

GLASS

SCIENCE

AND TECHNOLOGY

Edited by

D. R. UHLMANN AND N. J. KREIDL

VOLUME 3

Viscosity and Relaxation

GLASS: SCIENCE AND TECHNOLOGY

Edited by D. R. UHLMANN

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VOLUME 3

Viscosity and Relaxation

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Preface

Glass: Science and Technology of which this volume is the fourth to appear after Volumes 5 (Mechanical Properties), 1 (Glass Forming Systems), and 2 (Processing), provides a central, convenient, and reasonably detailed reference source to the notable developments in the field of noncrystalline solids (glasses).

Flow processes as well as relaxation processes have become the subject of an ever increasing number of widely scattered investigations by glass scientists and engineers. Time-dependent phenomena not only reveal details about the nature of glass and the glass transition but also dramatically affect the engineering properties of glass products. This volume of *Glass: Science and Technology* attempts to provide an overview of the scientific and engineering aspects of viscosity, viscoelasticity, relaxation, and annealing. At the same time, in order to accommodate significant phenomenological and technological differences between classes of materials, separate chapters deal with inorganic nonmetallic, organic polymer, and metal glasses.

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CHAPTER 1

Viscoelasticity of Glass

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List of Symbols

Some symbols used in this chapter have several different meanings, but the context will clearly identify them. Some other symbols used for specific applications are not included in the list.

a	A constant
b	As subscript, biaxial
b	A constant
c	A constant
e or e_{12}	Shear strain
$[e]$	Matrix of shear strain tensor
e_f	Fictive strain
e_{fi}	Partial fictive strain
f	As subscript, fictive
g	As subscript, glassy state
l	As subscript, liquid state
p	Laplace transform parameter or glass property
q	Strain rate ($= \dot{\epsilon}$) or stress rate ($= \dot{\sigma}$)
s	Shear stress or relaxation frequency
s	As subscript, shear
s_{12}	Shear stress
s_f	Fictive stress
s_{fi}	Partial fictive stress
t	Time
t_m	Time of the maximum in a crossover experiment
u	As subscript, uniaxial
u_i	Weighting factor for structural retardation function
v	As subscript, denotes a volume property
v_i	Weighting factor for shear retardation function

w_i	Weighting factor for shear relaxation function or dynamic moduli
x	Nonlinearity factor in Four-Parameter Model
y_i	Weighting factors for uniaxial and biaxial stress relaxation functions
A	Constant in four-parameter model
C	Heat capacity
C_p	Heat capacity at constant pressure
D	Deborah number
E	Young's Modulus
F	Distribution of relaxation times
G	Gibbs free energy, shear modulus
G_1	Shear relaxation modulus
G'	Shear storage modulus or elastic modulus of Kelvin-Voigt model
G''	Shear loss modulus
G_2	Bulk relaxation modulus
H	Enthalpy, Hookean element, or shear relaxation spectrum
H_2	Bulk relaxation spectrum
J	Compliance
J_1	Creep shear compliance
J_{1E}	Shear compliance
J_{1D}	Shear delayed-elastic compliance
J_2	Bulk compliance
K	Bulk modulus
K_1	Bulk moduli of Pointing-Thomson model
K_2	
K'_1	
K'_2	
$L_2(\lambda_B)$	Spectrum of bulk retardation times
M	"Apparent" elastic modulus
M_p	Structural retardation function
N	Newtonian element, distribution function, "apparent" Poisson ratio
P	Pressure
P_f	Fictive pressure
R	Error term or ideal gas constant
S	Entropy, crossover experiment function
T	Temperature
T_g	Glass transition temperature
T_m	Melting temperature
T_r	Reference temperature
U	Displacement
V	Volume
V_k	Weighting coefficient for bulk retardation function
W	Work
W_k	Weighting coefficient for bulk relaxation function
Z_1	Loss function
α	Thermal expansion coefficient
α_p	Temperature derivative of property p
$\alpha_{p/l}$	Same as above for liquid
$\alpha_{p/g}$	Same as above for glass

α_{ps}	Structural part of α_{pl}
γ	Shear strain
δ	Partial derivative, Kronecker delta function or phase angle
ϵ	Component of strain tensor (if with subscripts), hydrostatic or dilatational strain
$[e]$	Matrix of strain tensor
ξ	Order parameter
η	Shear viscosity
η'	Viscosity of Kelvin-Voigt model
η_v	Volume viscosity or viscosity of Poynting-Thomson model
η'_v	Volume viscosity of Poynting-Thomson model
η_1	Viscosity of Burgers model
κ	Compressibility
λ	Shear or structural retardation time
λ_B	Bulk retardation time
$\bar{\lambda}_B$	Average bulk retardation time
$\bar{\lambda}_s$	Average shear retardation time
λ_{si}	Component of discrete spectrum of shear retardation times
λ_{Bk}	Same as above for bulk retardation
μ	Alternate shear stress relaxation modulus
ν	Poisson's ratio, frequency in radians/s
ξ	Reduced time
σ	Component of stress tensor (if with subscripts), or hydrostatic or dilatational stress
$[\sigma]$	Matrix of stress tensor
τ_s	Shear stress relaxation time
ϕ_1	Shear retardation function
ϕ_2	Bulk retardation function
Ψ	Relaxation function

I. Introduction

Viscoelasticity describes a time-dependent response of liquids and solids to either mechanical stress or strain. Temperature is the third major independent variable because it produces voluminal or dilatational strain and strongly affects the viscoelastic properties of the material. Many solids (and even liquids under superfast influences) appear as nearly perfect elastic bodies and are studied by means of elasticity theory. In steady-state conditions of flow, the liquids behave as incompressible fluids and are studied by means of fluid mechanics. It is the transient, time-dependent phenomena on the border between the solid- and fluidlike behavior that are the subject of viscoelasticity studies. Christensen (1982) offered a comprehensive review of the theory of viscoelasticity. A more limited task of this chapter is to review the viscoelastic behavior of glasses.

The viscoelastic approach is pertinent to the very essence of the glassy state, because the glass is obtained via the liquid/solid or liquid/glass transition.

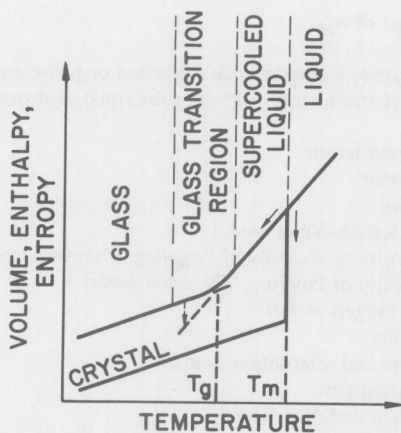


FIG. 1. Temperature dependence of properties of liquid, glass, and crystal.

Figure 1 shows schematically the change in volume V , enthalpy H , and entropy S of a liquid during cooling. For substances that can exist in both glassy and crystalline states, there are two paths available below the melting temperature T_m . By using the definition from classical thermodynamics $\partial G/\partial T = -S$, where G is the Gibbs free energy, we can derive from Fig. 1 the plot shown in Fig. 2. Similarly, a plot of G versus pressure P can be derived from the definition $\partial G/\partial P = V$. It follows from Fig. 2 that, in order to form glass, the liquid must make two thermodynamically unfavorable choices: one at the melting temperature T_m and another at the glass transition temperature T_g . In both cases, the liquid must follow the path with higher free energy. In both cases, the reason for this behavior lies in kinetics.

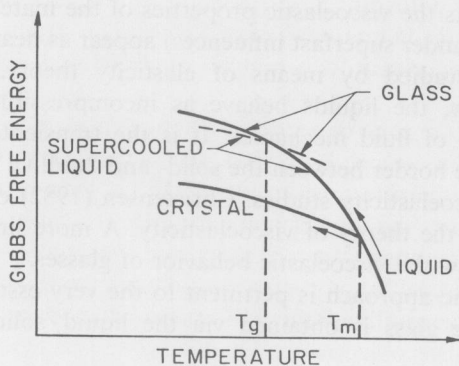


FIG. 2. Temperature dependence of the Gibbs free energy for liquid, glass, and crystal.

At temperature T_m , the crystallization is impeded by the finite viscosity of the liquid and therefore takes some characteristic time t^* . If rapid cooling makes the dwell time at T_m shorter than the time t^* , crystallization will be kinetically arrested. Inorganic glass-forming melts have a high viscosity at T_m and can be cooled slowly. In contrast, molten metals require cooling rates on the order of $10^5^\circ\text{C}/\text{sec}$ to form glass.

A supercooled liquid is in thermodynamic equilibrium with respect to any immediately neighboring structure. It is in a metastable equilibrium with respect to a crystalline solid. The free energy profile can be pictured as one with a minimum for the crystalline state and a "trough" above the minimum for the supercooled liquid state. The very considerable height of the "trough" explains why many metastable states can persist almost indefinitely under suitable conditions. The properties of a supercooled liquid are uniquely defined by two external thermodynamic parameters: temperature and pressure. Below T_g the material, a glass, is *not* in equilibrium. Its structure depends on thermal history and therefore one more parameter—the internal or order parameter—is needed to characterize the glassy state.

The nonequilibrium state of glass is securely frozen by a viscosity 10^{15} – 10^{20} times greater than that of ordinary liquids. This is why glass is often called a frozen liquid. It is a liquid because the phase transformation (crystallization) has never occurred; it is a frozen liquid because it has all the qualities of a solid. Curiously, the liquid is never completely frozen. Even at temperatures below T_g , the volume of the glass will decrease slowly in the direction of its equilibrium value. This process, called stabilization, evolves at room temperature on a geological time scale and can be observed only with the help of a sensitive instrument. For example, the glass bulb of a thermometer contracts slowly, causing the zero point to rise. A mechanical time-dependent response can be conveniently observed at room temperature, using a glass spiral. Figure 3 shows the delayed-elastic strain resulting from loading and unloading a large spiral of the soda-lime-silicate glass with the glass transition temperature of 530°C . The structural and mechanical stability is quite critical for a number of communication, electronic, and measuring devices.

Conveniently, the time-dependent structural and viscoelastic behavior is observed in the glass transition region (see Fig. 1). However, it can also be demonstrated at temperatures up to 1200°C . Absorption of ultrasonic waves clearly reveals the viscoelastic nature of glass-forming liquids. Some of the glass-forming operations are so intense that they force a molten glass to respond in a viscoelastic, or even elastic, manner.

Above we have outlined the subject and the scope of a viscoelastic study. What are the objectives? We deal with this question in the next section.

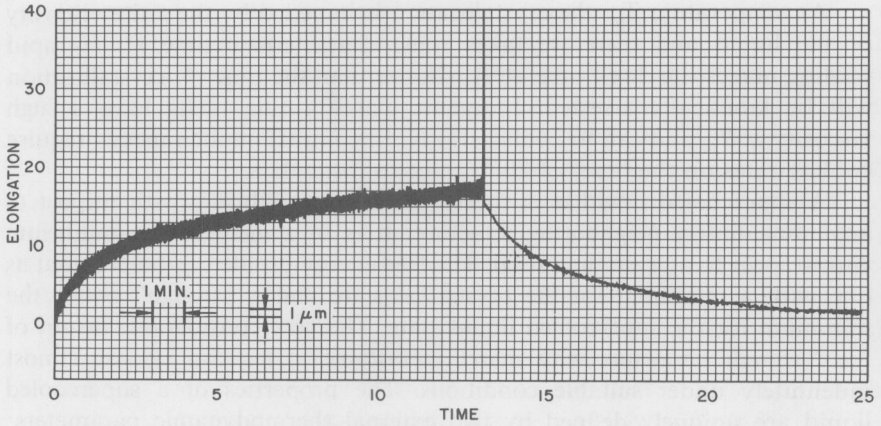


FIG. 3. Creep and recovery of a soda-lime-silicate glass at room temperature.

II. Formulation

The objectives and limitations of a viscoelastic study can be shown using a general formula delineating the change in strain e_{ij} in response to the stress and temperature:

$$\begin{aligned}
 de_{ij} &= de_{ij} + \frac{1}{3}\delta_{ij}d\epsilon \\
 &= \left[\left(\frac{\partial e_{ij}}{\partial s_{ij}} \right)_{T, \xi_1, \dots, \xi_n} ds_{ij} + \sum_{\rho, k=1}^n \left(\frac{\partial e_{ij}}{\partial \xi_k} \right)_{T, \xi_{\rho \neq k}} d\xi_k + \frac{s_{ij}}{2\eta} dt \right] \\
 &\quad + \frac{1}{3}\delta_{ij} \left[\left(\frac{\partial \epsilon}{\partial \sigma} \right)_{T, \Theta_1, \dots, \Theta_m} d\sigma + \sum_{\rho, k=1}^m \left(\frac{\partial \epsilon}{\partial \Theta_k} \right)_{T, \Theta_{\rho \neq k}} d\Theta_k \right] \\
 &\quad + \frac{1}{3} \left[\left(\frac{\partial \epsilon}{\partial T} \right)_{\sigma, \chi_1, \dots, \chi_l} dT + \sum_{\rho, k=1}^l \left(\frac{\partial \epsilon}{\partial \chi_k} \right)_{\sigma, \chi_{\rho \neq k}} d\chi_k \right]. \quad (1)
 \end{aligned}$$

In Eq. (1) e_{ij} , ϵ and s_{ij} , σ are deviatoric (shear, shape-changing) and dilatational (hydrostatic, volume-changing) strains and stresses, respectively, δ_{ij} is the Kronecker delta, ξ , Θ , and χ are order parameters, η is the viscosity, and T the temperature. The three bracketed terms describe responses to the shear stress, hydrostatic stress, and temperature. The first bracketed term consists of instantaneous or glassy response, structural or relaxation response, and viscous flow. The second and third bracketed terms consist of instantaneous and structural responses only.

The structural or relaxation response is controlled by order parameters. In the glassy state, the structure is frozen, the order parameters are constant, and the viscosity is infinitely large. The response is that of a solid. In the liquid state, the structure changes in response to the change in stress and temperature. The time variation of order parameters determines the kinetics of a liquidlike response.

The objective of the theory is to ascertain the meaning of the order parameters and suggest equations describing their rate dependencies. The objective of the experimental work is to determine compliances (partial derivatives in the first two bracketed terms), thermal expansion coefficients (partial derivatives in the third bracketed term), and rate constants of the order parameters (retardation times). Constraints commonly used in viscoelastic studies are small strains and stresses. The temperature changes can be large. Therefore, the temperature induced glass transition is within the scope of such studies whereas the pressure induced glass transition is not. Similarly, linear viscous flow is within the scope, but not non-newtonian flow.

The thrust of the present chapter is to show that a meaningful interpretation of the experiment is possible only on the basis of a theoretical model. Special emphasis throughout the entire chapter will be given to definitions of viscoelastic properties. In this context, an apparent neglect of terminology in the title of this work may seem incongruous. Strictly speaking, there is no viscoelasticity of glass because the glassy state is a frozen state. We should admit that we use the term "glass" loosely, although traditionally, to denote the class of inorganic materials both in glassy and supercooled liquid states.

III. Elasticity

In a cartesian coordinate system x_i , $i = 1, 2, 3$, a state of stress is given by

$$[\sigma] = \begin{pmatrix} \sigma_{11} & \sigma_{12} & \sigma_{13} \\ \sigma_{21} & \sigma_{22} & \sigma_{23} \\ \sigma_{31} & \sigma_{32} & \sigma_{33} \end{pmatrix}, \quad (2)$$

where σ_{ij} is the component parallel to the j direction and acting on the cubical element face perpendicular to the i direction, as shown in Fig. 4. If only dilatational stresses σ_{11} , σ_{22} , and σ_{33} are imposed, the resulting strains are parallel to the axes and, in terms of displacements U_i , are

$$\epsilon_1 = \partial U_1 / \partial x_1, \quad \epsilon_2 = \partial U_2 / \partial x_2, \quad \text{and} \quad \epsilon_3 = \partial U_3 / \partial x_3. \quad (3)$$

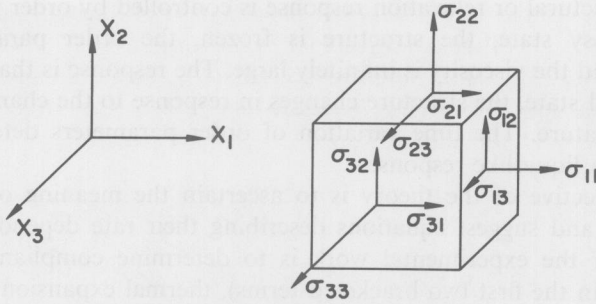


FIG. 4. Components of the stress tensor.

The sum of these strains is the relative volume change $\Delta V/V$, i.e.

$$\Delta V/V \equiv \varepsilon = \varepsilon_1 + \varepsilon_2 + \varepsilon_3. \quad (4)$$

If $\sigma_{11} = \sigma_{22} = \sigma_{33} = -P$, the hydrostatic pressure, we have

$$P = -K \Delta V/V, \quad (5)$$

where K is the bulk modulus. The shear stress σ_{ij} , $i \neq j$, produces a shear strain γ_{ij} developed by an edge originally parallel to the direction i turning toward the edge parallel to the direction j (see Fig. 5). The relationship between these quantities is given by

$$\sigma_{12} = G\gamma_{12}, \quad \sigma_{23} = G\gamma_{23}, \quad \text{and} \quad \sigma_{31} = G\gamma_{31}, \quad (6)$$

where G is the shear modulus and γ_{ij} is related to the displacements as

$$\gamma_{12} = \frac{\partial U_1}{\partial x_2} + \frac{\partial U_2}{\partial x_1}, \quad \gamma_{23} = \frac{\partial U_2}{\partial x_3} + \frac{\partial U_3}{\partial x_2}, \quad \text{and} \quad \gamma_{31} = \frac{\partial U_3}{\partial x_1} + \frac{\partial U_1}{\partial x_3}. \quad (7)$$

Shear strain can be represented in three different ways shown in Fig. 5. They are equivalent because they can be obtained one from another simply

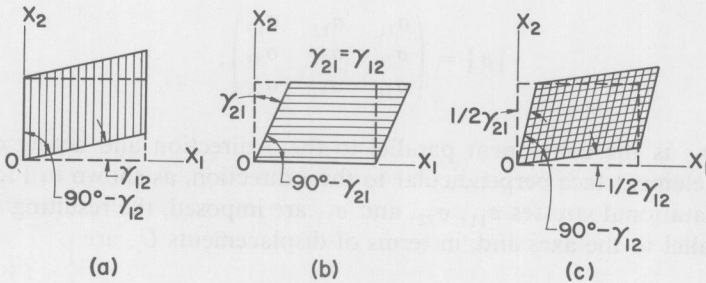


FIG. 5. Components of the shear strain tensor.

by rotation, without any additional deformation and, hence, effort. Note that the final angle of the parallelepiped is the same: $90^\circ - \gamma_{21}$ or $90^\circ - \gamma_{12}$, since $\gamma_{21} = \gamma_{12}$. It is the third method that is conventionally used, thus introducing a new shear strain equal to one-half of the total shear strain appearing between the axes x_i and x_j ,

$$\epsilon_{ij} = \frac{1}{2} \gamma_{ij} = \frac{1}{2} \left(\frac{\partial U_i}{\partial x_j} + \frac{\partial U_j}{\partial x_i} \right). \quad (8)$$

Equation (8) defines a component of a strain tensor that can also be represented as

$$[\epsilon] = \begin{pmatrix} \frac{\partial U_1}{\partial x_1} & \frac{1}{2} \left[\frac{\partial U_1}{\partial x_2} + \frac{\partial U_2}{\partial x_1} \right] & \frac{1}{2} \left[\frac{\partial U_1}{\partial x_3} + \frac{\partial U_3}{\partial x_1} \right] \\ \frac{1}{2} \left[\frac{\partial U_2}{\partial x_1} + \frac{\partial U_1}{\partial x_2} \right] & \frac{\partial U_2}{\partial x_2} & \frac{1}{2} \left[\frac{\partial U_2}{\partial x_3} + \frac{\partial U_3}{\partial x_2} \right] \\ \frac{1}{2} \left[\frac{\partial U_3}{\partial x_1} + \frac{\partial U_1}{\partial x_3} \right] & \frac{1}{2} \left[\frac{\partial U_3}{\partial x_2} + \frac{\partial U_2}{\partial x_3} \right] & \frac{\partial U_3}{\partial x_3} \end{pmatrix}. \quad (9)$$

Note that $\epsilon_{ij} = \epsilon_{ji}$ and that the definition of extensional components is still the same as in Eq. (3), i.e., $\epsilon_{11} = \epsilon_1$, etc. The strain tensor $[\epsilon]$ can be represented as a sum of the two tensors

$$[e] = \begin{pmatrix} \frac{\partial U_1}{\partial x_1} - \frac{\epsilon}{3} & \frac{1}{2} \left[\frac{\partial U_1}{\partial x_2} + \frac{\partial U_2}{\partial x_1} \right] & \frac{1}{2} \left[\frac{\partial U_1}{\partial x_3} + \frac{\partial U_3}{\partial x_1} \right] \\ \frac{1}{2} \left[\frac{\partial U_2}{\partial x_1} + \frac{\partial U_1}{\partial x_2} \right] & \frac{\partial U_2}{\partial x_2} - \frac{\epsilon}{3} & \frac{1}{2} \left[\frac{\partial U_2}{\partial x_3} + \frac{\partial U_3}{\partial x_2} \right] \\ \frac{1}{2} \left[\frac{\partial U_3}{\partial x_1} + \frac{\partial U_1}{\partial x_3} \right] & \frac{1}{2} \left[\frac{\partial U_3}{\partial x_2} + \frac{\partial U_2}{\partial x_3} \right] & \frac{\partial U_3}{\partial x_3} - \frac{\epsilon}{3} \end{pmatrix} \quad (10)$$

and

$$\epsilon = \begin{pmatrix} \frac{\epsilon}{3} & 0 & 0 \\ 0 & \frac{\epsilon}{3} & 0 \\ 0 & 0 & \frac{\epsilon}{3} \end{pmatrix}. \quad (11)$$

Although the sum of Eqs. (10) and (11) is evidently Eq. (9), this procedure conveys an important message: the strain tensor can be thought of as consisting of a pure shear, or shape-changing, strain $[e]$ and a pure hydrostatic strain ϵ . The strain $[e]$ is called deviatoric, and indeed produces no change in the volume because the sum of the diagonal components in

Eq. (10) is zero (cf. Eqs. (3) and (4)). The strain ϵ is called dilatational. It is important to make this distinction between deviatoric and dilatational components because glasses behave very differently when being deformed in these ways. This concerns both elastic and viscoelastic behavior, and fracture.

The same is true for the stress tensor σ_{ij} , which can be similarly decomposed into a deviatoric component s_{ij} and a dilatational component σ .

A concise way of writing this is

$$\sigma_{ij} = s_{ij} + \frac{1}{3}\delta_{ij}\sigma \quad (12)$$

and

$$\epsilon_{ij} = e_{ij} + \frac{1}{3}\delta_{ij}\epsilon, \quad (13)$$

where

$$\sigma = \sigma_{11} + \sigma_{22} + \sigma_{33} \quad (14)$$

and

$$\epsilon = \epsilon_{11} + \epsilon_{22} + \epsilon_{33} \quad (15)$$

and δ_{ij} is the Kronecker symbol

$$\delta_{ij} = \begin{cases} 0, & i \neq j \\ 1, & i = j \end{cases} \quad (16)$$

Now, the constitutive equations (5) and (6) can be written as

$$s_{ij} = 2Ge_{ij} \quad (17)$$

and

$$\sigma = 3K\epsilon. \quad (18)$$

The factors 2 and 3 appear as a result of the definitions of ϵ_{ij} and σ .

Simple tension or compression are the first practically important cases when both shear and dilatation are involved. Consider a uniaxial stress, i.e., $\sigma = \sigma_{11}$, $\sigma_{22} = \sigma_{33} = 0$. From Eqs. (12) and (13)

$$s_{11} = \sigma_{11} - \frac{1}{3}\sigma_{11} = \frac{2}{3}\sigma_{11} \quad (19)$$

and

$$e_{11} = \epsilon_{11} - \frac{1}{3}\epsilon_{11} = \frac{2}{3}\epsilon_{11}. \quad (20)$$

Thus, a uniaxial stress is $\frac{2}{3}$ shear and $\frac{1}{3}$ dilatation.

The relationship between extensional stress and strain is given by

$$\sigma_{11} = E\epsilon_{11}, \quad (21)$$

where E is the Young's modulus. The lateral extensional strains are of