Special Topics in Relaxation in Glass and Polymers

Lecture 7: Viscoelasticity III
Dynamic Testing

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Research and Technology Development

SCHOTT AG

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In the following, relaxation experiments on glass will be discussed.

Disclaimer:

As all lectures in this course, the manuscript may contain errors despite its careful preparation.

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Contact your local instructor for further information.

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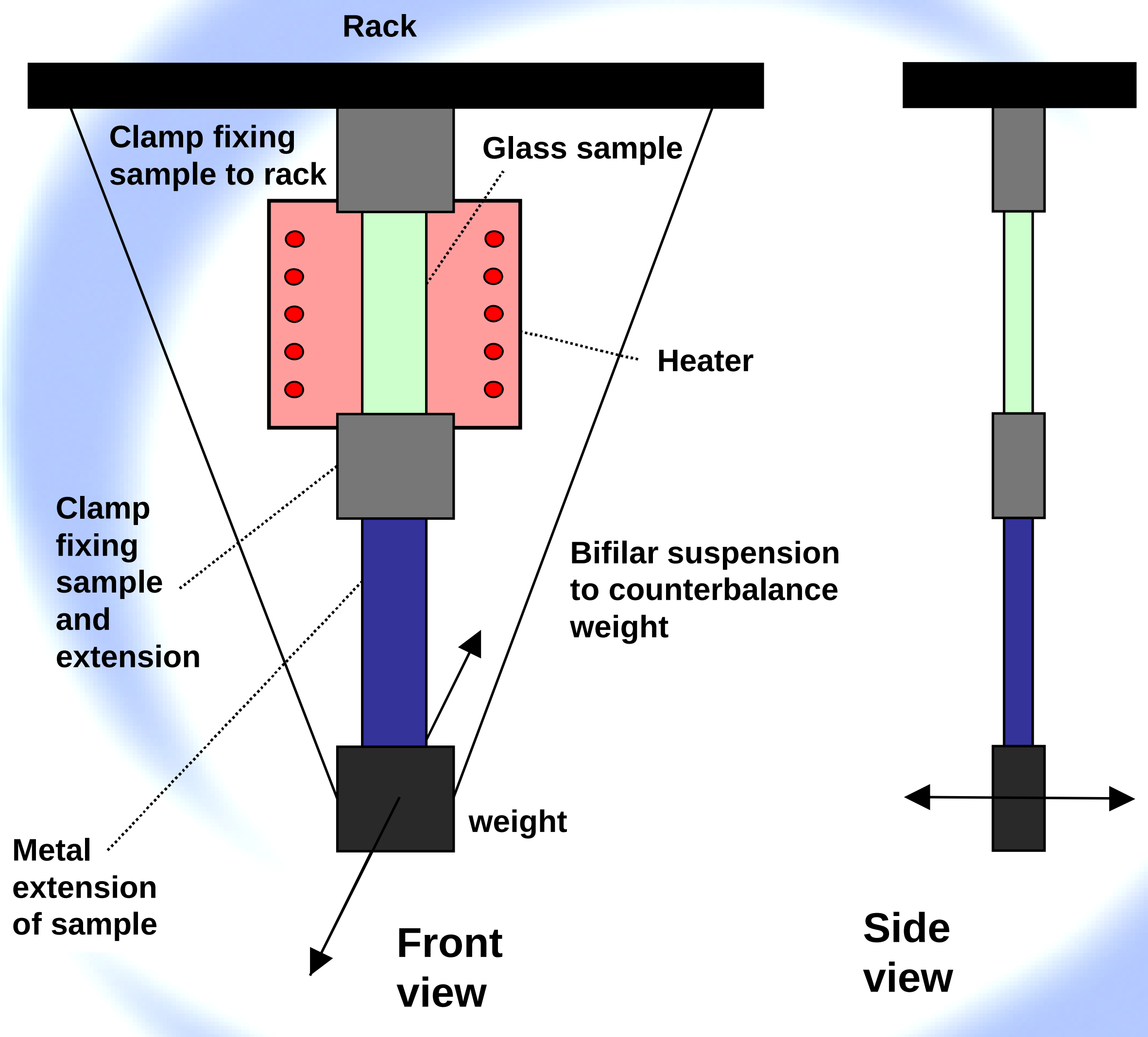
Dynamic testing via self-oscillations (at the eigenfrequency)

Example: Flexure pendulum, after Rötger, revitalised in the 1990s by Bark-Zollmann et al.

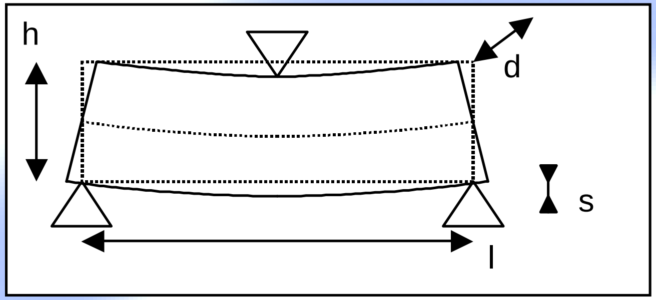


The flexure pendulum monitors self oscillations in a bending mode.
From the oscillation period, Young´s modulus is determined.
From the logarithmic decrement, relaxation time is determined.

The bifilar suspension counterbalances the weight at the bottom in order to prevent other restoring forces than those coming from bending the sample and its metal extension.
The metal extension works as "analogue amplifier" of the displacement signal.

Analysis of the flexure pendulum

Starting point: Bending (of thin plates) and elongation are equivalent:



For thin plates, bending is equivalent to compression of one side and dilatation of the other side.

So the viscoelastic behaviour of both bending and elongation is determined by Young´s modulus E and the extensional viscosity η_e. The extensional viscosity describes the creep of, e.g., a glass rod which is subject to continuous elongation.

$$s = F \cdot \frac{l^3}{4 \cdot E \cdot d \cdot h^3} \propto \frac{1}{E}$$ Elastic deformation (bending)

$$\frac{\Delta l}{l} = \frac{F/A}{E} \propto \frac{1}{E}$$ Elastic deformation (elongation)

$$\frac{ds}{dt} = F \cdot \frac{l^3}{4 \cdot \eta_e \cdot d \cdot h^3} \propto \frac{1}{\eta_e}$$ Creep (bending)

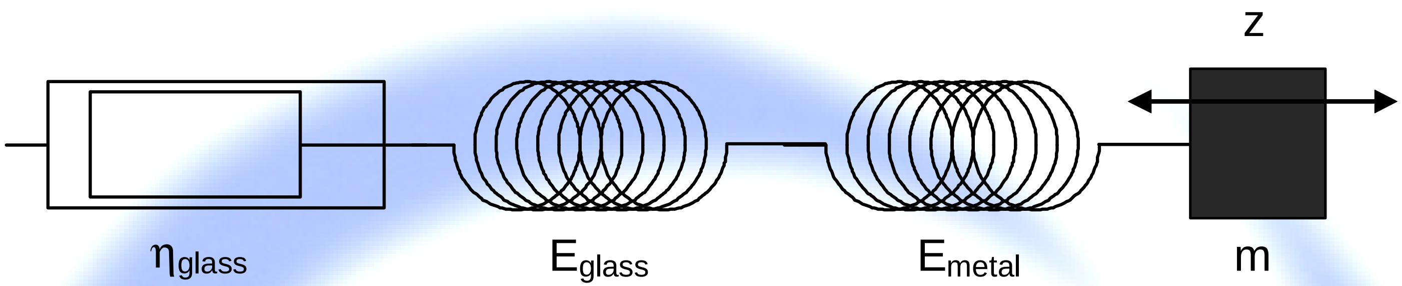
$$\frac{dl}{dt} = l \cdot \frac{F/A}{\eta_e} \propto \frac{1}{\eta_e}$$ Creep (elongation)

For incompressible*) Newtonian (η independent from deformation rate) fluids one can derive $\eta_e = 3 \cdot \eta_{(shear)}$ **so that with** $\frac{1}{E} = \frac{1}{3G} + \frac{1}{9K} \approx \frac{1}{3G}$ **one has** $\frac{\eta_{(shear)}}{G} \approx \frac{\eta_e}{E}$

the first being Maxwell´s relaxation time and the latter measurable by the relaxometer.

***) Course approximation. In reality we do not have K = ∞ and should not neglect bulk viscoelasticity.**

Analysis of the flexure pendulum (continued)



$$3\cdot\eta_{glass}\cdot\dot{\varepsilon}_{viscos,glass}\cdot A_{glass}=\varepsilon_{elastic,glass}\cdot E_{glass}\cdot A_{glass}=\varepsilon_{metal}\cdot E_{metal}\cdot A_{metal}$$

$$=f(t)=-m\cdot\ddot{z}=-m\cdot\left(l_{glass}\cdot\left(\ddot{\varepsilon}_{viscos,glass}+\ddot{\varepsilon}_{elastic,glass}\right)+l_{metal}\cdot\ddot{\varepsilon}_{metal}\right)$$

$$\Rightarrow$$

$$m\cdot\left(l_g+l_m\cdot\frac{E_g\cdot A_g}{E_m\cdot A_m}\right)\cdot\ddot{\varepsilon}_{el,g}+m\cdot l_g\cdot\frac{E_g}{3\cdot\eta_g}\cdot\dot{\varepsilon}_{el,g}+E_g\cdot A_g\cdot\varepsilon_{el,g}=0$$

$$\Rightarrow$$

$$\varepsilon_{el,g}(t)=\varepsilon_{el,g,0}\cdot e^{-\left(\frac{m\cdot l_g\cdot\omega_{gm}^2}{6\cdot\eta_g\cdot A_g}\right)t\pm i\cdot\omega_{gm}\cdot t}\quad,\quad \omega_{gm}=1\Big/\sqrt{m\cdot\left(\frac{l_g}{E_g\cdot A_g}+\frac{l_m}{E_m\cdot A_m}\right)}$$

The last equations hold for small attenuations only.

The overall oscillation will have the same time dependence as $\varepsilon_{el,g}$.

In a first approach, one may treat the set-up of the flexure pendulum as an oscillating series of a simple Maxwell-model representing the glass, an 2nd spring representing the metal, and a load.
Of course, a more sophisticated glass model (Burger etc.) would be possible also.
If excited once, this system will carry out damped oscillations.

Analysis of the flexure pendulum (further continued)

If one introduces the logarithmic decrement L as the attenuation after one oscillation:

$$\Lambda = -Ln\left(\frac{z\left(t + 2\pi / \omega_{gm}\right)}{z(t)}\right) = -Ln\left(e^{-\left(\frac{m \cdot l_g \cdot \omega_{gm}^2}{6 \cdot \eta_g \cdot A_g}\right)\frac{2\pi}{\omega_{gm}}}\right)$$

and the eigenfrequency of the system with metal only (no glass):

$$\omega_m = 1 \Big/ \sqrt{m \cdot \left(\frac{l_m}{E_m \cdot A_m}\right)}$$

, one gets:

Note: τ is independent from geometry and therefore the same for the flexure pendulum and its linear representation.

Maxwell´s relaxation time:

$$\tau = \frac{\eta_g}{G_g} \approx \frac{3 \cdot \eta_g}{E_g} = \frac{\left(1 - \omega_{gm}^2 / \omega_m^2\right) \cdot \pi}{\Lambda \cdot \omega_{gm}}$$

and $E_g = \ldots$

For Eg, the result one would get here would be valid for the linear representation only.

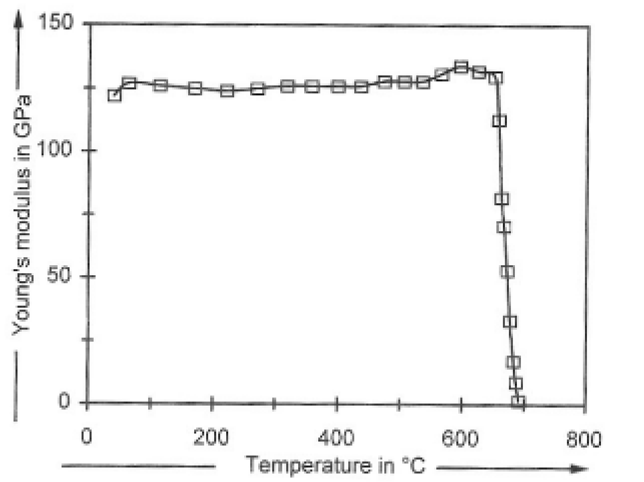


Figure 5. Young's modulus of the glass LaSK3 in dependence on temperature.

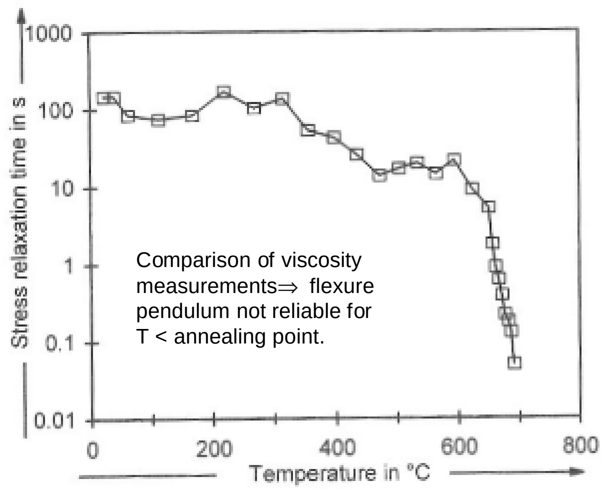


Figure 6. Stress relaxation time of the glass LaSK3 in dependence on temperature.

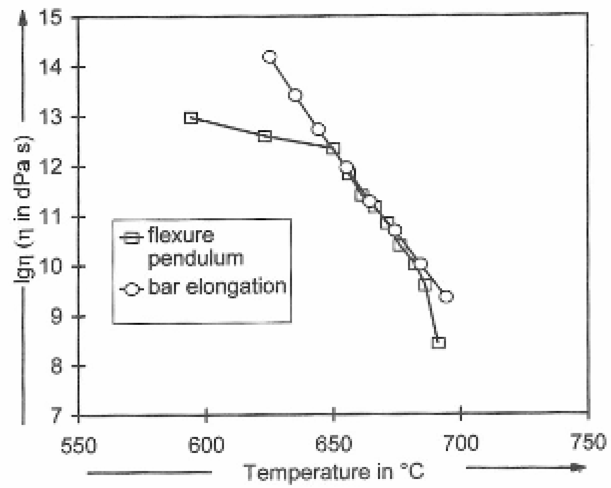


Figure 9. Comparison of the viscosities obtained by means of the flexure pendulum equipment and with the help of the bar elongation method, in dependence on temperature, example glass LaSK3.

Glastech. Ber. Glass Sci. Technol. 71 (1998) No. 3

Further experiments with the flexure pendulum

(Note again that more sophisticated models and data reductions would be possible also.)

Table 1. Syntheses of the investigated optical and technical glasses

glass	SiO_2	B_2O_3	Al_2O_3	La_2O_3	Y_2O_3	TiO_2	ZrO_2	PbO	BaO	CaO	ZnO	K_2O	Na_2O	KHF_2	As_2O_3	AlF_3	K_2TiF_6	WO_2
FK6	++	***	***											+		+		
SF6	+							+++				*	*		*			
F16	++	**	*			**		***				***					**	
SF10	++					*		+++				*	*		*			
KzFS1	*	+++	**					++										
ZK7	+++	***	*								***		**		*			
BK7n	+++	***							*			**	**		*			
LaF86		++		+		**					++							**
F11	+++	*				+						**	**		*			
PSK1	+++	***	*						+			*	*		*			
LaSK3	**	++		++	**		*		*		**							
Na28Si	+++												+					
G20	+++	**	**						*	*		*	**		*			

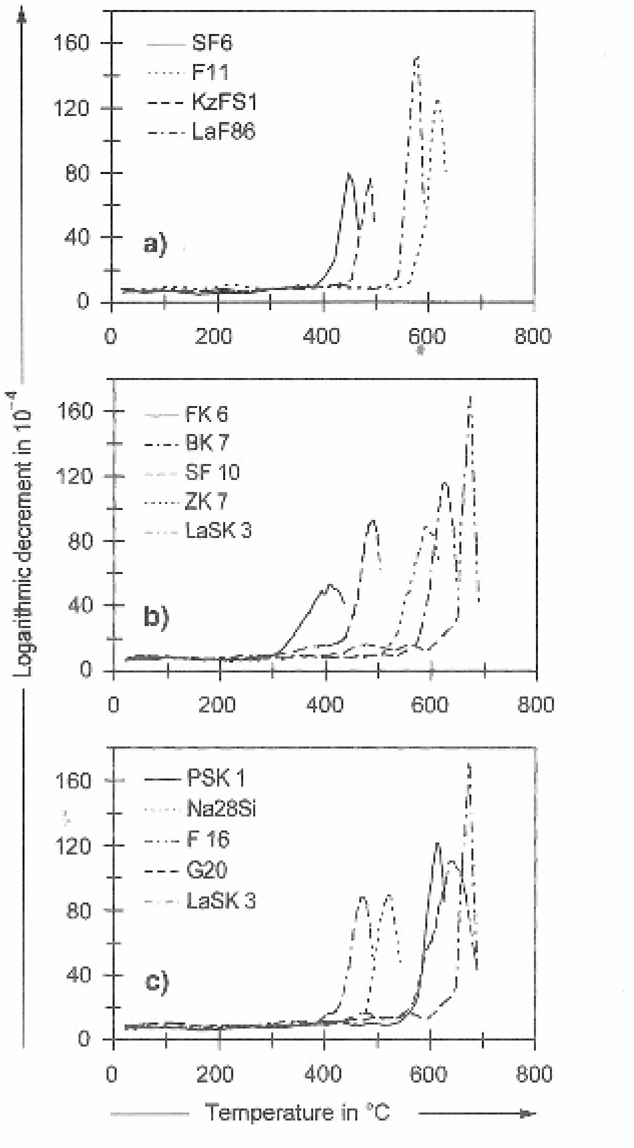
Explanations: * = 0 to 5 wt%, ** = 5 to 10 wt%, *** = 10 to 20 wt%, + = 20 to 30 wt%, ++ = 30 to 50 wt%, +++ = ≥50 wt%.

Note that many of these glasses have been replaced with so-called N-types in the meantime (no Pb, no As).

Table 2. Some material properties of the investigated glasses

glass	refractive index	Young's modulus in GPa	shear modulus in GPa	Poisson number	density in g/cm³	glass transition temperature in °C
FK6	1.44690	40	16	0.25	2.29	325
SF6	1.81262	53	22	0.24	5.18	426
F16	1.60344	64	26	0.24	2.87	450
SF10	1.73430	61	25	0.21	4.26	470
KzFS1	1.61637	55	22	0.27	3.16	475
ZK7	1.5105	69	29	0.20	2.50	525
BK7n	1.51859	84	50	0.15	2.52	565
LaF86	1.77491	104	40	0.31	4.18	570
F11	1.62507	82	34	0.22	2.66	580
PSK1	1.54979	81	33	0.22	2.87	590
LaSK3	1.73444	112	43	0.30	4.05	645
Na28Si		62				450
G20		74				565

Glastech. Ber. Glass Sci. Technol. 71 (1998) No. 3



Figures 4a to c. Logarithmic decrements of the coupled system pendulum-specimen bar for different glasses (f_P = 0.79 Hz), a) SF6, LaF86, KzFS1 and F11; b) FK6, BK7, SF10, ZK7 and LaSK3; c) PSK1, Na28Si, F16, G20 and LaSK3.

Some exercises:

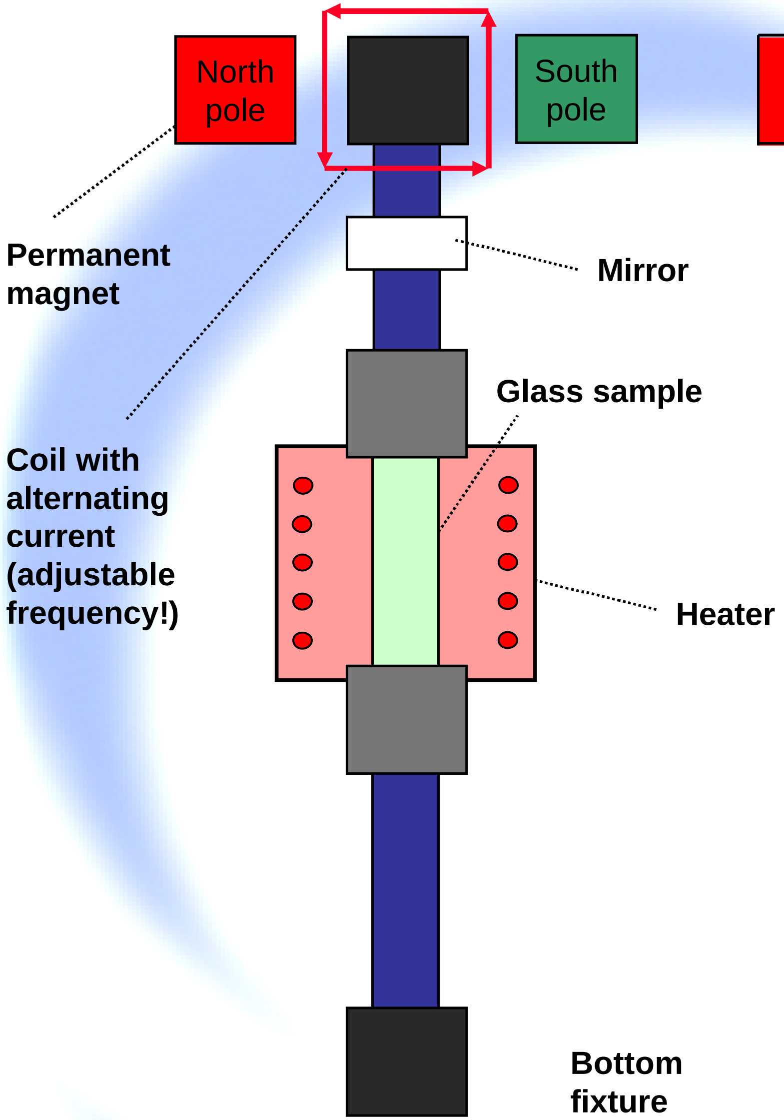
1. **Consider 3-point bending with constant load. How is the relation of viscosity to the constant velocity at which the middle of the sample moves downward?**

2. **Consider the simple representation of the flexure pendulum. Does the eigenfrequency increase or decrease if the glass sample is removed and the metal strip is tested alone?**

3. **Cos(ωt) can be written as a linear combination of Exp(iωt) and Exp(-iωt). How?**

4. **Consider again the flexure pendulum. The time dependence is Exp(-t/10s)*Cos(2*Pi*10Hz*t). Calculate the logarithmic decrement.**

Dynamic testing via forced oscillations



Example: Torsional device, after de Bast and Gilard

An alternating angular momentum is applied which is caused by alternating current running through the coil in the permanent magnetic field. The relation between angular moment and current is known from calibration.
This angular momentum causes torsional stress*) in the sample. The resulting torsion is recorded via the course of a light beam which is reflected at the mirror fastened to the upper sample holder.
The size of the torsional angle as well as its phase shift to the angular momentum is recorded.
From this phase shift δ, the relaxation kinetics may be determined.

***) Note that in contrast to the flexure pendulum, we have pure shear here. Bulk viscoelasticity does not exist here and need not be neglected therefore.**

Analysis of the torsional device

$$\sigma(t) = 2\cdot G\cdot \int_{-\infty}^{t} e^{-((t-t')/\tau)^b}\cdot\frac{d\varepsilon(t')}{dt'}\cdot dt' \quad , \quad \varepsilon(t)=\varepsilon_0\cdot\cos(\omega t) \quad or \quad \varepsilon(t)=\varepsilon_0\cdot e^{i\omega t} \quad , \quad \omega = 2\pi\cdot\upsilon$$

$$= 2\cdot G\cdot i\cdot\omega\cdot\varepsilon_0\cdot e^{i\omega t}\cdot\int_0^{\infty} e^{-(t'/\tau)^b}\cdot e^{-i\omega t'}\cdot dt' = 2\cdot G^*\cdot\varepsilon_0\cdot e^{i\omega t} = 2\cdot(G_1+iG_2)\cdot\varepsilon_0\cdot e^{i\omega t}$$

$$= 2\cdot G\cdot i\cdot\omega\cdot\varepsilon_0\cdot e^{i\omega t}\cdot\lim_{\gamma\to 0}\int_0^{\infty} e^{-(t'/\tau)^b}\cdot e^{-\gamma t'}\cdot e^{-i\omega t'}\cdot dt'$$

$$\approx 2\cdot G\cdot i\cdot\omega\cdot\varepsilon_0\cdot e^{i\omega t}\cdot\lim_{\gamma\to 0}\int_0^{\infty}\left(1-\left(\frac{t'}{\tau}\right)^b\right)\cdot e^{-\gamma t'}\cdot e^{-i\omega t'}\cdot dt' \quad \text{for high values of } \omega\tau$$

$$= 2\cdot G\cdot i\cdot\omega\cdot\varepsilon_0\cdot e^{i\omega t}\cdot\lim_{\gamma\to 0}\left[\frac{1}{\lambda+i\omega}-\frac{1}{\lambda+i\omega}\cdot\frac{\Gamma(1+b)}{((\gamma+i\omega)\tau)^b}\right]$$

$$= 2\cdot G\cdot\varepsilon_0\cdot e^{i\omega t}\cdot\left[1-\frac{\Gamma(1+b)}{(\omega\tau)^b}\cdot e^{-\frac{i\pi b}{2}}\right]$$

$$\Rightarrow \tan(\delta) = \frac{\Gamma(1+b)\cdot\sin\left(\frac{\pi b}{2}\right)}{(\omega\tau)^b}$$

Results by de Bast and Gilard 1964

Soda-lime-glass with the composition: 72% SiO_2, 14.5% Na_2O + K_2O, 12% CaO + MgO, 1.5% rest

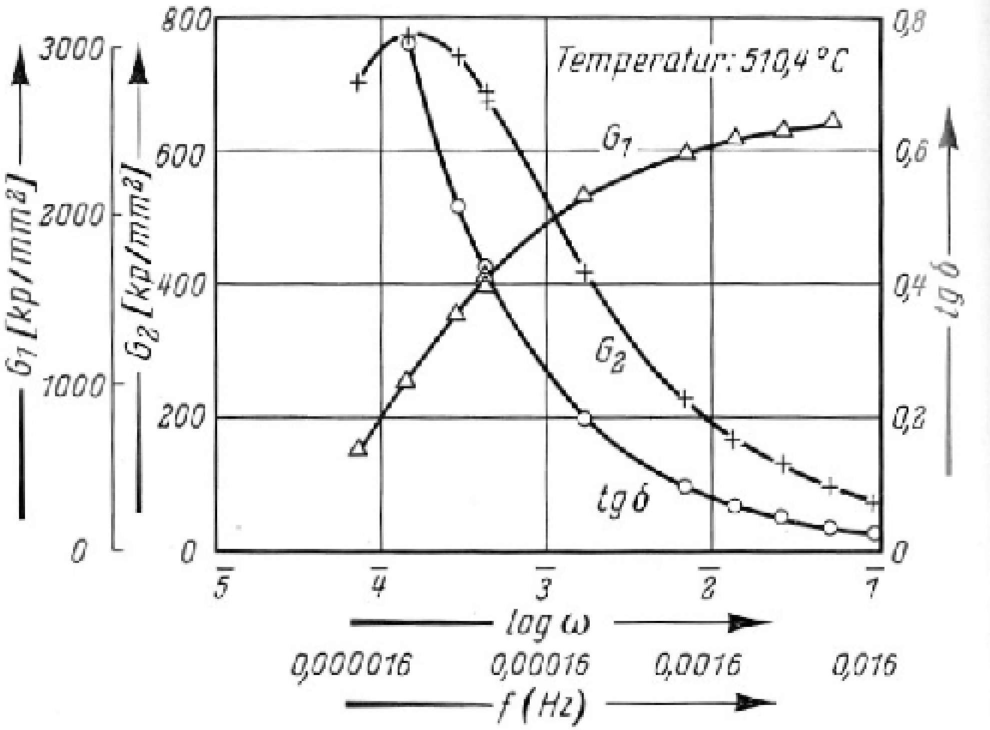


Bild 19. Komponenten des komplexen Moduls und des tg δ als Funktion der Frequenz bei 510,4 °C.

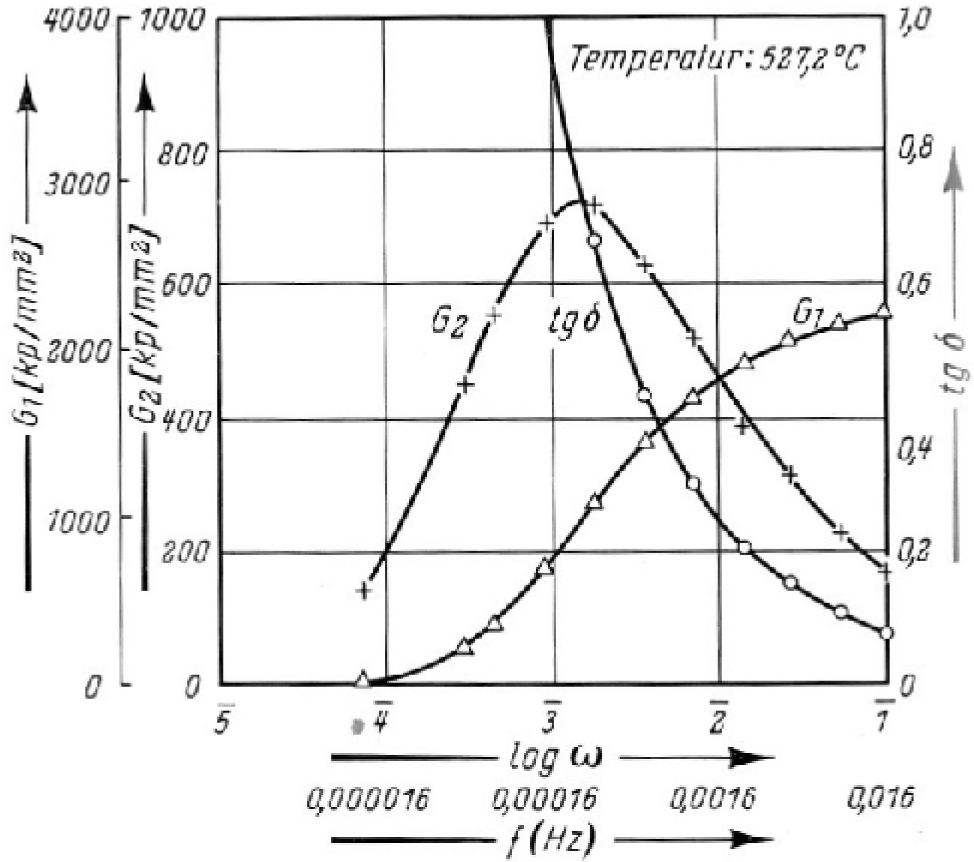


Bild 20. Komponenten des komplexen Moduls und des tg δ als Funktion der Frequenz bei 527,2 °C.

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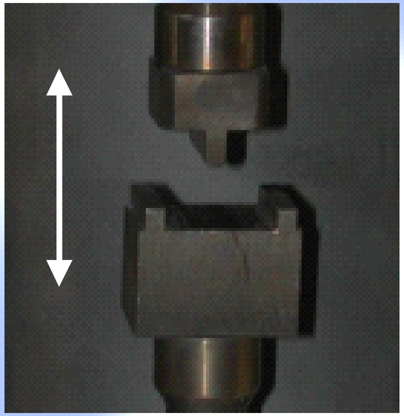
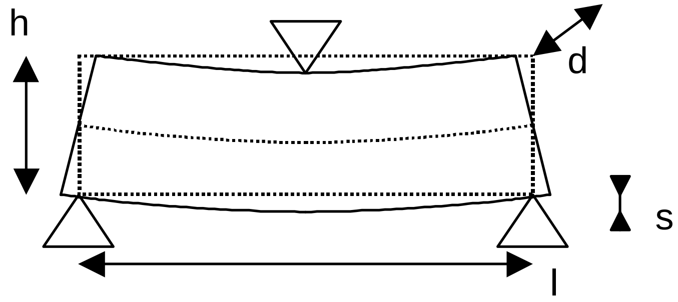
182 Glastechn. Ber. *Jean de Bast und Pierre Gilard:* 37. Jahrg., Heft 4

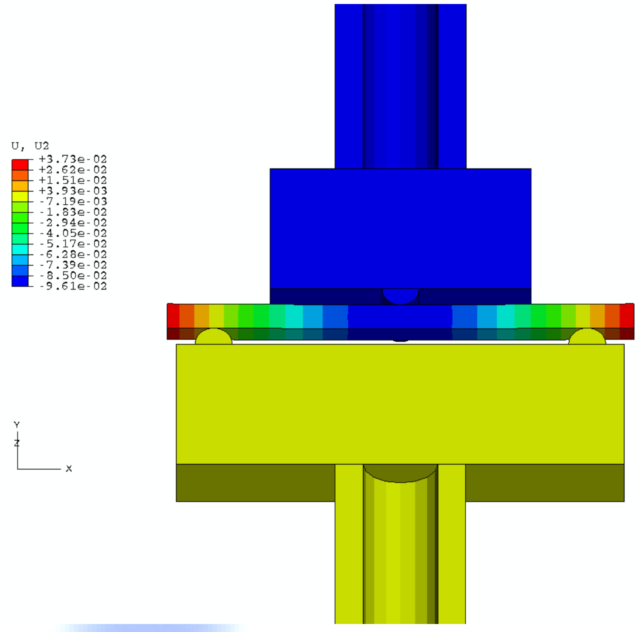
April 1964 *Das dynamische Verhalten eines dünnen Glasfadens bei sehr tiefen Frequenzen* Glastechn. Ber. 183

Forced oscillations / alternative to torsion: bending

First approach: oscillatory 3-point bending (preload plus oscillating load)







$$s = F \cdot \frac{l^3}{4 \cdot E \cdot d \cdot h^3} \propto \frac{1}{E}$$

However: the problem of mixing shear and bulk viscoelasticity is back.

With typical values for glass, i.e. E = 60 GPa and ν = 0.2, and $\frac{1}{E} = \frac{1}{3G} + \frac{1}{9K}$

one arrives at about 80% of the elongation being due to shear and the remaining 20% being due to compression/dilatation.

Forced oscillations / alternative to torsion: bending (continued)

Second approach: asymmetric 4-point bending I

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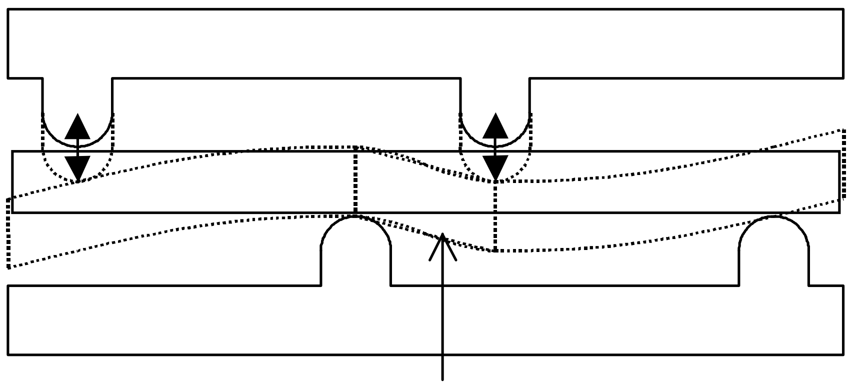
Idea for implementation of shear mode in DMA

• Increase sample height

Beside bending which is a composite mode consisting of shear and dilatation/compression (which will be called indirect shear and indirect dilatation/compression from here on), there is an additional direct shear which may be neglected for thin samples but not for thick samples.

• Asymmetric 4-point-bending



Analysis of asymmetric 4-point-bending:

• Size of bending part?

• Size of direct shear part?

• Size of indentation due to Hertzian pressing (of sample holder in sample)?

Forced oscillations / alternative to torsion: bending (continued)

Second approach: asymmetric 4-point bending II

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Balance of shear- and compression/dilatation-contributions

$$w_{bending} = \frac{a^2 b^2 F}{Edh^3} \cdot \frac{2a+b}{(a+b)^2} \quad + \quad \propto \frac{1}{E} = \frac{1}{3G} + \frac{1}{9K}$$

$$w_{direct\ shear} = \frac{6}{5Gdh} \cdot \frac{ab(2a+b)}{2(a+b)^2} \cdot F \quad + \quad \propto \frac{1}{G}$$

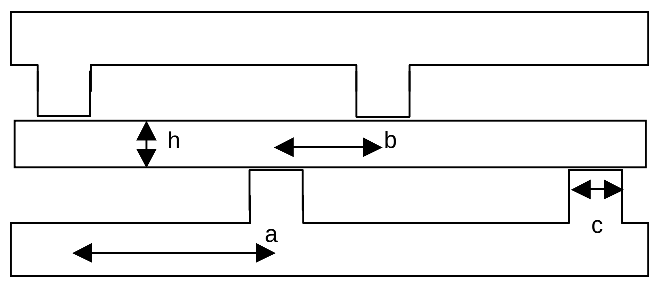
$$w_{elongation\ due\ to\ indentation} = \left[1 + \frac{a^2}{(a+b)^2}\right] \cdot \frac{2P}{\pi E} \cdot \left[ln\left(\frac{2h}{a_B}\right) - \frac{1+\nu}{2}\right] \quad \propto \frac{1}{E} = \frac{1}{3G} + \frac{1}{9K}$$

$$w_{total} = shear\ part + compression / dilatation\ part$$

Optimum geometry under condition (a, b > 1.5c) and for typical glass modules (E = 60GPa, ν = 0.2)
⇒ shear part = 87.7%

Allowing also for handling issues & manufacturing tolerances of sample holders ⇒

a = 10mm b = 12mm
c = 4mm h = 7mm,
shear part = 86%



Forced oscillations / alternative to torsion: bending (continued)

Second approach: asymmetric 4-point bending III

Check of Analytical Optimization by Finite Element Simulation

Basic Assumption:

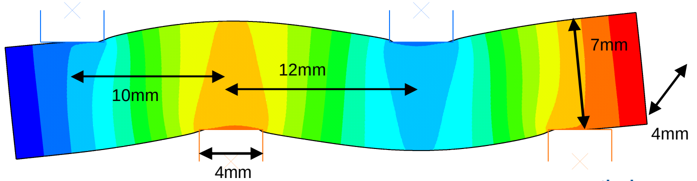
elongation $_{\text{total}}$ = elongation $_{\text{due to shear}}$ + elongation $_{\text{due to compression/dilatation}}$
$\propto$ 1/G $\propto$ 1/K

typical values from above: E = 60GPa, ν = 0.2 $\Leftrightarrow$ G = 25GPa, K = 33.33 GPa

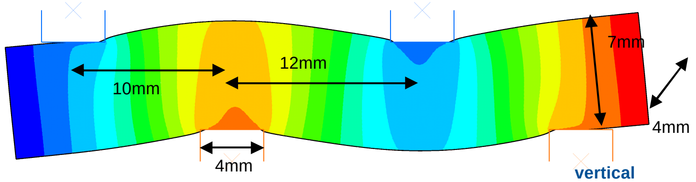
Now: make FE simulation with G = 25GPa, K = 33.33 GPa $\Rightarrow$ elongation $_{\text{total}}$
and with G = 25GPa, K = ∞ GPa $\Rightarrow$ elongation $_{\text{due to shear}}$

shear part = elongation $_{\text{due to shear}}$ / elongation $_{\text{total}}$

Check of the geometry from above:



FE-analysis of a glass with E=60GPa, ν=0.2
$\Leftrightarrow$ G=25GPa, K=33.33GPa.
Elongation = 100µm $\Leftrightarrow$ load = 9383N, i.e.
Elongation/load =0.0106575µm/N



FE-analysis of a glass with E=75GPa, ν=0.5
$\Leftrightarrow$ G=25GPa, K= ∞ .
Elongation = 100µm $\Leftrightarrow$ load = 11444N, i.e.
Elongation/load =0.0087382µm/N

0.0087382/0.0106575=0.82 => shear part is only 82%

Forced oscillations / alternative to torsion: bending (continued)

Second approach: asymmetric 4-point bending IV

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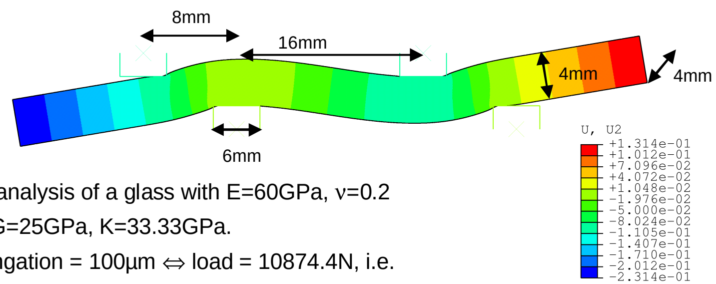
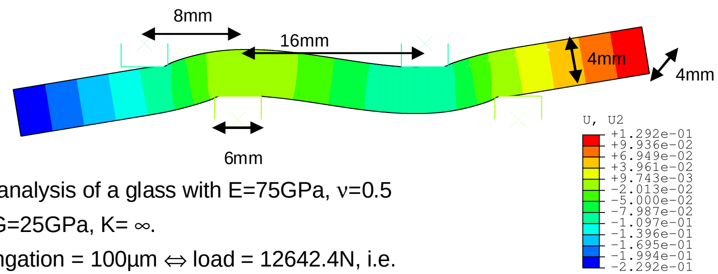
Optimum geometry according to Finite Element Simulation



FE-analysis of a glass with E=60GPa, ν=0.2
⇔ G=25GPa, K=33.33GPa.
Elongation = 100µm ⇔ load = 10874.4N, i.e.
Elongation/load =0.0091959µm/N

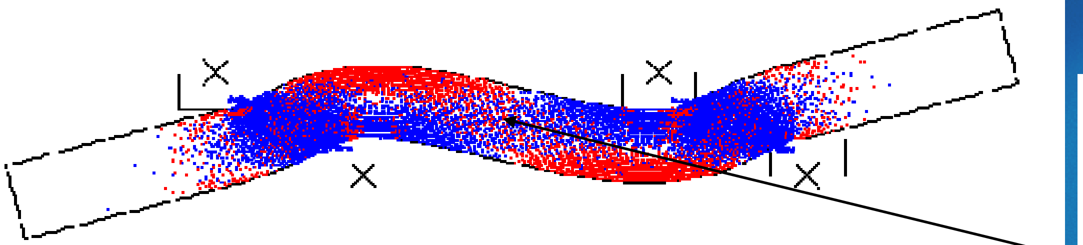
vertical displacement



FE-analysis of a glass with E=75GPa, ν=0.5
⇔ G=25GPa, K= ∞.
Elongation = 100µm ⇔ load = 12642.4N, i.e.
Elongation/load =0.00790989µm/N

vertical displacement

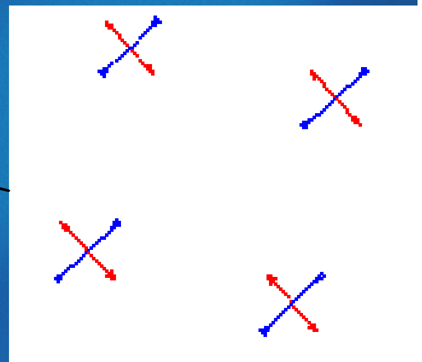
0.00790989/0.0091959=0.86 => shear part is 86%.



FE-analysis of a glass with E=60GPa, ν=0.2
⇔ G=25GPa, K=33.33GPa.
Elongation = 100µm ⇔ load = 10874.4N, i.e.
Elongation/load =0.0091959µm/N

S, Max. In-Plane Principal
S, Min. In-Plane Principal
S, Out-of-Plane Principal

main stresses (plane stress simulation)



Almost homogeneous shear in the middle.

Forced oscillations / alternative to torsion: bending (continued)

Second approach: asymmetric 4-point bending IV

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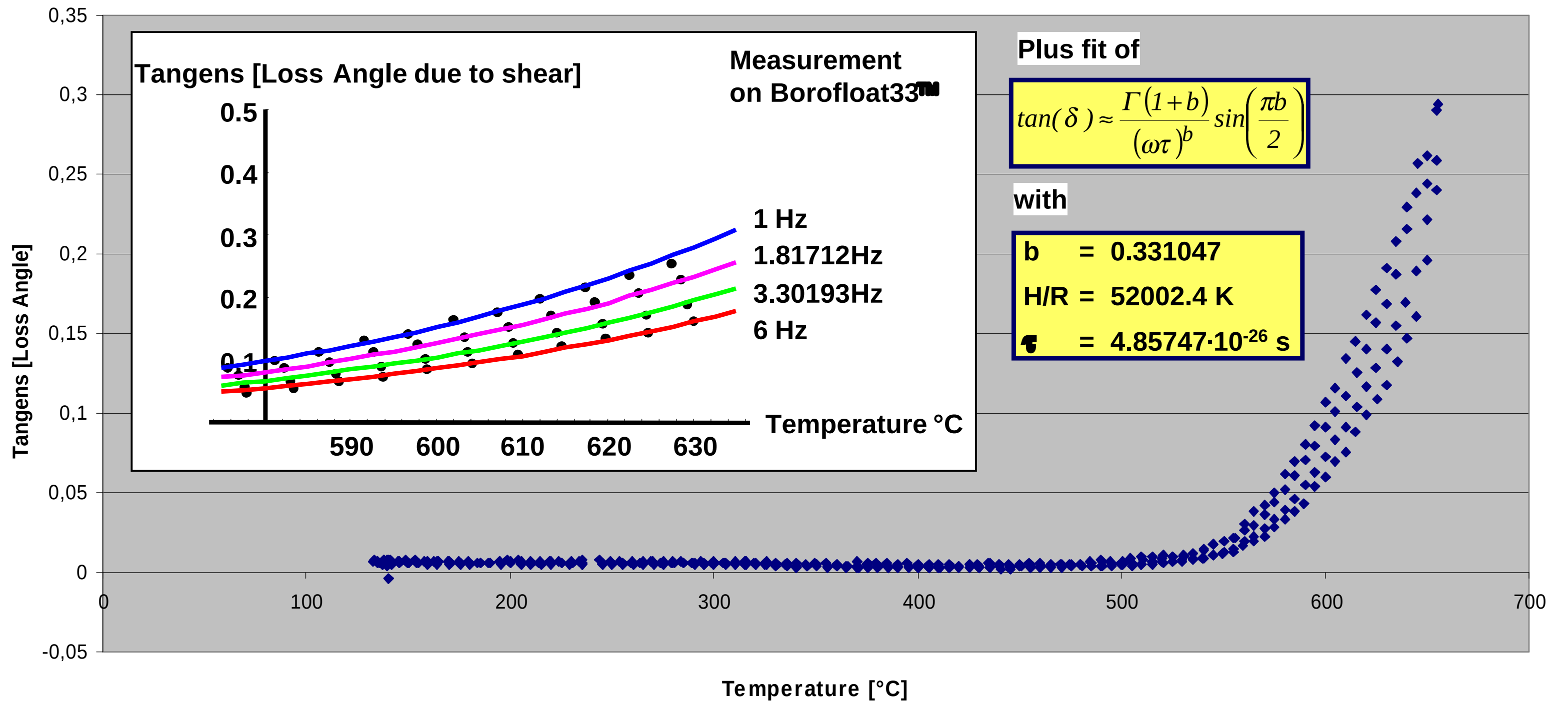
Measurement on Borofloat33™

Parameters: static load 400N, dynamic load 200N
dynamic elongation measured: 2µm
dynamic elongation calculated: direct shear 1.27µm + bending 2.16µm + indentation 0.78µm = 4.21µm

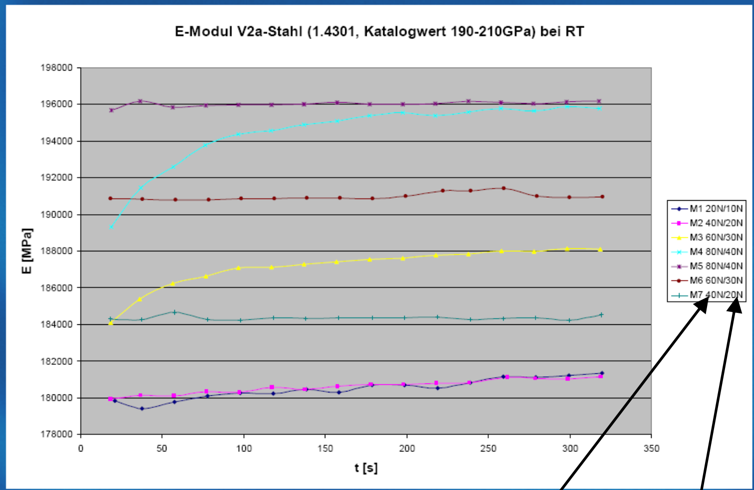
Dynamic-Mechanical Analysis on Borofloat33™ with an asymmetric four-point-bending load and the frequencies 1Hz (top curve), 1.8Hz, 3.3Hz, 6Hz (bottom curve)



Dynamic methods / general problem:

Measurement of sample or contact between sample and sampleholder?

Vergleich: Messung an V2a-Stahl (1.4301) / E-Modul



Interpretation:

Niedrige Kräfte => kein Formschluß, teilweise freies Federn und scheinbar niedrigerer E-Modul

Große Kräfte => erzwungener Formschluß, freies Federn nimmt ab, höherer Wert für E-Modul

Überschreiten der Plastizitätsgrenze => Probe wird auf Biegevorrichtung "gehämmert", realistischer Wert für E-Modul

Reduktion der Kräfte: teilweise Rückstellung und etwas freies Federn

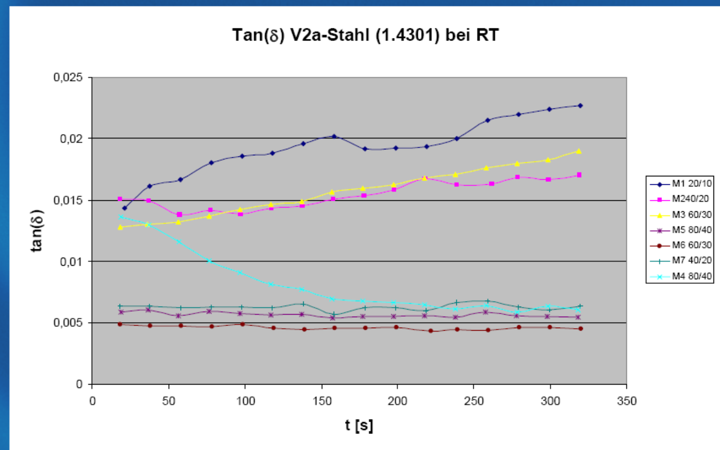
SCHOTT glass made of ideas

Measurement on steel: if preload and oscillating load exceed certain values, the values found for Young´s modulus and the loss angle become realistic: Better geometrical fit of sample and sampleholder; sample is "hammered" into the right shape.

preload

oscillating load

Vergleich: Messung an V2a-Stahl (1.4301) / tan(δ)



Interpretation:

Niedrige Kräfte => kein Formschluß, Reibung durch Öffnen und Schließen von Kontakten

Große Kräfte => erzwungener Formschluß, geringere Reibung

Überschreiten der Plastizitätsgrenze => Probe wird auf Biegevorrichtung "gehämmert", tan(δ) sinkt dabei; übrig bleibender Wert z.T. Hysterese-bedingt

Reduktion der Kräfte: zunächst weiter sinkender tan(δ) wg. Wegfall Hysterese

SCHOTT glass made of ideas

Some exercises:

1. Consider forced oscillations dynamic testing. What is the formula for tan(δ) in case b=1?

2. Consider again forced oscillations dynamic testing. Assume that a single simple Maxwell-model describes the shear viscoelasticity of glass well. At the temperature of the experiment, $\eta=10^{12}$Pa·s holds. The shear modules is 25GPa. What is Maxwell´s relaxation time? Which values will be measured for tan(δ) by the de Bast and Gilard apparatus if the frequency is, 1st, 1Hz, and, 2nd, 10Hz?

3. Consider b<1. By the de Bast and Gilard apparatus, you have measured tan(δ) as a function of ω. You make a plot Log(tan(δ)) vs. Log(ω). How can you obtain b from that?