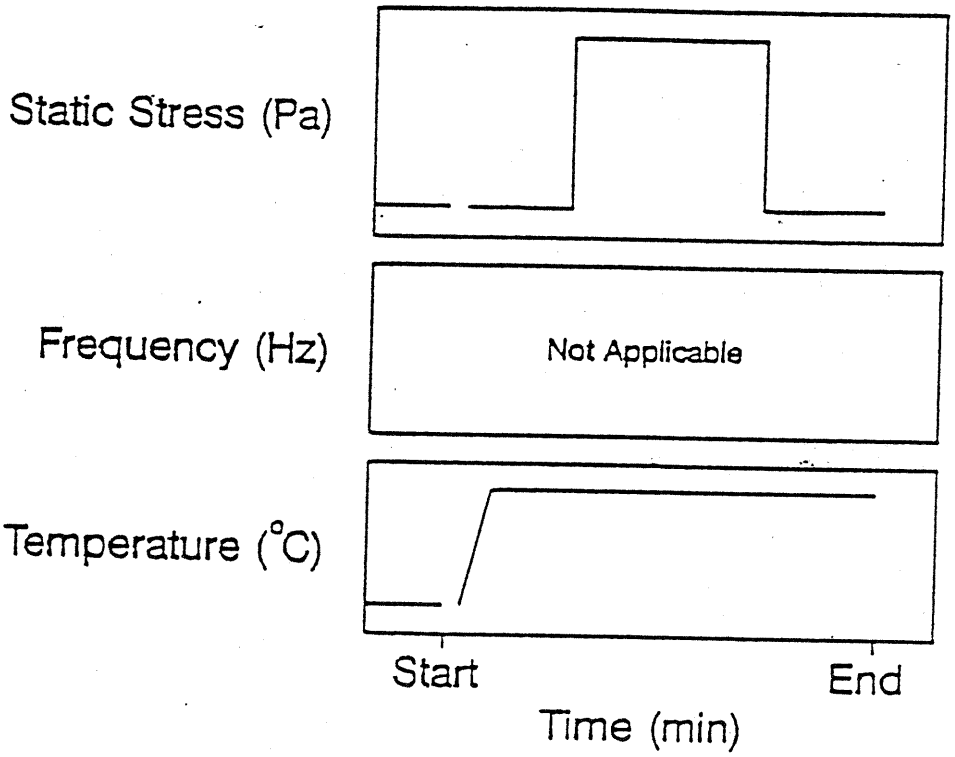
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Parallel Plate
Mounting Soft Samples



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DMA 7 Mode
Creep-Recovery



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Creep-Recovery
Typical Applications

Typical Measurements	Typical Uses
Creep compliance	Effect of load
Creep rupture	Strength evaluation
Equilibrium compliance	Long-time behavior
Equilibrium viscosity	Extensional viscosity (Meissner)
Stress relaxation	Dimensional stability
Retardation spectra	Molecular modeling

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The Basics of Creep-Recovery Testing

- No dynamic (sinusoidal) stress is applied
- The response to a step change in the constant stress is measured
- The DMA 7 Creep experiment measures the time-dependent strain in response to an increase in stress
- The DMA 7 Creep-Recovery experiment measures the time-dependent strain in response to a removal of a previous stress
- A similar test performed by controlled strain analyzers is the Strain-Relaxation test
- Any of the measuring systems can be used in this mode

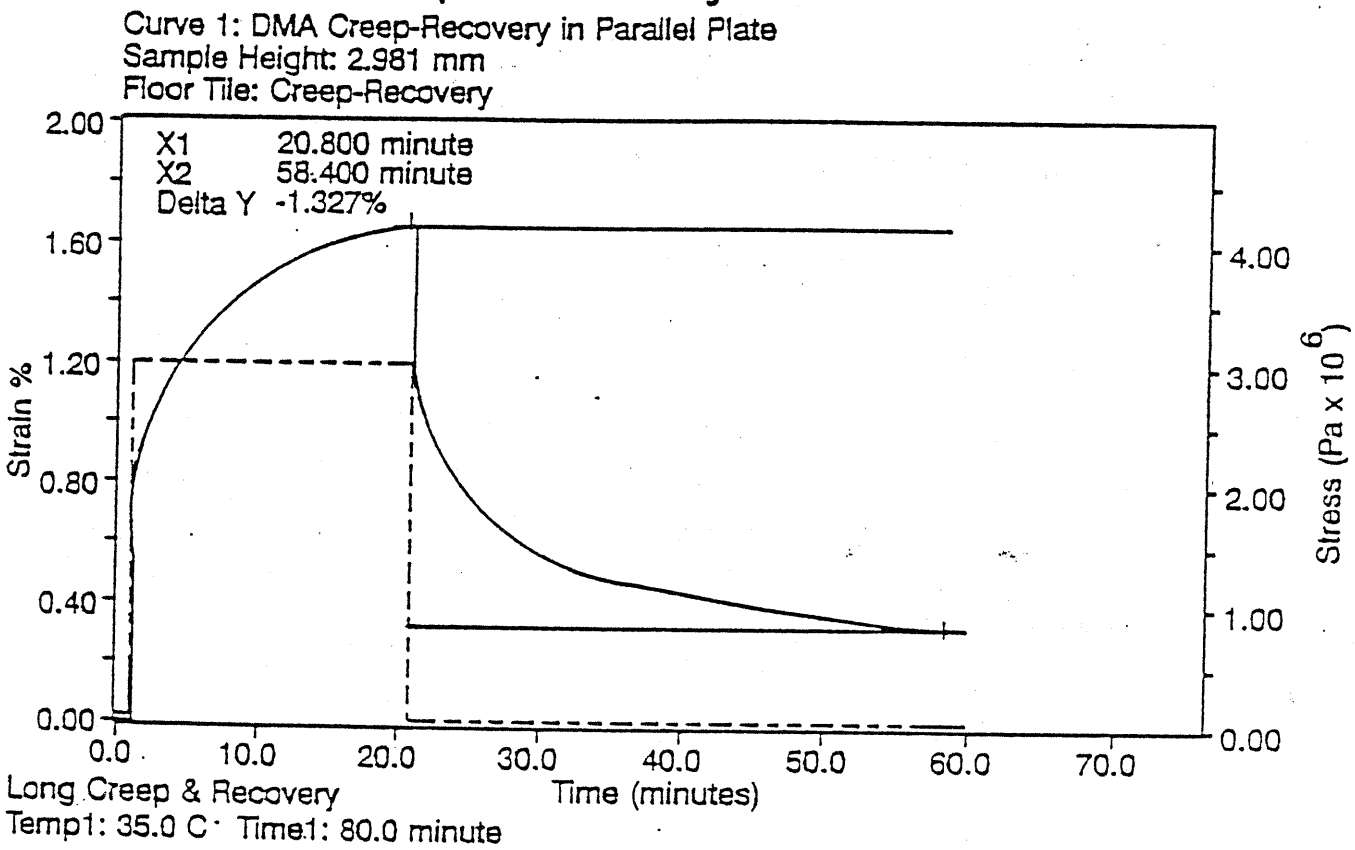
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Report Parameters for Creep-Recovery

- Time-dependent data
  - Time-dependent modulus
  - Time-dependent compliance
  - Retardation spectrum
- Equilibrium and steady state parameters
  - Equilibrium or steady state compliance
  - Equilibrium or steady state viscosity

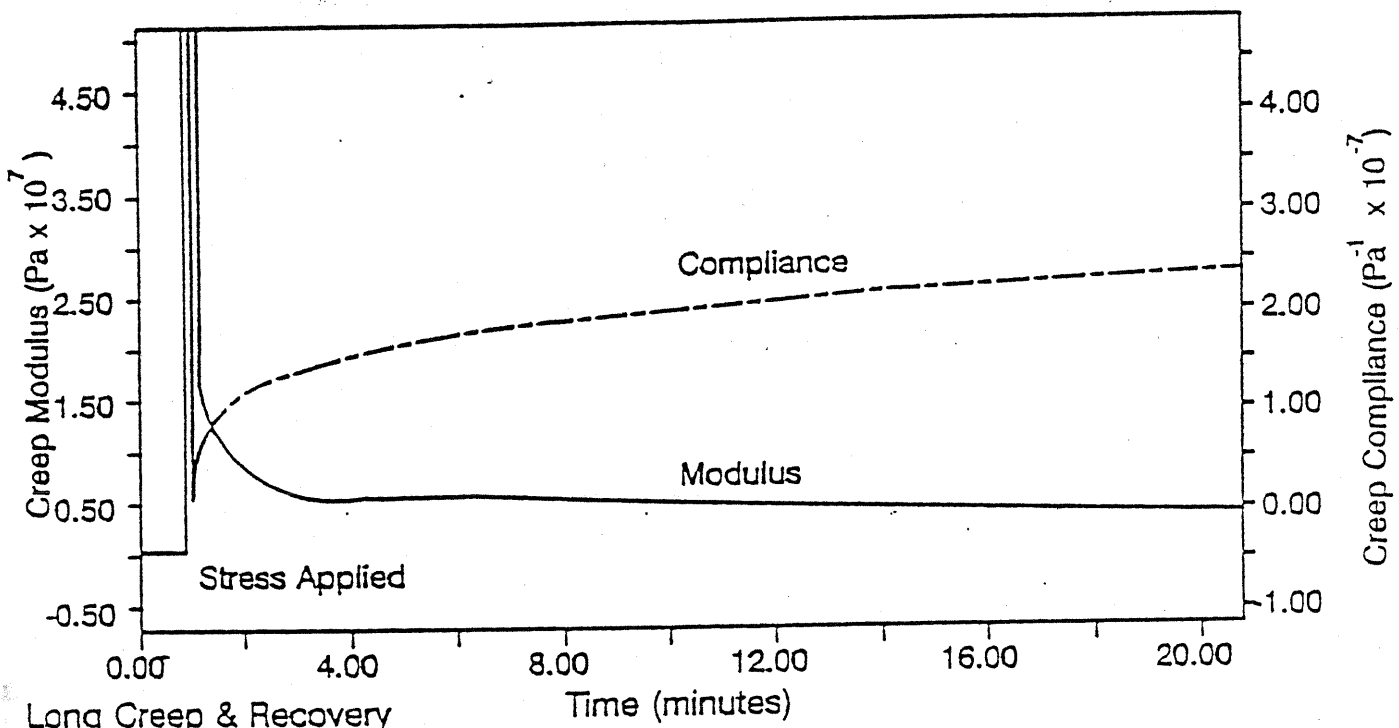
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Creep-Recovery of Floor Tile



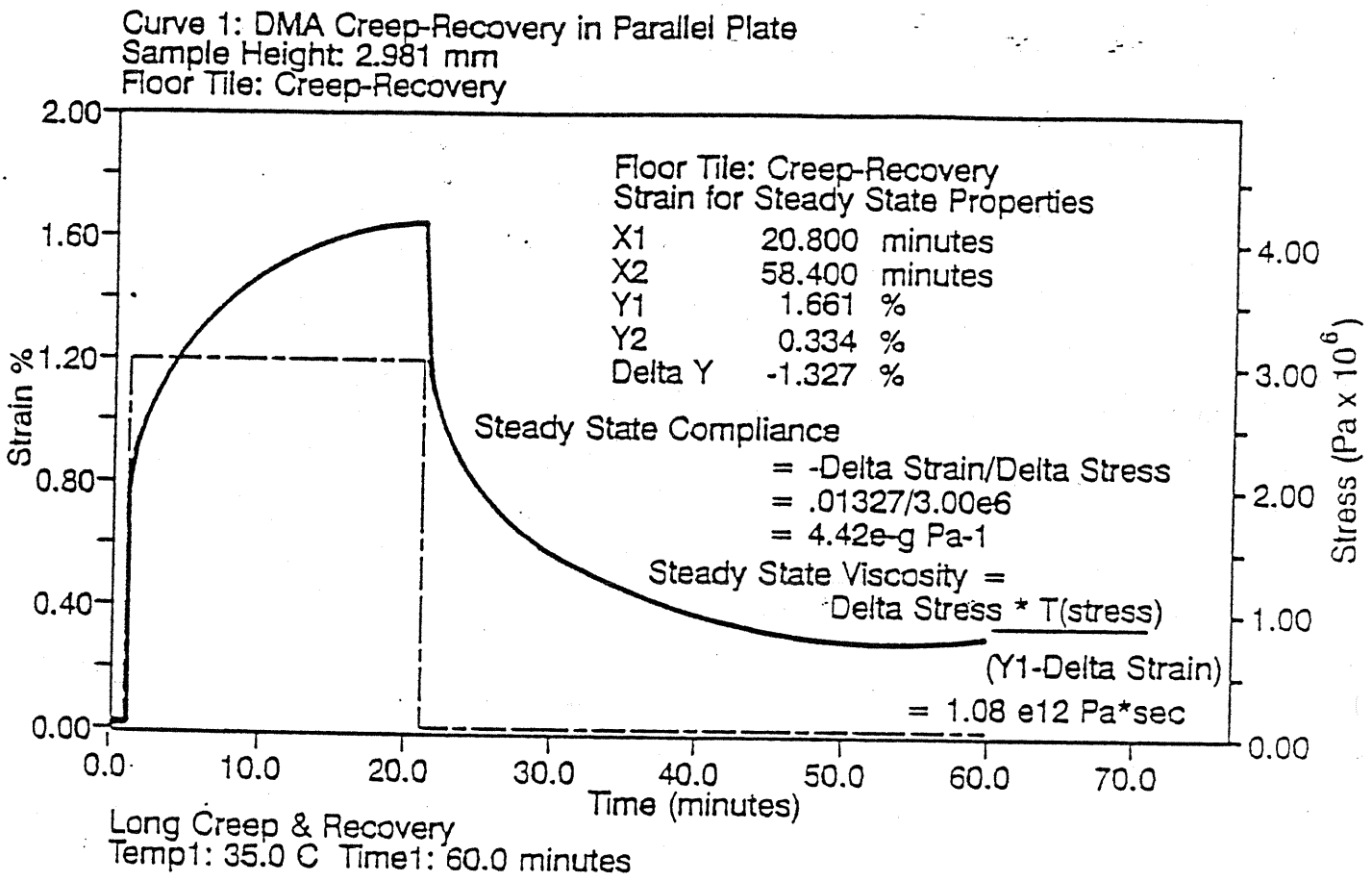
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Curve 1: DMA Creep-Recovery in Parallel Plate
Floor Tile: Creep-Recovery



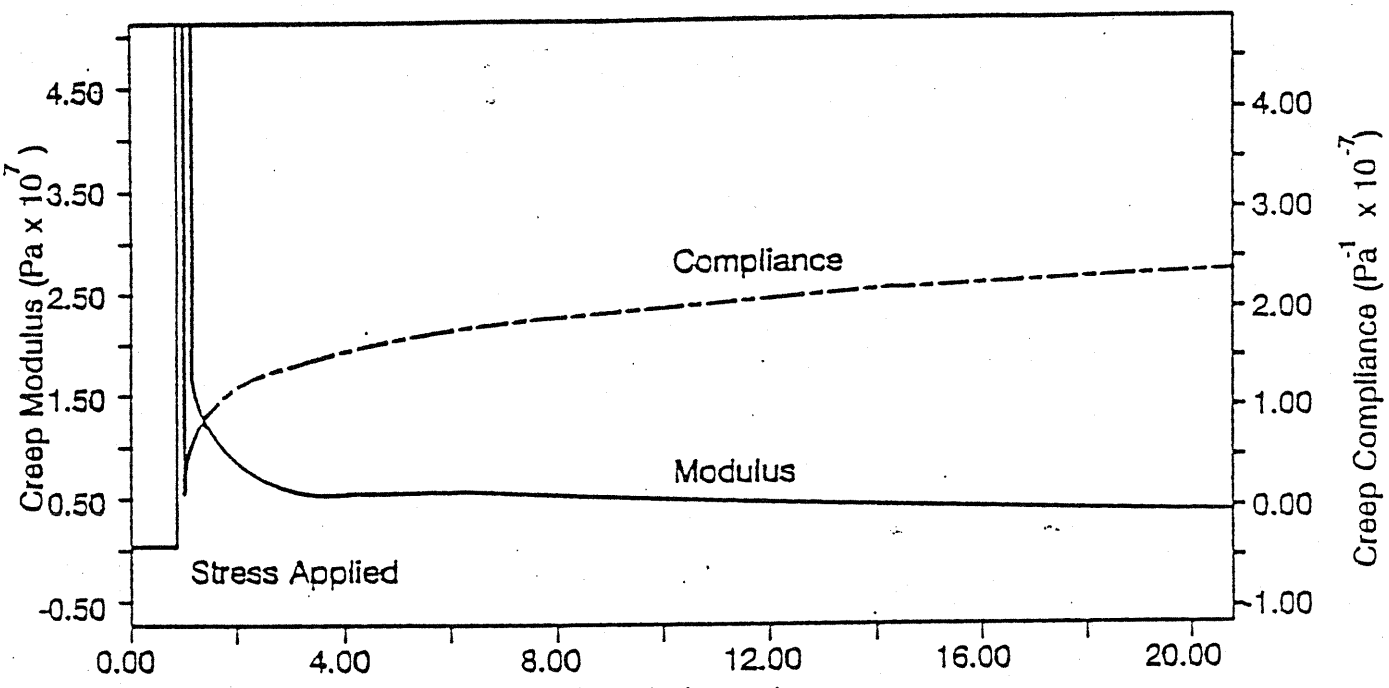
Long Creep & Recovery
Temp1: 25.0 C Time1: 60.0 minute

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Curve 1: DMA Creep-Recovery in Parallel Plate
Floor Tile: Creep-Recovery



Long Creep & Recovery
Temp1: 25.0 C Time1: 60.0 minutes

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Floor Tile: Creep-Recovery

Sample Height: 2.972 mm

Entry No.	Time (minutes)	Y Data Creep Modulus	Entry No.	Time (minutes)	Y Data Creep Modulus
1	1.00	1.289e+008	16	16.00	4.513e+006
2	2.00	8.543e+006	17	17.00	4.431e+006
3	3.00	7.053e+006	18	18.00	4.370e+006
4	4.00	6.365e+006	19	19.00	4.320e+006
5	5.00	5.994e+006	20	20.00	4.284e+006
6	6.00	5.653e+006			
7	7.00	5.460e+006			
8	8.00	5.284e+006			
9	9.00	5.140e+006			
10	10.00	5.025e+006			
11	11.00	4.892e+006			
12	12.00	4.791e+006			
13	13.00	4.698e+006			
14	14.00	4.627e+006			
15	15.00	4.574e+006			

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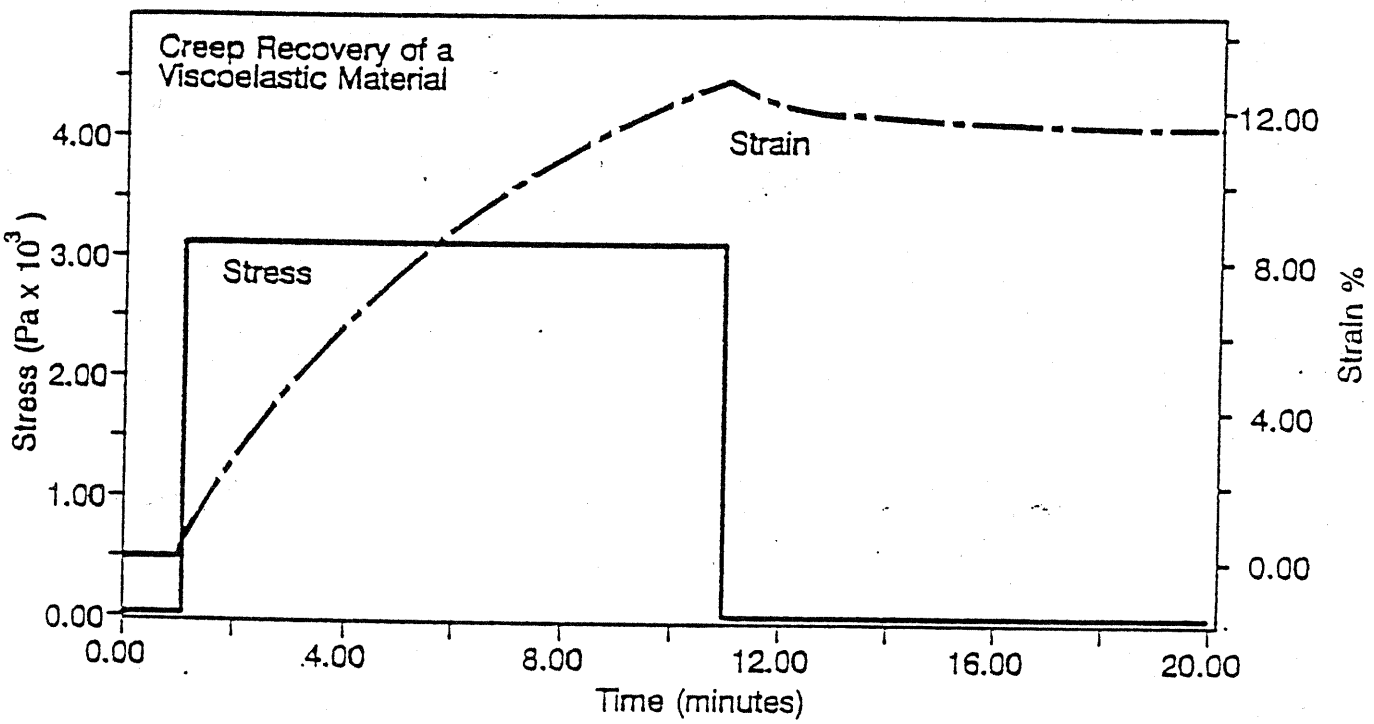
Floor Tile: Creep-Recovery

Sample Height: 2.972 mm

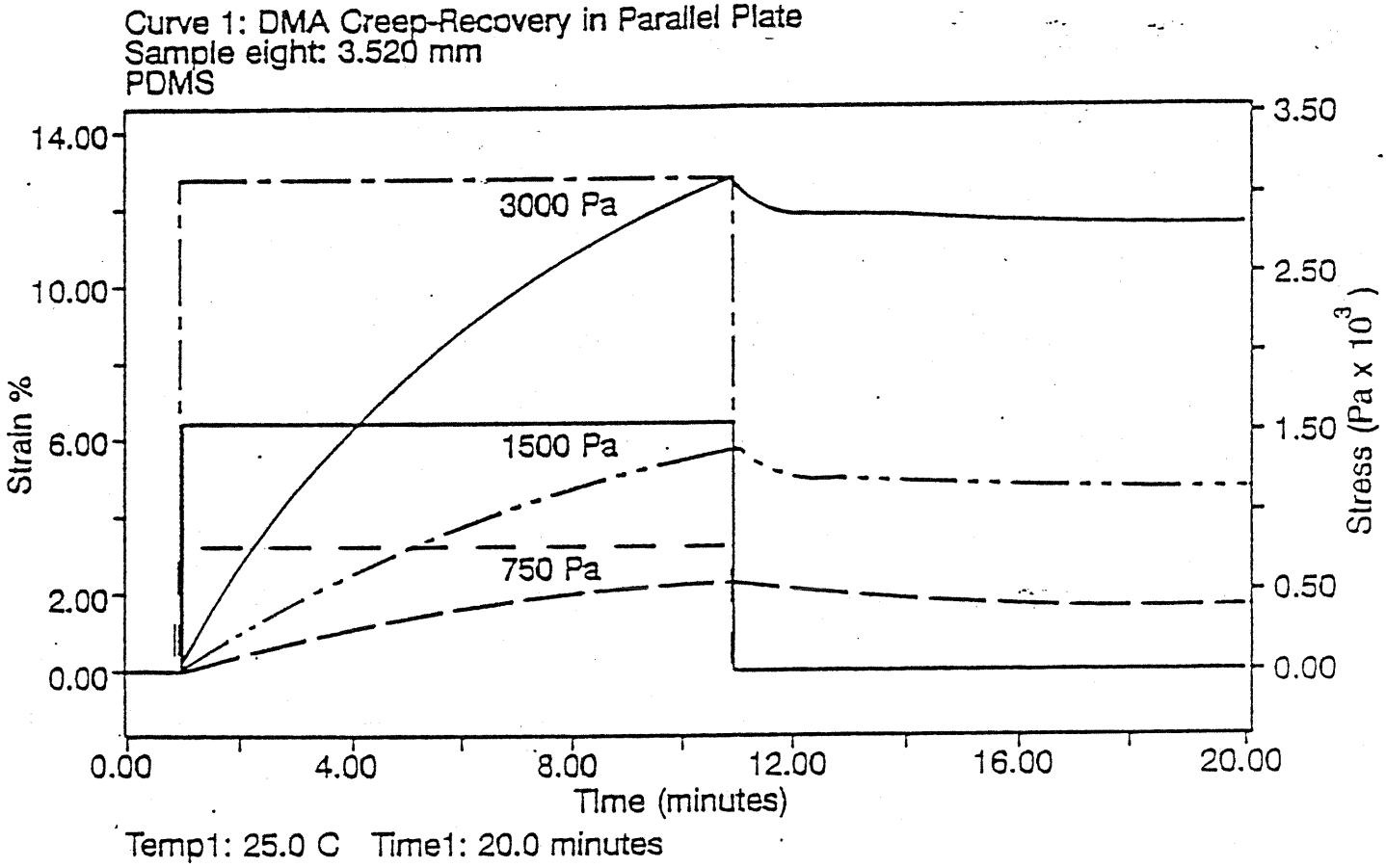
Entry No.	Time (minutes)	Y Data Creep Compliance	Entry No.	Time (minutes)	Y Data Creep Compliance
1	1.00	7.760e-009	16	16.00	2.216e-007
2	2.00	1.170e-007	17	17.00	2.257e-007
3	3.00	1.418e-007	18	18.00	2.288e-007
4	4.00	1.571e-007	19	19.00	2.315e-007
5	5.00	1.668e-007	20	20.00	2.334e-007
6	6.00	1.769e-007			
7	7.00	1.832e-007			
8	8.00	1.893e-007			
9	9.00	1.945e-007			
10	10.00	1.990e-007			
11	11.00	2.044e-007			
12	12.00	2.087e-007			
13	13.00	2.128e-007			
14	14.00	2.161e-007			
15	15.00	2.186e-007			

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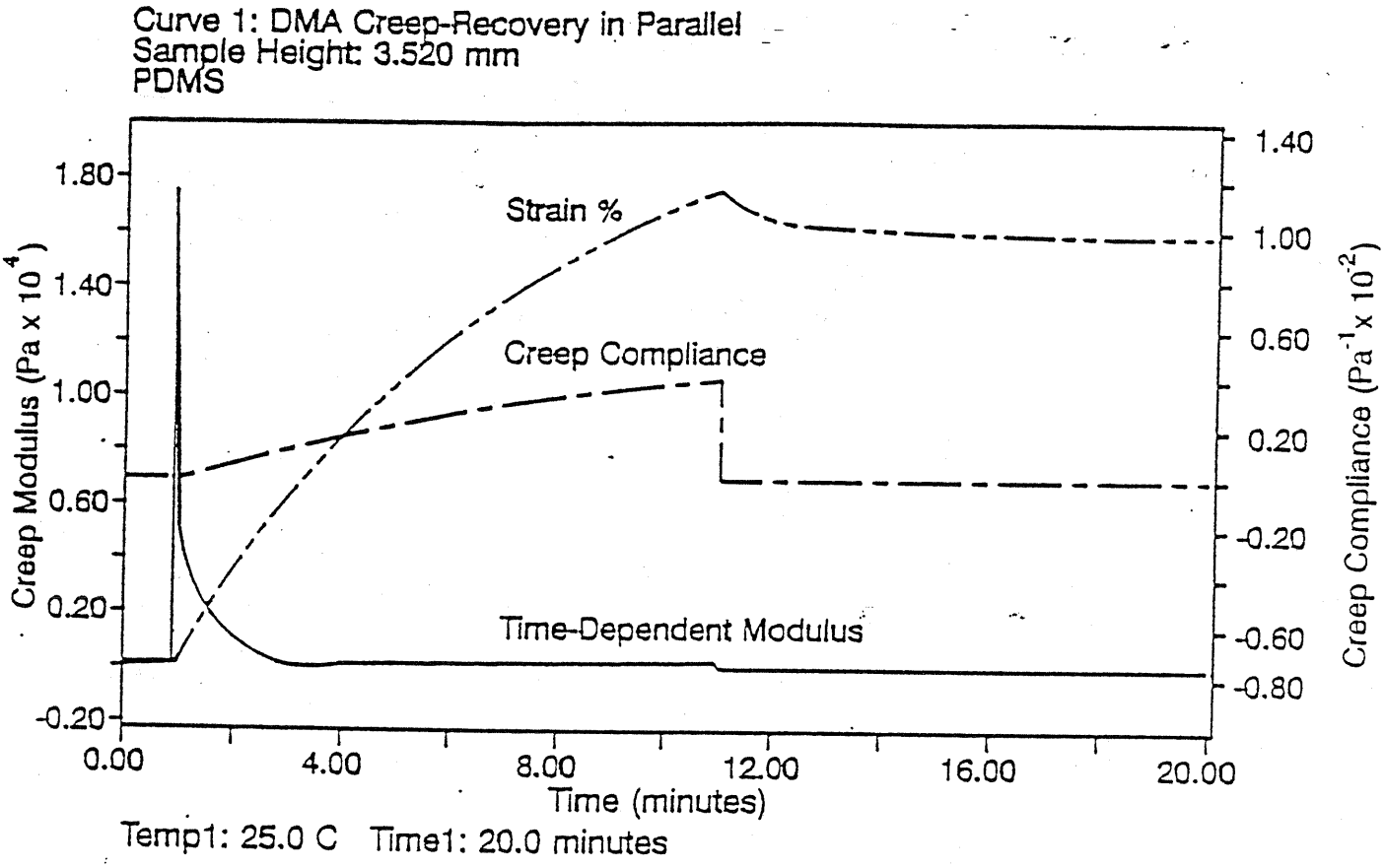
DMA Creep-Recovery in Parallel Plate
Sample Height: 3.520 mm
PDMS



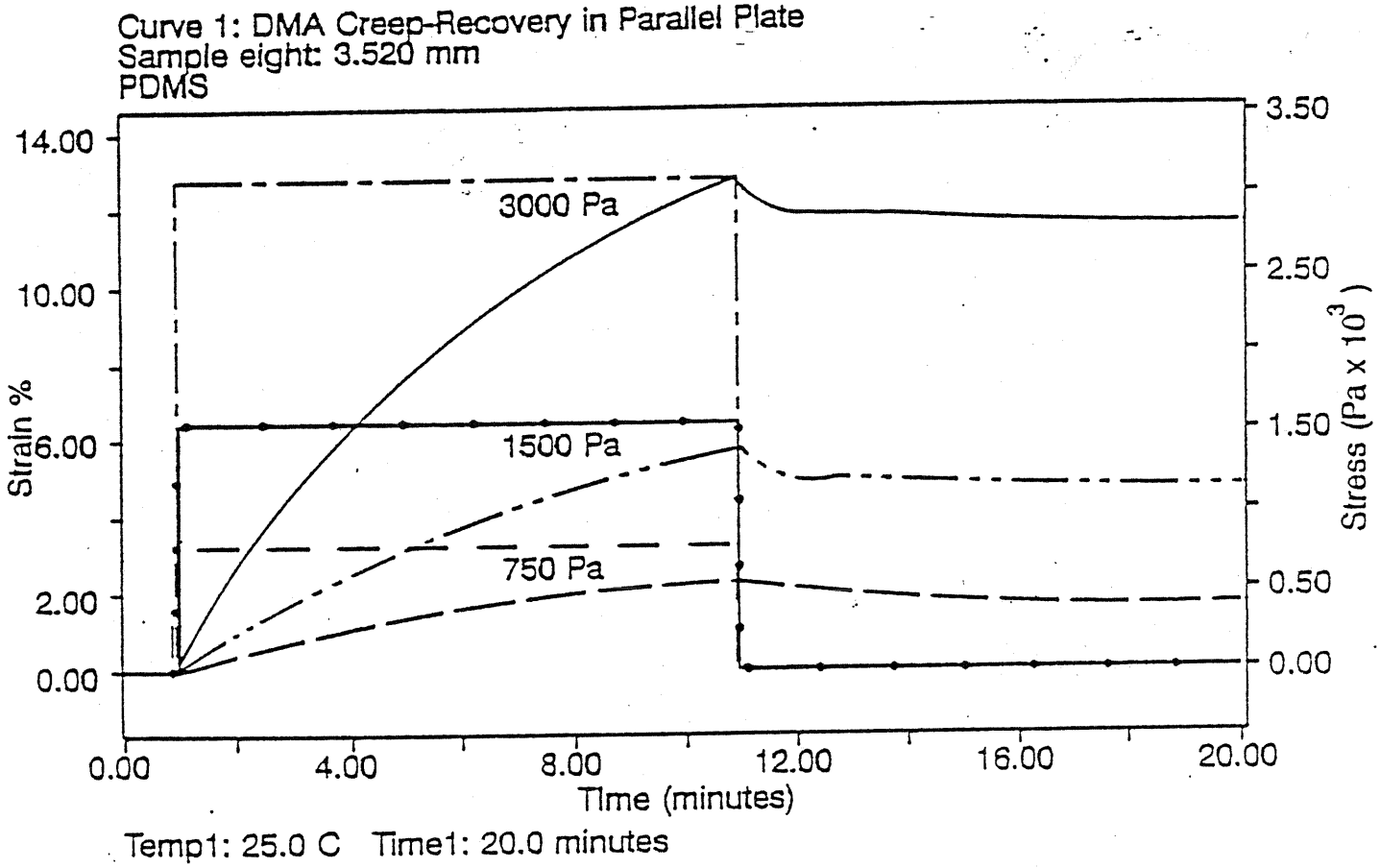
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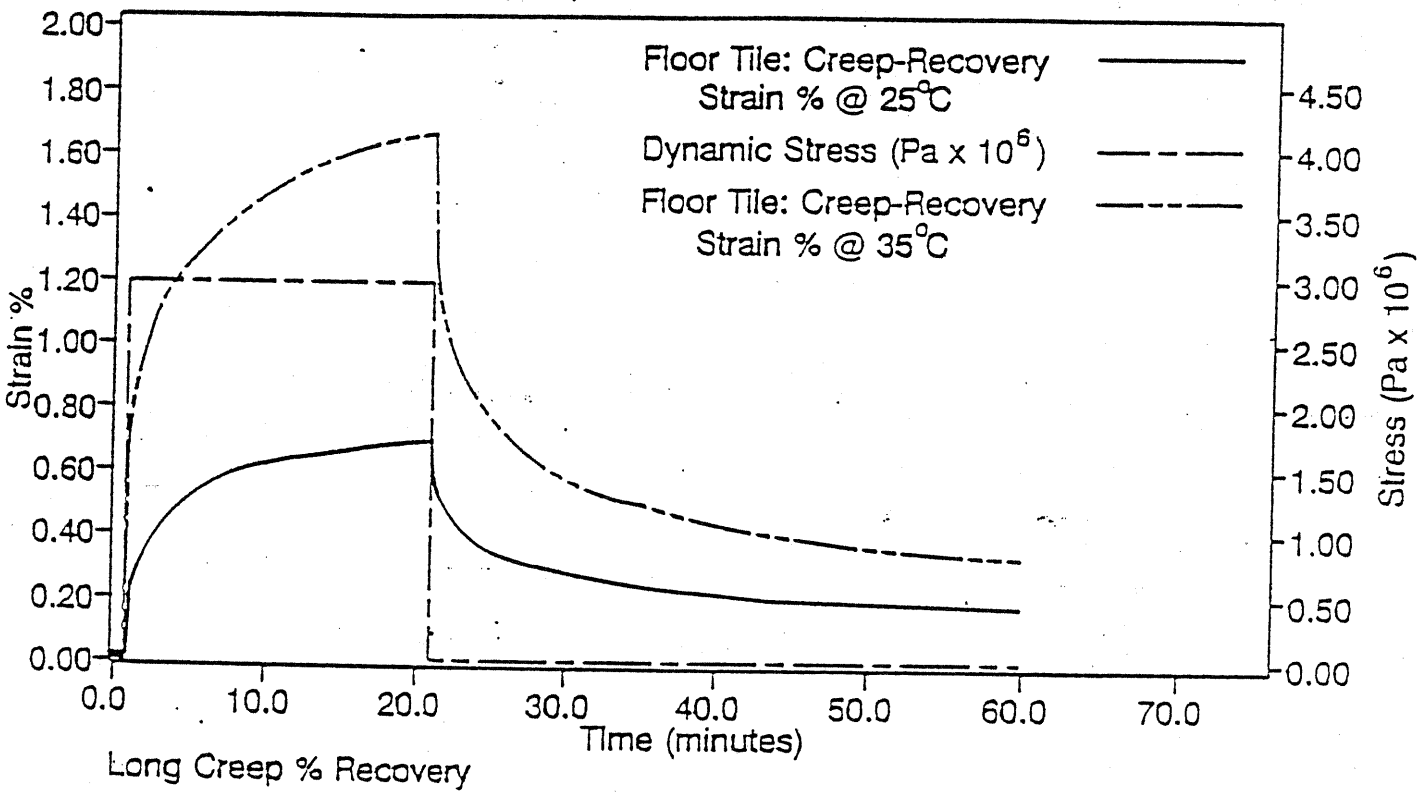


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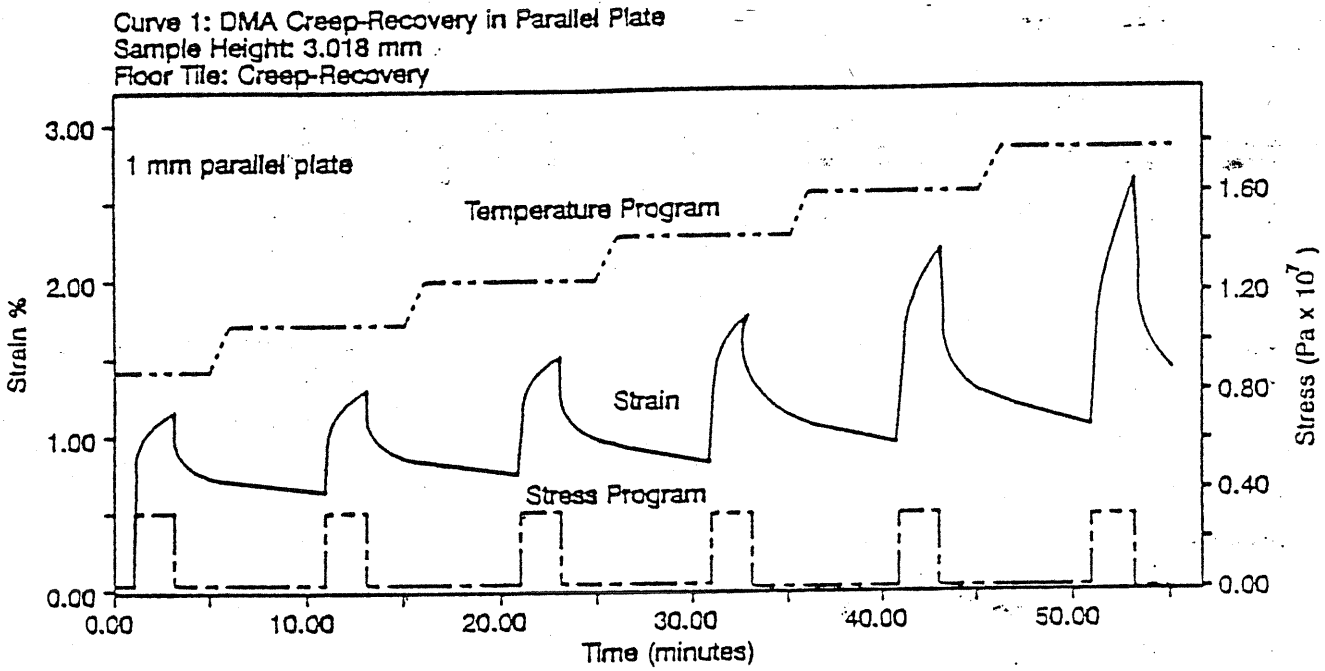
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Curve 1: DMA Creep-Recovery in Parallel Plate
Sample Height: 2.972 mm
Floor Tile: Creep-Recovery



Long Creep % Recovery

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Multi-Ramp Creep Setup

Temp1:	20.0 C	Time 1:	5.0 min	Rate 1:	4.0 C/min
Temp2:	24.0 C	Time 2:	9.0 min	Rate 2:	4.0 C/min
Temp3:	28.0 C	Time 3:	9.0 min	Rate 3:	4.0 C/min
Temp4:	32.0 C	Time 4:	9.0 min	Rate 4:	4.0 C/min
Temp5:	36.0 C	Time 5:	9.0 min	Rate 5:	4.0 C/min
Temp6:	40.0 C	Time 6:	9.0 min	Rate 6:	4.0 C/min
Temp7:	44.0 C	Time 7:	9.0 min	Rate 7:	4.0 C/min
Temp8:	48.0 C	Time 8:	9.0 min		

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DMA LABORATORY
Experiment #4
Parallel Plate

FREQUENCY SCAN
SILLY PUTTY (PDMS)

OBJECTIVE: This laboratory experiment will allow the student to become familiar with:

Set-up for making a run
Sample handling
Creating a method
Making a run
Data handling/manipulation

DIRECTIONS: Read the entire question or instruction before responding.

1. Check the purge gas and rate.
2. Check coolant.
3. Install the 15mm parallel plate measuring system.
4. Lower probe.
5. Check T/C position.
6. Change mode to frequency scan and exit.
7. Run system.
   - o Set up and run

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8. Set-up and run a parallel plate experiment as follows:

- Modify Parameter
  Sample Parameters
  - Measuring system = Parallel Plate
    - Sample ID = _____
    - Operator ID = _____
    - Comment = _____
    - Disc Geometry
      - Type in Diameter = 15 mm
      - Sample zero (read zero via the insert key)

9. On the DMA

- Raise probe and install sample (sample should be the size of a pea)
- Raise the furnace
- Raise the Analyzer temperature to 30°C

NOTE: To change Dynamic and Static stress from pascals to millinewtons or vice-versa, one must exit to "change defaults" to do so.

DMA 7 Parameters
- Static Force = 85 mN
- Dynamic Force = 100 mN

DMA 7 Program
- Initial Frequency = 0.1 Hz
- Final Frequency = 0.01 Hz
- End Condition = Go To Load
- Static Control = Tension
- Static Set point = 85%

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9. continued

- o Dynamic Control = Force
- o Dynamic Set point = None

Display Parameters

- o Modify as required

Data/Atmosphere Parameters

10. View Status line, wait until height equilibrium is reached.

11. Modify Parameters
    - o Sample Height (read height via the insert key)

12. On the DMA - Check the control light.

13. Begin Run.

14. Modify Display during run.

15. Save File.

16. Change curve types.

17. Analyze data.

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DMA LABORATORY
Experiment #5

Parallel Plate
Creep-Recovery Mode
Septum: Multi-Ramp

OBJECTIVE: This laboratory experiment will allow the student to become more familiar with:

- Set-up for making a run
- Sample handling
- Creating a method
- Making a run
- Data handling

DIRECTIONS: Read the entire question or instruction before responding.

1. What 2 variables should be checked before beginning a run.

   #1_____

   #2_____

2. Measure the Diameter of the septum._____mm

3. Set-up and run a parallel plate experiment as follows.

   - Change Mode to creep-recovery
   - On the DMA
     - Lower probe

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3. continued
   - Modify Parameters
     - Geometry =_____
     - Sample Zero (Read zero via the _____ key.)
   - On the DMA
     - Raise the probe
     - Install the sample, centered to the probe.
     - Lower the probe
   - Modify Parameters
     - Sample height
   - DMA 7 Program
     - Creep Ramp = Off
     - Hold for = 1.0 Min
     - Creep Force = 2500mN (or max)
     - For = 2.5 min
     - Recovery Force = 10 mN
     - For = 1.0 min
     - Static Control = Position
     - Static Set point = Current
   - Temperature Program
     - Temp 1 = 30°C
     - Time = 22.5 min
     - End Condition
   - Modify Display Parameters
   - Modify Data/Atmosphere Parameters

4. Why is the temperature program time 22.5 min?

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5. Wait till the system reaches equilibrium.

6. Read height again.

7. Start Run with static control off

8. Modify the display during the run.

9. After the run, save the data file.

10. Analyze the data.

11. Create a table.

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DMA LABORATORY.
Experiment #6

Parallel Plate
Creep-Recovery Mode
Septum

OBJECTIVE: This laboratory experiment will allow the student to become more familiar with:

- o Set-up for making a run
- o Sample handling
- o Creating a method using creep ramp
- o Making a run
- o Data handling

DIRECTIONS: Read the entire question or instruction before responding.

1. What 2 variables should be checked before beginning a run?

   #1_____

   #2_____

2. Measure diameter of septum _____

3. Zero probe

4. Install sample and read sample height

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4. Set-up and run a parallel plate experiment as follows.

   - Exit
   - Run system
     - Set-up
   - DMA Program
     - Creep Ramp = ON
     - Hold For = 1.0 min
     - Creep Force = 2500 mN
     - Creep Rate = 500 mN/min
     - For = 5.0 min

5. Press hard key, go to temp and go to 30°.

6. Temp Program
   - Temp = 30
   - Time = _____

   (draw a sketch of the cycles you want)

7. Wait till the system reaches equilibrium.

8. View status line. Are your ready to run? _____

9. Start Run.

10.. Modify the display during the run.

11. What information is displayed in the red block on upper left of monitor?

    _____

12. After the run, save the data file.

13. Repeat this experiment with creep and recovery for 5 min. (Temperatures: 30, 40, 50)

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14. Analyze the data.

15. Create a table.

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