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MECHANICAL PROPERTIES – VISCOELASTIC METHODS

Part I: Creep, Relaxation, and Stress-Strain Methods

by

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U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

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1. INTRODUCTION

Since many restorative as well as natural dental materials have
deformation characteristics which are time-dependent as well as stress-
dependent, viscoelastic theory and methods offer the most advantageous
means for description and characterization of their mechanical behavior.
The viscoelastic description has numerous advantages: (1) it enables
researchers to describe analytically and to predict the mechanical be-
havior of these materials both in laboratory experiments and in the
mouth; (2) it enables one to separate and quantitatively describe the
relative contribution of the various time-dependent and non-time-
dependent mechanical responses occurring simultaneously in a material;
(3) it furnishes a unified theory whereby the mechanical behavior of
time-dependent materials can be compared with non-time-dependent mater-
ials used for the same purpose, such as the different types of anterior
restorative materials; (4) it enables one to compare mechanical response
of a material under one test condition to that under another test in a
quantitative manner by appropriate transformation equations; (5) it en-
ables one to select a test or group of mechanical tests that will fully
measure the various mechanical phenomena occurring in the material and
evaluate the mechanical parameters of each; and (6) it isolates individ-
ual mechanical phenomena and their parameters at the macroscale level
and together with micro viscoelastic theory, permits better description
of the relation of mechanical response to microstructure.

Both static and dynamic test methods have been used to investigate
the viscoelastic properties of dental materials. These test methods may
be interrelated by appropriate analytical and viscoelastic theory. Common-
ly employed static tests are classified as: (1) a constant strain test,
such as a creep test, (2) a constant stress test, such as a stress relaxa-
tion test, or, (3) a test where the stress and strain are both varied

slowly and in some cases cycled[*]; an example is the classical stress-
strain test. All of the above types of static testing, including
spherical indentation test, have been employed in dental research
and testing. Dynamic test methods may be generally classified as fol-
lows: (1) free vibrational methods, (2) forced vibrational methods,
both resonance and non-resonance, and (3) propagation methods either
using pulses or continuous waves. In many cases, the same dynamic ap-
paratus may determine the viscoelastic properties of a material by using
two or more of the above methods. Whether the method be static or dynam-
ic, the following factors should be considered when testing a material:

(1)   the mechanical information obtained about the material;

(2)   how this mechanical information on the material tested
      may be interrelated to that obtained for the material
      using other methods; and

(3)   how the mechanical phenomena and their parameters obtained
      for the material may be directly and/or indirectly related
      to the microstructure of the material.

2. CREEP OF LINEAR VISCOELASTIC MATERIALS

The creep test as a method of investigation of viscoelasticity of a
material has the advantage of simple instrumentation, but the disadvan-
tage of the long testing time required for those materials having retar-
dation times that extend over a long time scale, as well as the insensi-
tivity to the retardation behavior of the material in the initial short
portion of the experimental time scale. A creep test is normally con-
ducted on the material in the form of a specimen having a uniform cross-
sectional area. A constant load is applied either in tension, compression
or shear, and the deformation is measured in the direction of load appli-
cation as a function of time. The deformation may be detected by such de-
vices as a cathetometer, strain gauge or differential transformer, where
length changes may be measured as a function of time for periods of less
than one second to many years, if necessary. Creep curves for the material

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[*]   It becomes difficult to separate static stress-strain testing
      classification wise from dynamic testing when cycling occurs.

are obtained for different stresses, and then deformation behavior of
the material as a function of time and stress is extracted from the
family of creep curves at different stresses.

The creep curve of a strain-hardened specimen of a material con-
sists commonly of one of three phenomena, or some combination of these,
each of which may or may not be a linear function of the applied stress;
while as a function of time, one is independent, one linear and one non-
linear. The three deformation or strain phenomena are:

(1) instantaneous elastic strain ε_I, described by analogy to a
spring having a compliance J_o, or a modulus G_o, where J_o =
$1/G_o$,

(2) viscous strain ε_v which may be described as analogous to a
dashpot when linear, having a coefficient of viscosity η equal
to the applied stress divided by the strain rate, and

(3) retarded elastic strain ε_r usually described by analogy to
the so-called Voight element or series of Voight elements, the
components of which consist of a spring of compliance J_R, or
modulus G_R in parallel with dashpot having a coefficient of
viscosity η_R, where the retardation time τ of the Voight element
is defined as $\tau = \eta_r J_R$.

If a material exhibits a linear combination of all three types of strain,
$\varepsilon = \varepsilon_I + \varepsilon_r + \varepsilon_v$, it may be represented by a spring of compliance J_o
(instantaneous behavior) and a dashpot having a coefficient of viscosity
η (viscous behavior) in a series with one or more Voight elements (retarded
elastic strain). The graphical behavior of each of the three types of
strain, along with its accompanying model and corresponding analytical
strain function, where the strain is assumed to be a linear function of
the stress σ, may be seen in Figures Ia, b, and c. The combination of
the three types of strain is shown in Figure Id. When the strain is a
linear function of the stress, the data obtained at different stresses
may be reduced to a single creep curve by plotting the creep compliance
$[J(t) = \varepsilon(t)/\sigma_o]$ as a function of time or logarithmic time. The creep
compliance $J(t)$ of the combination of the three types of phenomena versus

time and the logarithm of time may be seen in Figures II-a and b. When
the retarded elastic creep compliance has a continuous distribution of
retardation times τ_r, the combination creep compliance $J(t) =$
$J_I + J_v + J_R$ may be described by the following analytical equation:

$$J(t) = J_o + t/\eta + \int_o^\infty J(\tau)[1-e^{-t/\tau}]d\tau \tag{1}$$

as shown graphically in Figure IIa. Upon substitution of $L(\tau)/\tau$ for
$J(\tau)$ in Equation (1), the following is obtained

$$J(t) = J_o + t/\eta + \int_{-\infty}^{+\infty} L(\tau)[1-e^{t/\tau}]d\ln\tau \quad . \tag{2a}$$

Next, the subtraction of t/η from both sides of Equation (2a) results in

$$J(t) - t/\eta = J_o + \int_{-\infty}^{+\infty} L(\tau)[1-e^{t/\tau}]d\ln\tau \tag{2b}$$

where

   $J(t)$ is the creep compliance

   J_o is the instantaneous elastic compliance

   t/η is the viscous response where t is the time after application
         of the stress σ and η is the coefficient of viscosity

   $\int_o^\infty J(\tau)(1-e^{-t/\tau})d\tau$ or $\int_{-\infty}^{+\infty} L(\tau)[1-e^{-t/\tau}]d\ln\tau$ are analytical forms

         of the retarded elastic response with $J(\tau)$ and $L(\tau)$ being forms
         of the retardation spectrum of the material.

The graphical representation of Equations (2a) and (2b) is seen in
Figure II-b. It might be mentioned that $J(t) - t/\eta$ represents the
elastic portion (instantaneous plus retarded) of the creep compliance
curve in a constant stress experiment and also represents, after appro-
priate transformation, the recovery portion of the curve in Figure II-a
when the stress has been removed. Plots of creep compliance $J(t)$ or
$(J(t)-t/\eta)$ versus lnt rather than versus t for a linear viscoelastic
material, have the advantages of more direct utilization - (1) in calcu-
lating the retardation spectrum $L(\tau)$ by various approximation methods as
well as giving a better visual picture of the distribution of retardation
times τ_r for the material, and (2) in application of the time-temperature

superposition principle[1,2,3] to a linear viscoelastic material to obtain its creep compliance behavior at a given temperature that otherwise would require experimental data over many decades at that temperature. Therefore, a creep curve may be obtained at a specific temperature T for times outside the range of practical observation at this temperature by obtaining creep curves at higher and lower temperatures within the time scale of the creep experiment, and then shifting the higher and lower temperature creep curves for the linear material along the axis of the logarithmic time scale until they all join into a continuous master curve for the temperature T. Before the experimental creep curves can be shifted to make the master curve at temperature T, the compliance values should in theory have a density (or specific volume) correction, but, in practice, the density changes with temperature are often small enough to be neglected. The amount the curves are shifted along the ln time axis is described by the equation:

$$(\ln t - \ln t_o) = \ln t/t_o = \ln A_T ,$$

where the shift is said to be positive when the curve is shifted to shorter times (to the left on the log time scale) in forming the master curve. The time-temperature superposition principle is not only applicable to creep data, but to stress relaxation data as well as dynamic mechanical testing data where the A_T values obtained from the shift on the ln time scale or $\ln 1/\omega$ (reciprocal frequency) scale in the case of dynamic measurement - for these different methods of testing are equivalent. The theory and application of the time-temperature superposition principle to a linear viscoelastic material, as well as the theoretical significance of A_T for the material, will be discussed in a later part of this paper.

3. STRESS RELAXATION OF LINEAR VISCOELASTIC MATERIALS

Stress relaxation behavior is observed in viscoelastic materials; that is to say, the stress in the material relaxes or decreases with time when the material is deformed quickly and the deformation is held constant. Stress-relaxation tests require slightly more complex instrumentation than creep tests in that, in addition to requiring a device for detecting deformation, a load measuring device is required in order to follow the force

change with time. Like the creep test, the stress-relaxation test has
the disadvantage of the long test time required for some materials as
well as the lack of sensitivity to relaxation behavior exhibited by
the material in the initial short portion of the time scale. Stress-
relaxation data are generally more directly interpreted in terms of
viscoelastic theory than are creep data.   A tensile stress-
relaxation device often consists simply of two clamps between which the
specimen is attached; the upper clamp is usually attached to a load
detecting cell which is rigidly attached to a frame; the lower clamp can
be adjusted up or down in respect to the fixed upper clamp to obtain
various deformations in the specimen. Once the lower clamp has been ad-
justed to obtain the desired deformation value in the specimen, the
clamp is then held fixed in respect to the frame. The amount of deforma-
tion in the specimen can be detected by means of a strain gauge, differ-
ential transformer or cathetometer. The stress relaxation curves are
obtained at different deformation levels, thus the stress-relaxation as
a function of time and deformation is obtained for the material from the
family of stress-relaxation curves at different deformations. The
stress is plotted as a function of linear time or of logarithmic
time.   If stress is plotted as function of strain for a common
time value from each of the family of curves for various deformations,
this will demonstrate whether the stress and strain bear a linear relation
to each other for the material. If nonlinear, the stress-strain curves
for the fixed time values will yield the functional relationship between
the stress and the strain. If the material exhibits a linear relationship
between the stress and the strain, then, if the stress values for each
curve   are   divided by the corresponding fixed strain value for that
stress relaxation curve and the resulting relaxation modulus $G(t) = \sigma(t)/\varepsilon_o$
is plotted for each of the curves, the family of stress-relaxation curves
should reduce to a single master modulus relaxation curve. If the mater-
ial is linear in its stress-strain behavior at a fixed time, the usual
curves plotted are relaxation modulus curves either as a function of
linear time or logarithmic time. The simplest analog to describe stress-
relaxation behavior is the Maxwell model which is a series combination of

a spring of modulus G_o and a linear dashpot having a coefficient of viscosity η. The differential equation of motion of a material described by a single Maxwell model is

$$\frac{d\epsilon}{dt} = \frac{\sigma}{\eta} + \frac{1}{G_o} \frac{d\sigma}{dt} \tag{3}$$

where σ and ϵ are stress and strain, respectively. In the normal stress relaxation test as mentioned previously, the strain is held constant after initial rapid deformation. Hence, in the above differential equation, $\frac{d\epsilon}{dt}$ becomes zero for a constant strain test. Therefore, for a constant strain test, the above differential equation has a solution of the following form:

$$\sigma(t) = \sigma_o e^{-\frac{G_o t}{\eta}} = \sigma_o e^{-t/\tau} \tag{3a}$$

A material that can be described by a Maxwell model is said to have a single relaxation time defined as $\tau = \eta/G_o$. Such a material is shown graphically by a plot of stress as a function of linear time, logarithmic time and also by a plot of relaxation modulus (stiffness) $G(t) = \sigma(t)/\epsilon$ as a function of logarithmic time in Figures III-a, b and c. These plots are accompanied by the Maxwell model diagram and corresponding analytical equations. As previously mentioned, there is the lack of initial sensitivity to stress-relaxation behavior of certain materials as a consequence of the finite time required for application of the fixed strain (rather than idealized instantaneous application of the strain). This lack of initial stress-relaxation sensitivity can be discussed in terms of a material which can be represented by a single Maxwell element. If such a material has a short relaxation time, τ, or expressed another way, the stress-relaxation time scale is short compared with the time required for application of the strain, the instantaneous maximum stress and corresponding limiting modulus $G_o = \sigma_o/\epsilon$ are indeterminant. The result of this can be seen for a simple Maxwell material represented by the dotted line in Figure III-a.

Most materials, including dental materials, cannot be characterized by a single Maxwell model having a single relaxation time. These materials

have more than one relaxation time and, in many cases, so numerous are the relaxation times that they can be treated as continuous in their distribution, the continuous function $H(\tau)$ being called the relaxation spectrum. When the relaxation modulus $G(t)$ for a linear viscoelastic material is plotted against logarithmic time (over an extended time range), most often a decreasing sigmoidal shape curve is obtained, as illustrated in Figure IV. In Figure IV, the limiting short time value of the relaxation modulus is called the glass modulus G_o, while the value of $G(t)$ at infinite times approaches an equilibrium modulus value G_e or zero, depending on the microstructure of the material. The difference between the glass modulus G_o and the equilibrium modulus G_e is defined as the decay modulus $G_R = G_o - G_e$. For example, in the case of amorphous polymer, the value of G_e would depend on whether the polymer was crosslinked or not. The crosslinked polymer would exhibit a value G_e at infinite time, but the non-crosslinked would approach zero at infinite time. Commonly, even though $G_e = 0$ for non-crosslinked polymers, an intermediate plateau is observed, the length of which is molecular weight dependent. Since most materials must be described by a finite number of relaxation times or a continuous relaxation spectrum, the modulus-relaxation behavior shown in Figure IV may be described by one of the following equations. In the case of a finite number of relaxation times, the relaxation modulus equation takes the form

$$G(t) = \sum_i G_i e^{-t/\tau_i} + G_e \quad . \tag{4}$$

In the case of continuous distribution of relaxation times, the relaxation modulus equation takes the form for a linear material

$$G(t) = \int_0^\infty G(\tau) e^{-t/\tau} d\tau + G_e \tag{5}$$

or, upon substituting $G(\tau) = H(\tau)/\tau$, the equation becomes

$$G(t) = \int_{-\infty}^{+\infty} H(\tau) e^{-t/\tau} d\ln\tau + G_e \quad . \tag{6}$$

The monotonic decrease of $G(t)$ from G_o to G_e, as shown in Figure IV, can be described by Equation (6). It is noted that the sigmoidal

relaxation modulus G(t) curve in Figure IV roughly approximates the mirror image of the creep compliance J(ε) plot shown in Figure II, both plots being on a logarithmic time scale. As recalled from earlier in the discussion, the glass modulus and glass compliances are related as $G_o = 1/J_o$, as well as the equilibrium modulus G_e and steady-state compliance J_e are reciprocally related. However, the relaxation modulus G(t) and the creep compliance function J(t) are reciprocally related only at the limiting values discussed in the previous sentence, and, therefore, their curves on a logarithmic time scale are not true mirror images for a given linear viscoelastic material. It has been shown by Gross,[4] and more specifically by Leaderman,[5] that the two functions G(t) and J(t) are related by a reciprocal relationship between their respective Laplace transforms:

$$pL[J(t)] = 1/pL[G(t)] \qquad (7)$$

where the Laplace transform is defined as follows:

$$L[f(t)] = \int_o^\infty e^{-Pt} f(t)dt$$

While Equation (7) relates the two functions J(t) and G(t), there is difficulty in obtaining one function from the other by means of Equation (7) due to the obstacles involved in the inversion of the Laplace transform. More often the functions G(t) or J(t) are given as empirically determined in data form, thus, numerical inversion is required, which is only an approximation; but, even if the analytical function is known for G(t) or J(t), it may not be possible to find the inverse. One of the most severe drawbacks in using the above equation is that more often either of the two functions has been determined only over a limited time scale.

The logarithmic time plot for relaxation modulus G(t) illustrated in Figure IV, as in the case of creep compliance versus ln(t), has the same advantages of direct use: (1) in determining the relaxation spectrum H(τ) by some approximation methods and by virtue of which gives a qualitative visual picture of the distribution of relaxation times $τ_r$, and (2) in applying the time temperature superposition principle to the relaxation

modulus data for a linear material. Again, as in the case of creep
compliance data, if the relaxation modulus G(t) data are obtained at
various temperatures above and below some reference temperature T,
these higher and lower temperature modulus curves may be shifted along
the log time axis until they form a master curve at the reference
temperature T. The amount of shift again being described by $(\ln t - \ln t_o) =$
$\ln t/t_o = \ln A_T$, where the A_T values obtained by the shifts of the re-
laxation data are equivalent to those obtained from the shift of the
creep compliance data on the same linear viscoelastic material - this
result is encompassed in the theory of the time-temperature superposi-
tion principle.

4.   BOLTZMAN SUPERPOSITION PRINCIPLE - RELATION TO STRESS-STRAIN BEHAVIOR

Recalling the definition of creep compliance, $J(t) = \varepsilon(t)/\sigma$ dis-
cussed earlier, it has been noted that in a linear viscoelastic material,
when a single stress σ is applied at time θ to a specimen which has no
previous stress history, the strain in the specimen at any time t is
related to the applied stress by the following relationship:

$$\varepsilon(t) = \sigma J(t - \theta). \tag{8}$$

Next, consider a series of stress increments σ_i applied at times θ_i prior
to the time t; now the strain $\varepsilon(t)$ at time t would be related to the stress
increments σ_i by the following equation:

$$\varepsilon(t) = \sum_{-\infty}^{t} \sigma_i J(t - \theta_i) \tag{9}$$

Thus, the above finite relationship relates the strain $\varepsilon(t)$ at time t
to all the previous stress history by way of the creep compliance J.
The above equations are finite forms of the Boltzmann superposition
principle. These equations are useful in describing step-function ex-
periments. For example, consider the creep experiment described by
Figure II-a where the stress has been introduced and removed in a step-
wise manner and the stress σ was maintained over a sufficient time for
a steady state condition to be reached. If the time of removal of the

stress σ is considered $t = 0$, then the stress σ was applied at a time $-t_L$. Since $t = 0$ at load removal, the strain at the instant before load removal is designated $\epsilon(0)$ and is related to its elastic, retarded elastic and viscous compliances as follows:

$$\epsilon(0) = \sigma(J_o + J_R + t_L/\eta) \tag{10}$$

At a time t after stress removal $(-\sigma)$ as shown in Figure II-a, the strain $\epsilon(t)$ is related to the two incremental stresses σ and $-\sigma$ as follows:

$$\epsilon(t) = \sigma[J_o + J_R + \frac{(t_L + t)}{\eta}] - \sigma J(t). \tag{11}$$

Now, substituting $\epsilon(0)$ for $\sigma(J_o + J_R + t_L/\eta)$

$$\epsilon(0) - \epsilon(t) = \sigma[J(t) - t/\eta] \tag{12}$$

which is the transformation mentioned earlier for the unloaded portion of the curve.

If the stress increments are not introduced in steps, but in a continuous manner with time, the strain function becomes a strain integral in accordance with the superposition principle taking the following form:

$$E(\epsilon) = \lim_{\Delta\sigma \to 0} \sum_{o}^{\sigma^t} J(t - \theta_i)\Delta\sigma_i = \int_{o}^{\sigma^t} J(t - \theta)d\sigma(\theta) \tag{13}$$

or by the use of the chain rule and where the entire stress history from $\theta \to -\infty$ is considered, the above equation takes the following form:

$$E(t) = \int_{-\infty}^{t} J(t - \theta)\frac{d\sigma(\theta)}{d\theta} \, d\theta \tag{14}$$

The Boltzmann superposition principle in the form of Equations (13) and (14) specifies the transformation relationship (for a linear visco-elastic material) between the creep experiment by way of the creep compliance, and the stress-strain experiment where the stress time relation is prescribed, on the same material. Similarly, the Boltzmann principle can be applied to the determination of stress as a function of stepwise introduction of strain increments leading to the equation:

$$\sigma(t) = \sum_{-\infty}^{t} G(t - \theta_i)\epsilon_i \tag{15}$$

where G is the relaxation modulus defined earlier, thus relating the stress to prior strain history. When the strain increments are introduced in a continuous manner, the above equation becomes a stress integral as follows:

$$\sigma(t) = \int_{-\infty}^{t} G(t - \theta) \frac{d\varepsilon(\theta)}{d\theta} d\theta \qquad (16)$$

relating the total stress to the strain history.

The superposition Equation (14) is the transformation equation between the creep function obtained in a creep experiment on a linear viscoelastic material and the strain function (strain integral) in a stress-strain experiment on that same material, where the stress is known as a point by point function of experimental time. Also, the superposition Equation (16) is the transformation equation between a stress relaxation experiment and a stress-strain experiment on a given material where the strain is known in a point by point relation to experimental time. For the case of a constant stress rate, $(\frac{d\sigma(\theta)}{d\theta} = \dot{\sigma} = a$ constant), substitution of the continuous spectrum form of the creep function given by Equation (2a),

$$J(t) = \int_{-\infty}^{+\infty} L(\tau)[1 - e^{t/\tau}]d\ln\tau + t/\eta \quad ,$$

in Equation (14) followed by differentiation results in

$$\frac{dE(t)}{d\sigma} = \frac{1}{\dot{\sigma}} \frac{d\varepsilon}{dt} = J(t) \qquad . \qquad (17)$$

Thus, the slope of the strain-stress curve in a constant stress rate experiment is the creep function $J(t)$ for a linear material where the time is given by $t = \sigma/\dot{\sigma}$ since $\dot{\sigma} = a$ constant. Looking at the constant strain rate case for a stress-strain experiment, by substituting Equation (6) into the superposition Equation (16), and assuming $\frac{d\varepsilon(\theta)}{d\theta} = \dot{\varepsilon}$ (a constant), one obtains the result

$$\sigma(t) = \dot{\varepsilon}[\int_{-\infty}^{+\infty} \tau H(\tau)(1 - e^{-t/\tau})d\ln\tau + G_e t] \qquad (18)$$

which relates the stress integral $\sigma(t)$ to the relaxation spectrum $H(\tau)$ and thus to the relaxation modulus function of the relaxation experiment in a simple manner since for the constant stress rate experiment, $t = \varepsilon/\dot{\varepsilon}$; thus stress versus strain is equivalent to a stress-time plot.

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- 14 -
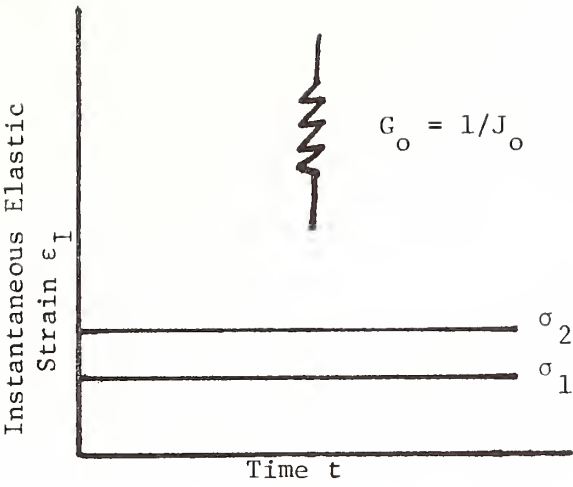
(a) Instantaneous Strain
$$\varepsilon_I = J_o \sigma$$
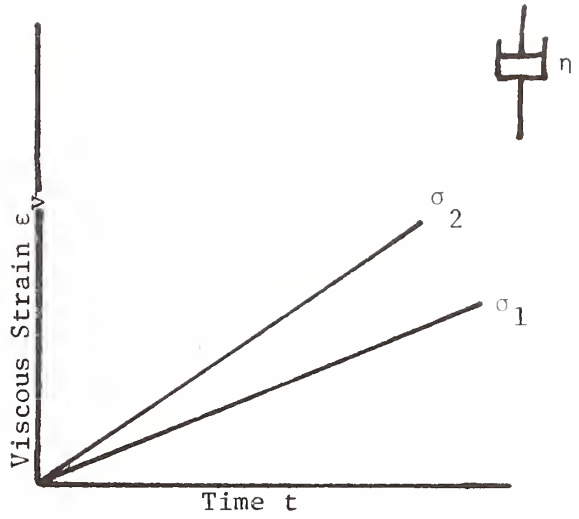
(b) Viscous Strain
$$\varepsilon_v = \frac{\sigma t}{\eta}$$
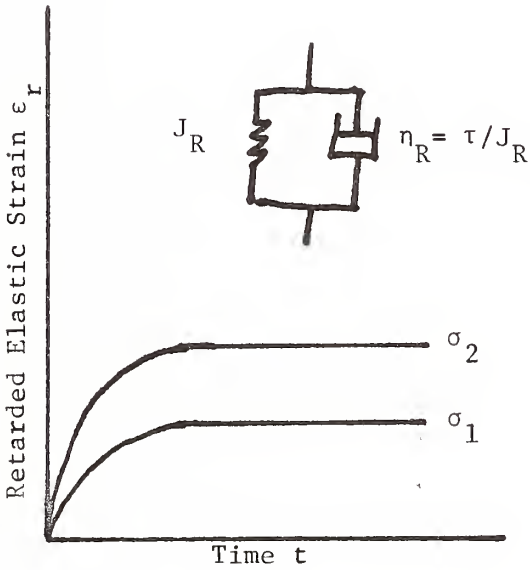
(c) Retarded Elastic Strain
$$\varepsilon_r = \sigma J_R (1 - e^{-t/\tau})$$
or
$$\varepsilon_r = \sigma \sum_{i=1}^{n} J_{R_i} (1-e^{-t/\tau_i})$$
or
$$\varepsilon_r = \sigma \int_o^\infty J_R(\tau)(1-e^{-t/\tau_i})d\tau$$
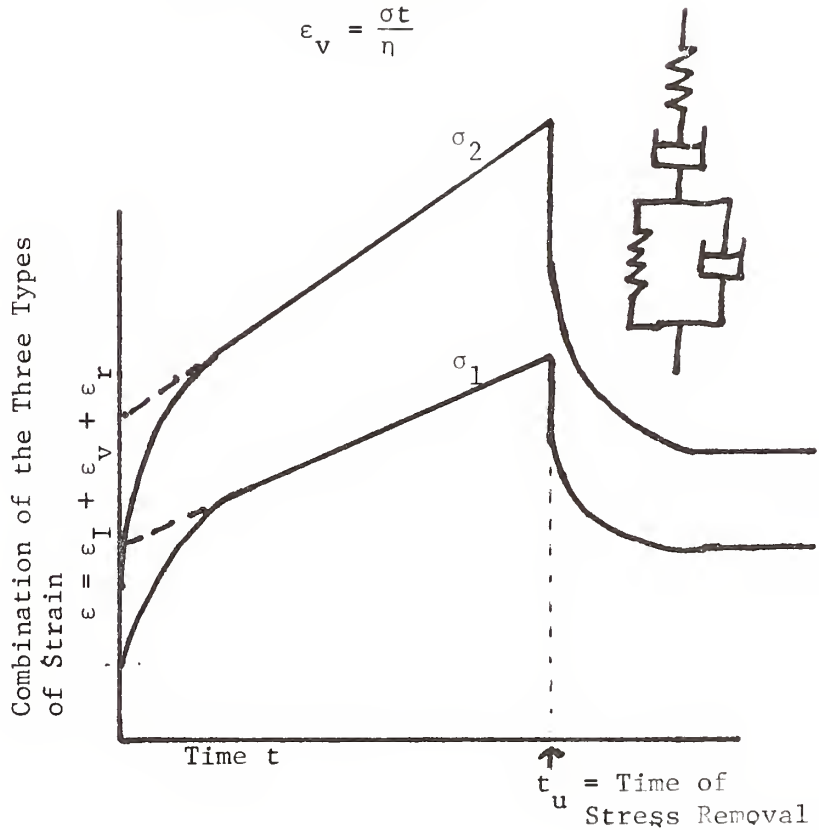
(d) Combination of Three Types of Strain
$$\varepsilon = \varepsilon_I + \varepsilon_v + \varepsilon_r = J_o\sigma + (\sigma t)/\eta + \sigma J_R(1-e^{-t/\tau})$$
or
$$\varepsilon = J_o\sigma + (\sigma t)/\eta + \sigma \sum_{i=1}^{n} J_{R_i}(1 - e^{-t/\tau_i})$$
or
$$\varepsilon = J_o\sigma + (\sigma t)/\eta + \sigma \int_o^\infty J_R(\tau)(1 - e^{-t/\tau})d\tau$$

Figure I

(a)

(b)

Figure II

MAXWELL MODEL (⟨spring⟩G_o ⟨dashpot⟩η)

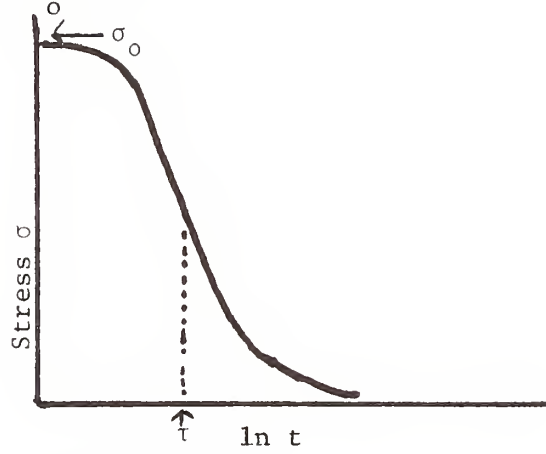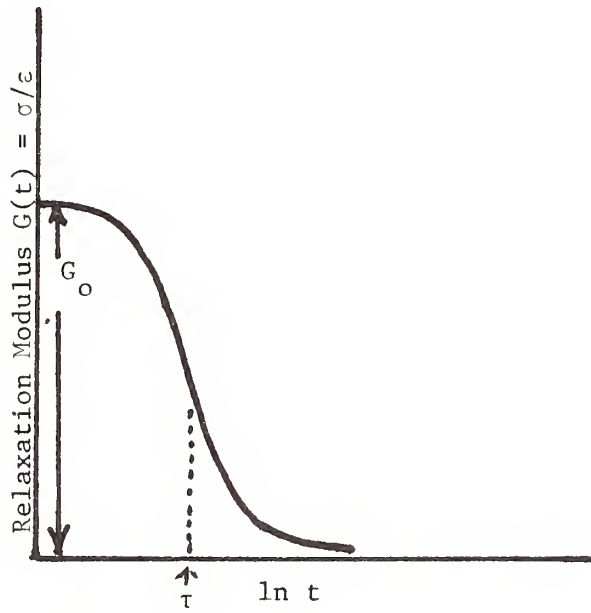$$\sigma(t) = \sigma_o e^{-t/\tau}$$

or
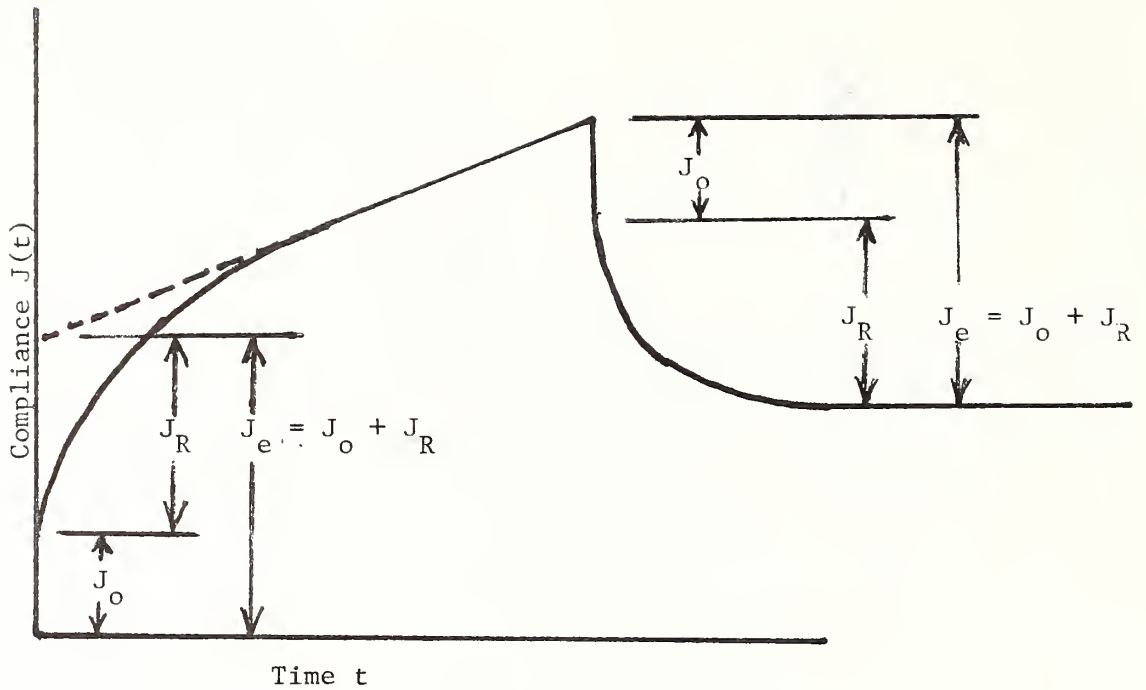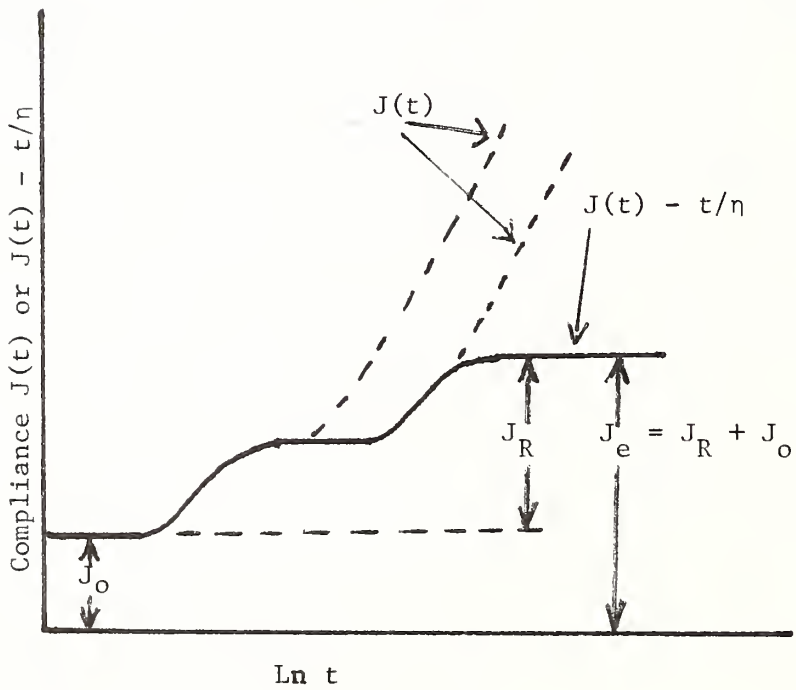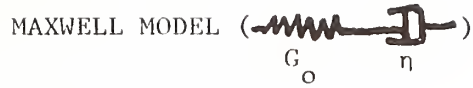
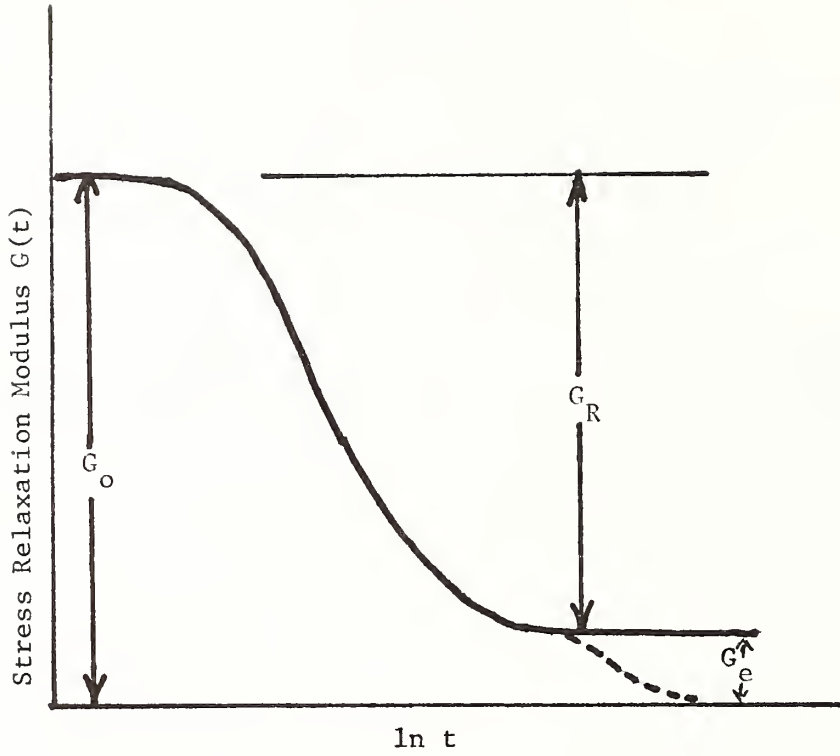$$G(t) = \sigma(t)/\varepsilon = G_o e^{-t/\tau}$$
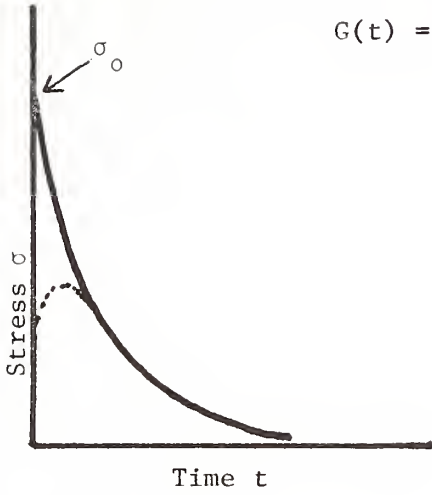
(a)

(b)

(c)

Figure III

Figure IV

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