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Determination of the Frequency Dependent Bulk Modulus of Liquids Using a Piezoelectric Spherical Shell

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Abstract.

Through the coupling between the electrical capacitance of a spherical piezoceramic shell and the mechanical stiffness of a liquid contained therein, the frequency dependent adiabatic bulk modulus $K_s(\omega)$ of the liquid can be derived.Using this method, $K_s(\omega)$ of glycerol in the range 15 Hz - 15 kHz has been measured at the glass transition. The loss peak frequencies of the compressibility $\kappa_s(\omega) = K_s^{-1}(\omega)$ and the specific heat $c_p(\omega)$ are found to be nearly equal .

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The elastic properties of an ideal isotropic elastic solid are characterized by two elastic constants, the shear modulus G and the bulk modulus K. Concerning the latter, the thermodynamic condition, isothermal (K_T) or adiabatic (K_S) should be stated. The viscous behaviour of an ideal Newtonian liquid is characterized by a shear viscosity η_0. Applying a harmonically varying shear strain with frequency f and cyclic frequency $\omega=2\pi f$, shear viscosity can be conceived as a frequency dependent shear modulus $G=-i\omega\eta_0$. Real liquids show both liquid and solid behaviour in their shear modulus. A simple phenomenological model reconciling these features is the Maxwell model [1]

$$G(\omega)=(G_\infty^{-1}+(-i\omega\eta_0)^{-1})^{-1} \qquad (1)$$

showing solidlike behaviour at high frequencies ($G\to G_\infty, \omega\to\infty$) and liquidlike behaviour at low frequencies ($G\to -i\omega\eta_0, \omega\to 0$). The transition takes place at $\omega_p=\tau_M^{-1}(T)$, where $\tau_M(T)=\eta_0/G_\infty$ is the Maxwell relaxation time. This is the characteristic phenomenon of the glass transition, and the glass transition temperature T_g in the Maxwell model is the temperature, where $\tau_M(T_g)$ is equal to a characteristic experimental time scale or reciprocal frequency. The imaginary part of G has its maximum at the loss peak frequency ω_p.

Real liquids cannot be described by a single relaxation time, since $G(\omega)$ has a more complicated frequency dependence. Other properties like the specific heat [2],[3] and bulk modulus [4] also show relaxation . It is an important experimental and

theoretical task in the study of the glass transition to find connections between these properties. In this respect one should distinguish between relationships which stem from linear irreversible thermodynamics [5] and relations which depend on more specific models [6]. Let s be the entropy density, T the temperature, ϵ_{ij} the strain tensor and σ_{ij} the stress tensor. Denote the relative volume change by $\epsilon = Tr(\epsilon_{ij})$ and the hydrostatic pressure by $p = -\frac{1}{3}Tr(\sigma_{ij})$. Then one has

$$\begin{pmatrix} ds \\ d\epsilon \end{pmatrix} = \begin{pmatrix} \frac{1}{T}c_p & \alpha_p \\ \alpha_p & \kappa_T \end{pmatrix} \begin{pmatrix} dT \\ -dp \end{pmatrix} = J \begin{pmatrix} dT \\ -dp \end{pmatrix} \qquad (2)$$

where c_p is the isobaric specific heat, α_p is the isobaric expansion coefficient and κ_T is the isothermal compressibility. These 3 quantities constitute the thermoelastic compliance matrix J. In equilibrium thermodynamics the symmetry of J is one of the Maxwell relations $(\frac{\partial s}{\partial p})_T = -(\frac{\partial \epsilon}{\partial T})_p$. The recipe of transfering to nonequilibrium thermodynamics for a relaxing medium is simply to let $ds, d\epsilon, dT, dp$ be the amplitudes of harmonically varying small perturbations $\propto e^{-i\omega t}$. Then c_p, α_p, κ_T become complex, thereby describing the phase shift introduced by the relaxation processes. The symmetry of J still holds, but now it expresses an Onsager relation [5]. Thus, there are three independent complex thermoelastic response functions, which should be investigated. Experimentally related functions may be more

convenient to measure. The triple c_p, α_p, κ_s would contain the same information since

$$\kappa_T(\omega) = \kappa_s(\omega) + \frac{T}{c_p(\omega)}\alpha_p^2(\omega) \qquad (3)$$

which follows from interchanging the variables of (2).Thus (3) is as equally valid in linear nonequilibrium thermodynamics as in equilibrium thermodynamics. An example of a specific model is Zwanzigs [6] proposal that the isochoric specific heat c_v is frequency independent and that $c_p(\omega)$ and $\kappa_T(\omega)$ are related by

$$c_p(\omega) = c_v + (c_p(0) - c_v)\frac{\kappa_T(\omega)}{\kappa_T(0)} \qquad (4)$$

A knowledge of three independent thermoelastic response functions could verify this model or give a clue to other models. The present work should be seen in this perspective.

We have developed a new method for measuring $K_s(\omega)$ $(= \kappa_s(\omega)^{-1})$ at low frequencies, i.e. frequencies at which the corresponding acoustic wavelength is much larger than the sample size (quasistatic regime). On the other the frequencies are sufficiently high to ensure adiabatic conditions, i.e. the corresponding thermal diffusion length is much smaller than the sample size.

Conventional methods [7] have to measure both the longitudinal, M and the shear, G modulus through the longitudinal, c_l and transversal, c_t sound velocity. The bulk

modulus is then given by the relation

$$M = K + \frac{4}{3}G \qquad (5)$$

These methods demand high and discrete frequencies say 10 MHz in order that the corresponding wavelength is smaller than or comparable to the sample size.

Thus, we can in the present experiment obtain information on $K_s(\omega)$ continous in a frequency range not usually considered. Furthermore, our method also determines M at certain resonance frequencies.

The method of Mckinney, Edelman and Marvin [8] is a quasistatic method as ours. The principle of their method, however, is quite different, involving an inert liquid as pressure transfering agent and depending on both an emitter and receiver of acoustic vibrations. We have no separate emitter and receiver, only one transducer constituting the sample cell also. The measuring cell is a spherical shell of a piezoceramic material (pz26, Ferroperm, Denmark) polarized in radial direction. We will call it the piezoelectric bulk modulus gauge (PBG). It is covered with electrodes on the inner and outer surfaces. An insulated wire is put through the shell and connected to the inner electrode and another wire is connected to the outer electrode. On applying a potential difference across the shell, the PBG will expand or contract radially depending on the polarity. For a mechanical free outer surface, the coupling between the complex amplitudes of the normal stress σ and the volume change ΔV respectively the surface charge Q and the potential difference U is given by a transfer matrix c_{ij}

$$\begin{pmatrix} U \\ Q \end{pmatrix} = \begin{pmatrix} c_{11} & c_{12} \\ c_{21} & c_{22} \end{pmatrix} \begin{pmatrix} \sigma \\ \Delta V \end{pmatrix} \tag{6}$$

The measured electrical capacitance therefore depends on whether the shell is free to move ($\sigma=0$) or clamped ($\Delta V=0$),

$$C_{free}(\omega) = \frac{c_{22}}{c_{12}}, \quad C_{clamped}(\omega) = \frac{c_{21}}{c_{11}} \tag{7}$$

If a medium of stiffness $S(\omega) = \frac{\sigma}{\Delta V}$ is placed inside the PBG, then the electrical capacitance becomes

$$C(\omega) = \frac{c_{12} + c_{22}S}{c_{11} + c_{12}S} \tag{8}$$

Thus, S can be found knowing c_{ij} and measuring $C(\omega)$. The quantity $\frac{c_{12}}{c_{11}}$ gives a characteristic stiffness where the PBG is most sensitive. For a thin piezoelectric ceramic shell, c_{ij} can be expressed by the inner radius r, thickness t, density ρ, elastic constants s_{11}, s_{12}, piezoelectric constant d_{13} and dielectric constant ϵ_{33} [9]. Introducing the "breathing mode" resonance frequency $\omega_c = \frac{1}{r}\sqrt{\frac{2}{(s11+s12)\rho}}$, the planar coupling constant $k_p = \sqrt{\frac{2d_{13}^2}{(s_{11}+s_{12})e_{33}}}$, the free capacitance at zero frequency $C_0 = C_f(0) = \frac{4\pi r^2}{t} e_{33}$ and the inertance $L = \rho\frac{t}{4\pi a^2}$, the result is

$$(C_{ij}) = \begin{pmatrix} \frac{1}{k_p \omega_c \sqrt{LC_0}} & \frac{\omega_c}{k_p}\sqrt{\frac{L}{C_0}}\left(1-\left(\frac{\omega}{\omega_c}\right)^2\right) \\ \frac{1-k_p^2}{k_p \omega_c}\sqrt{\frac{C_0}{L}} & \frac{\omega_c}{k_p} LC_0\left(1-(1-k_p^2)\left(\frac{\omega}{\omega_c}\right)^2\right) \end{pmatrix} \qquad (9)$$

In the specific case $\rho = 7.65\ gcm^{-3}, t = 0.10\ cm, r = 0.90\ cm$, whereby $L = 7.52 \cdot 10^{-2}\ gcm^{-4}$. By fitting a measurement of the free electrical capacitance to the theoretical expression

$$C_{free}(\omega) = \frac{c22}{c12} = C_0 \frac{1-(1-k_p^2)\left(\frac{\omega}{\omega_c}\right)^2}{1-\left(\frac{\omega}{\omega_c}\right)^2} \qquad (10)$$

the three constants C_0, k_p, ω_c are found (see fig. 1). These constants are both temperature and weakly time dependent due to annealing processes in the piezoceramic itself. Thus the same time and temperature scheme is exactly followed during reference measurement and modulus measurement. Typical values are $C_0 = 12nF$, $k_p = 0.51$, $f_k = \frac{\omega_c}{2\pi} = 85\ kHz$. One has to correct the expression (9) for the transfer matrix taking the finite thickness of the transducer into account. We have indeed calculated and used the general transfer matrix, but these lengthy expressions are omitted here. The corrections amounts to 15% on K_s.

At the top of the sphere a small hole of radius r_h makes it possible to fill the sphere with liquid. Also, a reservoir of liquid resides in a small tube on top of the sphere. The hole connects this to the inside of the sphere, allowing for thermal expansion of the liquid. Despite this hole, the liquid is

virtually confined in the sphere at the frequencies of interest: Assuming Poiseuille flow through the hole, a characteristic flow time τ_f will be

$$\tau_f = \frac{32}{3}\frac{r^3 t}{r_h^4}\frac{\eta}{K} \approx 10^5 \tau_M \qquad (11)$$

Thus, one has in fact quite a large range of times beyond the Maxwell relaxation time at disposal. On the other hand, the cell can of course only be filled in a reasonably time at high temperatures where the viscosity is low.

The stiffness $S(\omega)$ of a spherical isotropic viscoelastic solid is derivable from the solution of the equation of motion [10]. If the density is ρ_1, the longitudinal wavevector $k_1 = \sqrt{\frac{\rho_1}{M}}\omega$ and the volume $V = \frac{4}{3}\pi r^3$ then one finds

$$S(\omega) = \frac{1}{V}\{K - M(1 + \frac{1}{3}\frac{(k_1 r)^2 \sin(k_1 r)}{k_1 r \cos(k_1 r) - \sin(k_1 r)})\} \qquad (12)$$

At low frequencies $S(\omega)$ is simply $\frac{K_s(\omega)}{V}$. At higher frequencies it depends on both $K(\omega)$ and $M(\omega)$ because longitudinal waves are excited.

The new method was applied to that canonical example of the glass transition, glycerol. Fig. 2 shows how the electrical capacitance $C(\omega)$ of the PBG is reduced from its free value by the partial clamping of the transducer due to the contained liquid. The glass transition in this picture is seen indirectly in the decrease of C

with increasing frequency. Also shown is the measured C_{free} and the calculated $C_{clamped}$.

Fig. 3 and 4 presents the measured real and imaginary part of bulk modulus K_s as a function of frequency at different temperatures.Denote the relaxational part $K_s(\omega) - K_s(0)$ by $K_r(\omega)$. The solid line represents a fit to data of a phenomenological model, where $K_r(\omega)$ is given by an extended Maxwell model $K_r(\omega) = K_r(\infty)(1+(-i\omega\tau_b)^{-1}+q(-i\omega\tau_b)^{-\alpha})^{-1}$. It is found that $q = 1.40$ and $\alpha = 0.43$. τ_b is temperature dependent and corresponds to the Maxwell relaxation time.

The logarithm of the loss peak frequency f_p of the compressibility as a function of the reciprocal temperature is shown in fig. 5. f_p has been fitted to $f_p = f_0 \exp(-(\frac{T_0}{T})^3)$ finding $f_0 = 6.67 \cdot 10^{12} Hz$ and $T_0 = 612\ K$. τ_b is $0.126 f_p^{-1}$ in the fitting model for the present values of q and α. The figure shows, that the loss peak frequency of the specific heat $c_p(\omega)$ earlier measured [3] is almost the same as the loss peak frequency of $\kappa_s(\omega)$ if extrapolated down in temperature. This would also be expected for a comprehensive model of the thermoviscoelastic properties of the liquid.The expression (4) predicts equality of the loss peak frequencies of $c_p(\omega)$ and $\kappa_T(\omega)$ but not necessarily of $\kappa_s(\omega)$.

The real and imaginary part of the specific stiffness $S(\omega)V$ of the liquid sphere as a function of temperature at 1 kHz is shown in fig. 6. Two dispersion regions are seen with maxima in the

imaginary part at 214 K and 268 K respectively. The low temperature dispersion region is simply the glass transition in K_s, since at this frequency the measured specific stiffness (12) is equal to bulk modulus. At higher temperature the liquid is able to flow through the filling hole and this gives rise to the second dispersion region. Therefore although the measured stiffness still reflects relaxation processes at the glass transition, it does not give bulk modulus and cannot easily be analyzed in a rigorous way. The ratio of f_p at 214 K and 268 K is 10^5, which is in agreement with the estimate (11).

According to (12) a viscoelastic sphere will show stiffness resonances when $\tan(k_l r) = k_l r$. These resonances are seen in the electrical capacitance of the PBG. Although the resonances are moved due to the mechanical coupling of the PBG and the liquid, this is only of importance for the lowest lying resonances. Thus for resonance frequency ν_n, $n \geq 3$ the condition simply gives longitudinal modulus to a good approximation as $16(1+2n)^{-2}\rho_l \nu_n^2 r^2$.

In fig. 6 M calculated by the third resonance at 280-320 K is shown. At these temperatures the inverse Maxwell relaxation time is much higher than the resonance frequency. Thus shear modulus can be neglected compared to bulk modulus and (5) reduces to $M_0 = K_0$, where index 0 means the low frequency limit. The extrapolation of $K_0(T)$ measured by this resonance technique into the temperature region, where the quasistatic method works, agrees within 1%. In this way one has an independent and simple check on the validity of the procedure of the quasistatic method.

In conclusion, the main benefits of the method are the following.The transducer converts a mechanical impedance to an electrical impedance, which is convenient to measure. The small transducer is handy to place in a cryostat and reach thermal equilibrium in a reasonable time.The spherical symmetry makes it possible to calculate the stiffness of the liquid and the transfer matrix of the transducer analytically. Finally the transducer can operate in two modes, quasistatic and resonance. The results obtained for glycerol points towards a related relaxation of $\kappa_s(\omega)$ and $c_p(\omega)$. The development however of a method of measuring $\alpha_p(\omega)$ will be necessary to get full information of the thermoelastic properties of liquids at the glass transition.

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Figure captions.

Fig.1. The first resonance (breathing mode) of the piezoceramic shell without liquid at 210 K (□). The solid line is fit of data to equation (10).

Fig.2. The electrical capacitance of the PBG filled with glycerol at 210 K (□). Upper solid line is C_{free}. Lower solid line is $C_{clamped}$.

Fig.3. The real part of bulk modulus of glycerol at the glass transition as a function of frequency at different temperatures. Solid line is fit to an extended Maxwell model.

Fig.4. The imaginary part of bulk modulus corresponding to the real part shown in fig.3.

Fig.5. The logarithm of the loss peak frequency of the compressibility (□) and of the specific heat (×).

Fig.6. Real (+) and imaginary (□) parts of the specific stiffness at 1 kHz measured by the quasistatic method. K_0 (×) measured by the resonance method.

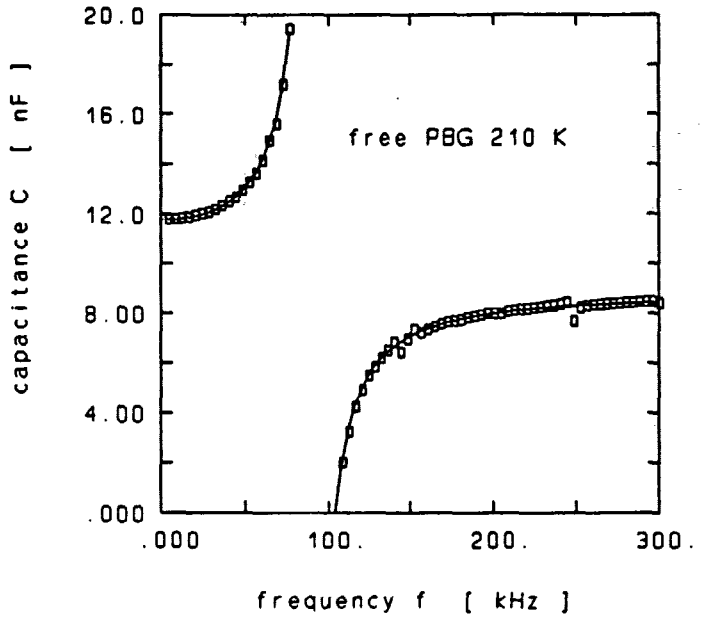
20.0
16.0
12.0
8.00
4.00
.000
capacitance C [nF]
free PBG 210 K
.000
100.
200.
300.
frequency f [kHz]

Figure 1.

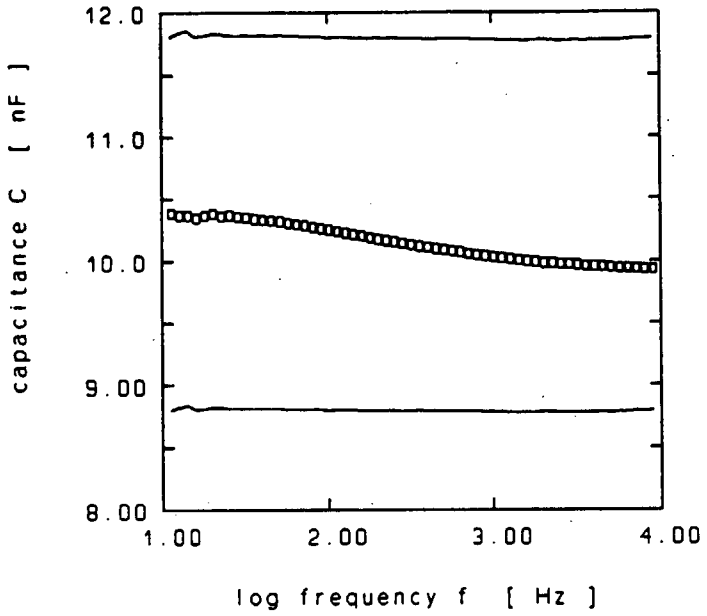
12.0
11.0
10.0
9.00
8.00
capacitance C [nF]
1.00
2.00
3.00
4.00
log frequency f [Hz]

Figure 2.

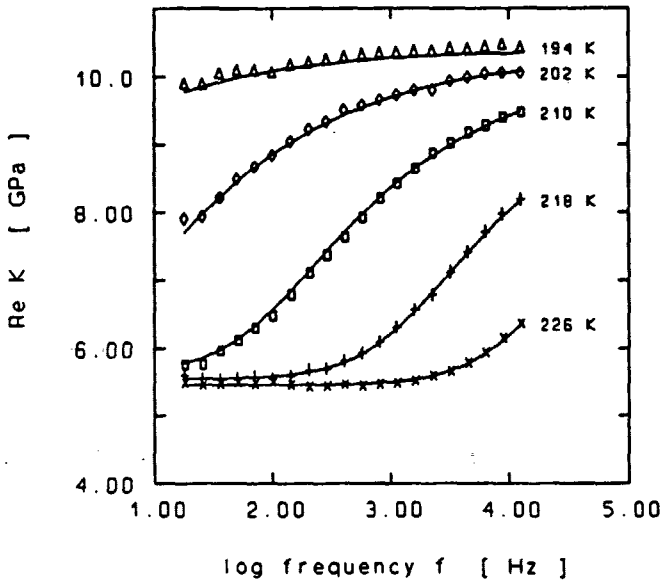
194 K
202 K
210 K
218 K
226 K
10.0
8.00
6.00
4.00
Re K [GPa]
1.00
2.00
3.00
4.00
5.00
log frequency f [Hz]

Figure 3.

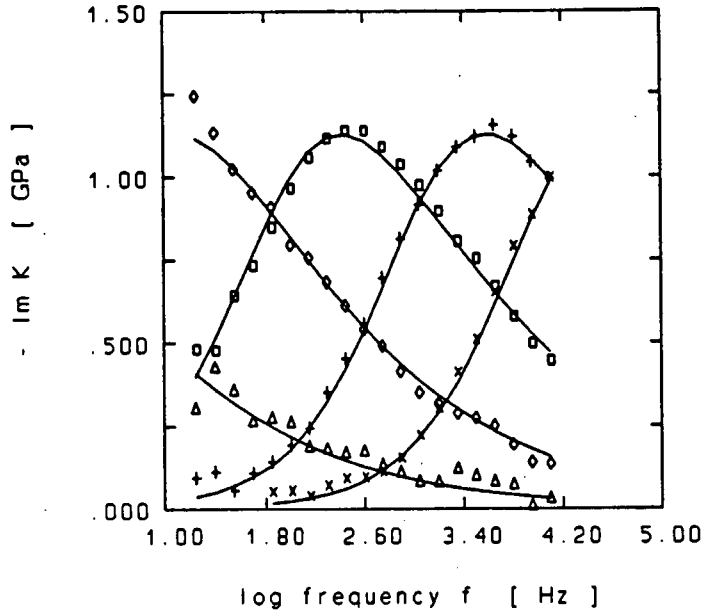
1.50
1.00
.500
.000
- Im K [GPa]
1.00
1.80
2.60
3.40
4.20
5.00
log frequency f [Hz]

Figure 4.

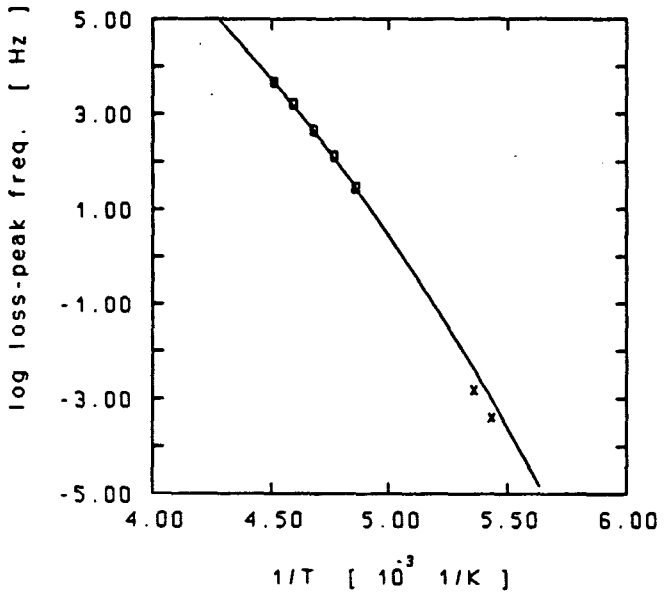
log loss-peak freq. [Hz]
5.00
3.00
1.00
-1.00
-3.00
-5.00
4.00
4.50
5.00
5.50
6.00
1/T [10^-3 1/K]

Figure 5.

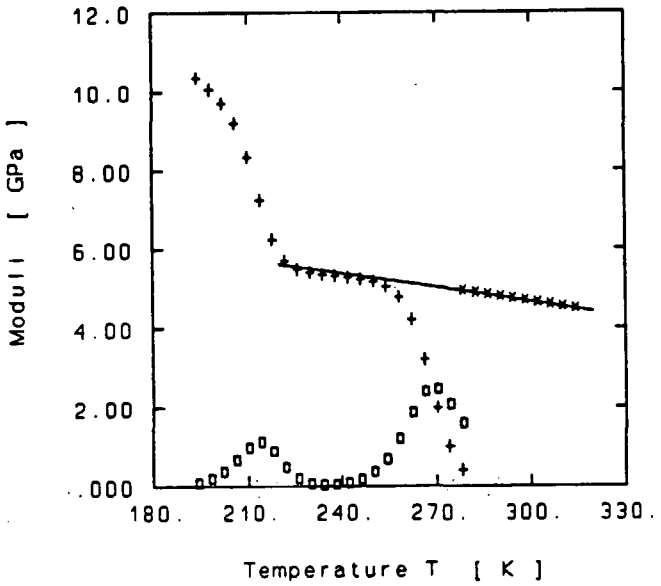
12.0
10.0
8.00
6.00
4.00
2.00
.000
Modul I [GPa]
180.
210.
240.
270.
300.
330.
Temperature T [K]

Figure 6.

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Vejleder: Stig Andur Pedersen

251/93 Genotypic Proportions in Hybrid Zones

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252/93 MODELLERING AF TILFÆLDIGE FÆNOMENER

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253/93 Kuglepakning

Teori og model

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254/93 Regressionsanalyse

Materiale til et statistikkursus

af: Jørgen Larsen

255/93 TID & BETINGET UAFHÆNGIGHED

af: Peter Harremoës