

Special Topics in Relaxation in Glass and Polymers

Lecture 8: Viscoelasticity IV Important Application of Pre-Stressing

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Research and Technology Development

SCHOTT AG

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In the following, applications of relaxation processes in glass will be discussed.

Disclaimer:

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

To illustrate glass properties, references will be made to different companies. These references have been picked arbitrarily for educational reasons, copyright issues etc., not to provide a balanced view of the achievements of different companies.

In general, no liability is assumed concerning any scientific or technical use of any lecture of this course. In particular, any experimental work inspired by these notes has to be in accordance with safety and other rules which are not given here. Any technical work such as production of goods which may be inspired by these notes has to be in accordance with the safety and other rules which apply for manufacture and later use. These rules are not given here either. They may differ significantly from one location to another, as they do, for example, in case of fire-protection glazings.

Contact your local instructor for further information.

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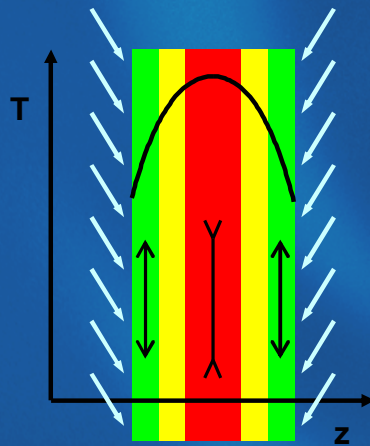
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Principle of pre-stressing (generation of permanent stress)

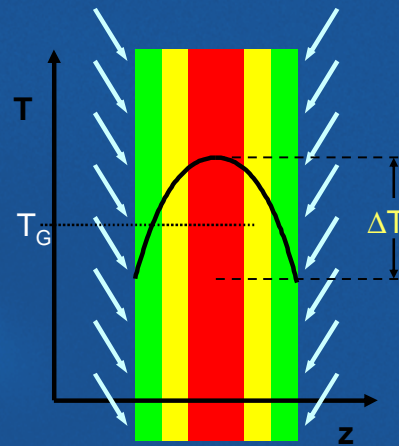
Heat transfer and temperature field determination in hot glass

Thermal stresses are caused by temperature differences during cooling

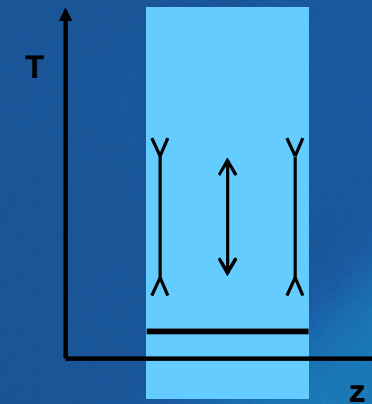
Example: plate cooling / pre-stressing



Cooling of hot glass plate:
Surface contracts relative to the core => tension at the surface, compression in the core.



Above glass transition temperature T_G :
All stresses relax, glass passes glass transition temperature without stresses, but with **temperature gradient ΔT**



At room temperature:

ΔT vanishes, core contracts relative to the surface => tension in the core, compression at the surface, not relaxing!

Crucial issues: ΔT and stress relaxation

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Prestressing: why?

Thermal stresses

Owing to the low thermal conductivity of glass (typically 0.9–1.2 W/(m·K) at 90 °C, or a minimum of 0.6 W/(m·K) for high-lead containing glasses), temperature changes produce relatively high temperature differences ΔT between the surface and the interior, which, depending on the elastic properties E (Young's modulus) and μ (Poisson's ratio), and on the coefficient of linear thermal expansion α , can result in stresses

$$\sigma = \frac{\Delta T \alpha E}{(1 - \mu)} \text{ [MPa]}.$$

In addition to the geometric factors (shape and wall thickness), the material properties α , E and μ decisively influence the thermal strength of glasses subjected to temperature variations and/or thermal shock. Thermal loads of similar articles made from different glasses are easily compared by means of the characteristic material value

$$\varphi = \frac{\sigma}{\Delta T} = \frac{\alpha E}{1 - \mu} \text{ [MPaK}^{-1}\text{]},$$

Prestressed glass: up to a certain limit, the surface is under compressive stress even in case of bending => no breakage.

If the limit is exceeded, the resulting fracture will set free the forces resulting from the tensile stress in the interior => complete destruction into little pieces, no big ones that might cause harm.

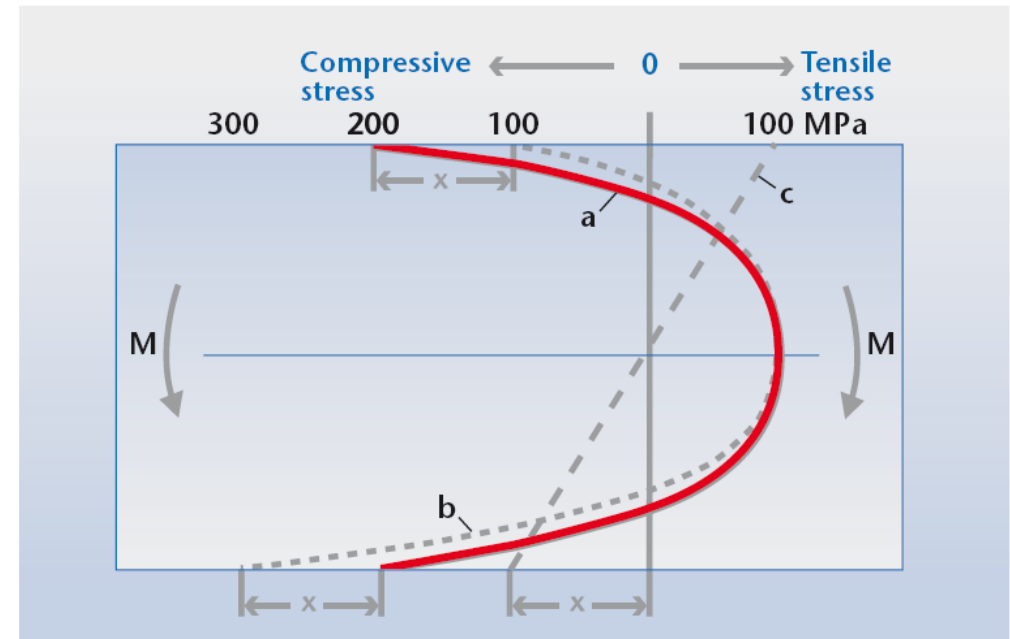
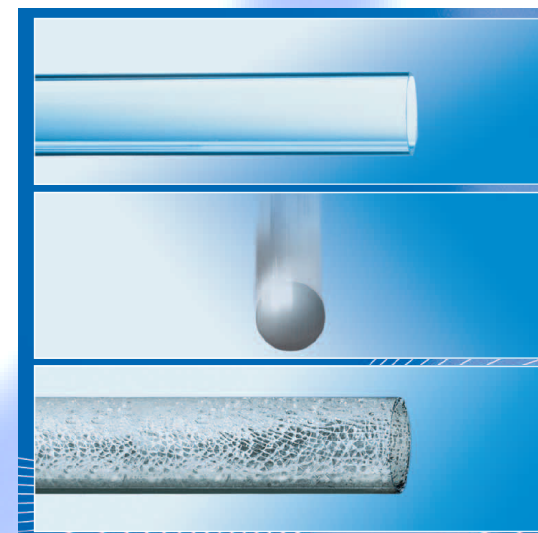


Fig. 15. Stress distribution across the thickness of thermally prestressed flat glass (a) without, (b) with additional bending M ; (c) stress distribution in bending without prestressing



From Schott AG,
Technical Glass
Handbook, available
via Internet.

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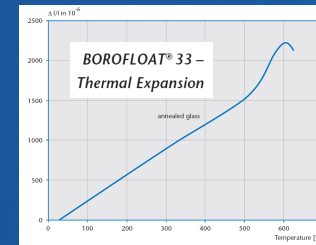
Thermally prestressed tubing of special glass

Simulation of prestressing

Heat transfer and temperature field determination in hot glass

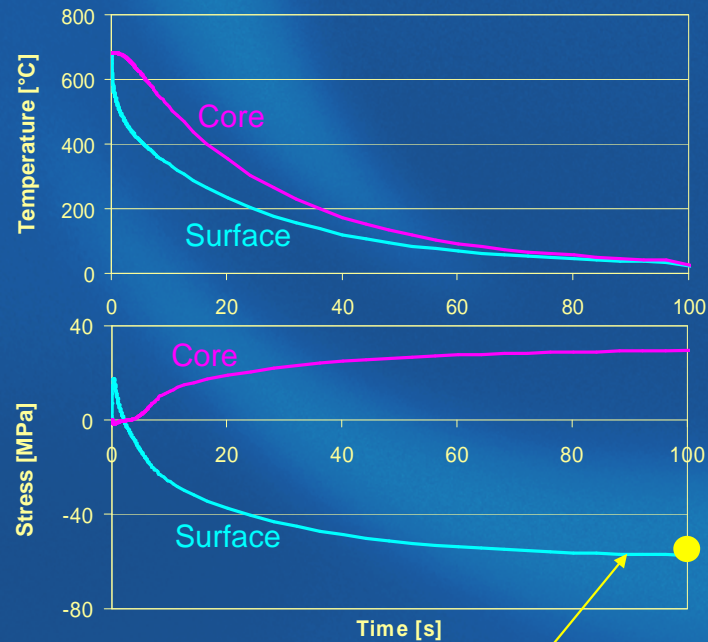
Simulation of plate cooling / pre-stressing

Quantitative (see comparison with differential stress refractometry) **calculation of temperature and stress history** in a 6.5 mm borosilicate glass plate during forced-convection cooling with heat transfer coefficient 500 W/(m²K)!



Specific Heat Capacity	C_p (20-100 °C)	0.83 kJ x (kg x K) ⁻¹
Thermal Conductivity	$\lambda_{(200 °C)}$	1.2 W x (m x K) ⁻¹

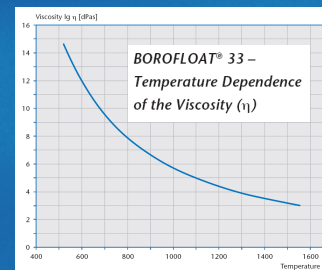
Calculated with Active Thermal Conductivity



Measurement with DSR

Because of the high heat transfer coefficient, the **surface cools rapidly (at 200K/s)**. Because of the small thermal conductivity, the core cools down slowly nevertheless. This gives rise to a **temperature gradient which is big (>200K)** when the glass is in the transition region and which gradually disappears on the further way to room temperature.

In the first 2-3s, there are **tensile stresses at the surface and compressive stresses in the core**. They relax fast; during the further process, **compressive stresses are built up in the surface and tensile ones in the core**.



Density (25°C)	ρ	2.2 g/cm ³	
Young's Modulus	E	64 kN/mm ²	(to DIN 13 316)
Poisson's Ratio	μ	0.2	(to DIN 13 316)
Knoop Hardness	HK _{0,1/20}	480	(to ISO 9385)
Bending strength	σ	25 MPa	(to DIN 52 292 T1)

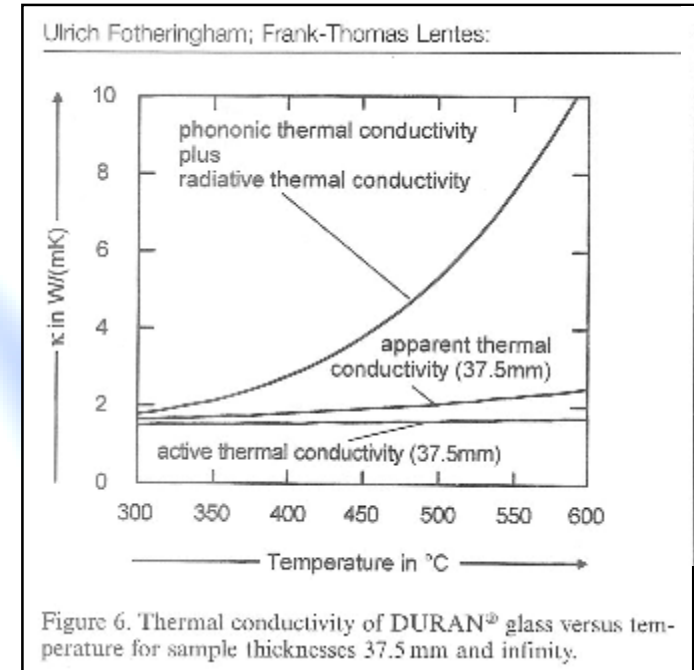


How to generate a high ΔT ?

1. Thermal conductivity is usually small, even in the glass transition range (despite thermal radiation – it is the so-called active thermal conductivity which counts here!)
2. Provide a high heat transfer coefficient h . Consider the steady state after ca. 5s when $\partial T/\partial t$ is the same in the core and at the surface.

$$\rho \cdot c_p \cdot \frac{\partial T}{\partial t} = -\kappa \cdot \frac{d^2 T}{dx^2} \quad \text{heat conduction equation}$$

ρ : density, c_p : specific heat, κ : thermal conductivity



Left side: heat loss per volume $V \Rightarrow \rho \cdot c_p \cdot \frac{\partial T}{\partial t} \cdot V = \rho \cdot c_p \cdot \dot{T} \cdot V = 2 \cdot h \cdot A \cdot (T_{surface} - T_{ambient})$
 A : surface area, d thickness

Right side: solve $const. = -\kappa \cdot d^2 T/dx^2$ for $T(x,t) \Rightarrow T(x,t) = T_{surface}(t) + \Delta T \cdot \left(1 - \frac{x^2}{(d/2)^2}\right)$
 x coordinate perpendicular to plate, $x=0$ in the middle

$$\Rightarrow 2 \cdot \kappa \cdot \Delta T \cdot V = \rho \cdot c_p \cdot \dot{T} \cdot V = 2 \cdot h \cdot A \cdot (T_{surface} - T_{ambient}) \Rightarrow \Delta T = \frac{h}{\kappa \cdot d} \cdot (T_{surface} - T_{ambient}) \quad **)$$

Stress freezes*) when $T_{core} = T_{surface} + \Delta T = T_G$. At this point $\Delta T = \frac{h/(\kappa \cdot d)}{1 + h/(\kappa \cdot d)} \cdot (T_G - T_{ambient})$

*) roughly speaking

**) ambient temperature means temperature of cooling medium

High ΔT : Pre-Stressing of low-expansion glass in oil-covered water

Glastech. Ber, 57 (1984) Nr. 9, S. 221–228 Werner Kiefer: Thermisches Vorspannen

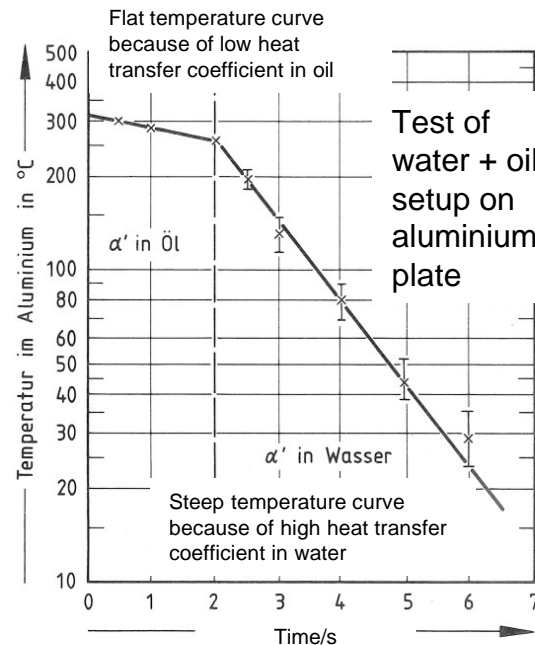
Tabelle 2. Wärmeübergangskoeffizienten thermischer Vorspannungsverfahren

Heat transfer coefficient for cooling by

- forced air
- diving in oil
- ... oil + CCl₄
- spraying of air+water
- diving in water+oil

thermisches Vorspannungsverfahren	Wärmeübergangskoeffizient in W/(m ² K)
Anblasen mit Luft	100 → 300
Mineralöle	200 → 600
Mineralöle mit CCl ₄	400 → 1500
Sprühen mit Luft-Wasser	500 → 1500
ölüberschichtetes Wasser	500 → 5000

Idea: lower heat transfer coefficient in the first 2 seconds (when the glass penetrates the oil) to avoid high intermediary tensile stress in the surface; higher heat transfer coefficient to maximize $T_{\text{core}} - T_{\text{surface}}$ when T_{core} reaches T_G (at that time the glass shall have reached the water).



7. Wärmeübergangskoeffizienten in ölüberschichtetem Wasser.

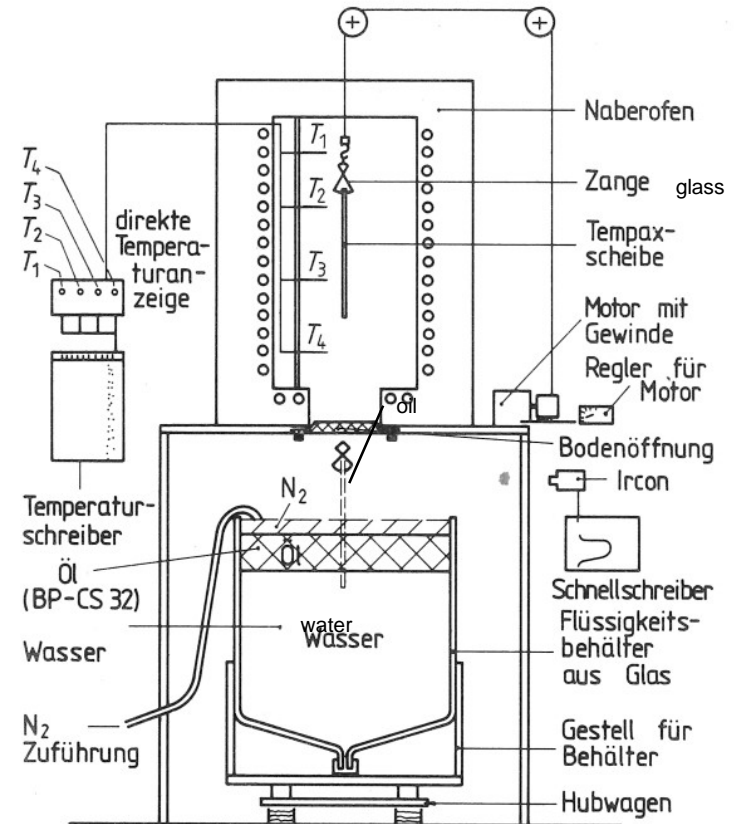


Bild 6. Versuchsanlage für das thermische Vorspannen von Glas in ölüberschichtetem Wasser.

Thus, compressive surface stress of 100 MPa is possible for glass with CTE = 3.3ppm/K.

Some exercises:

1. In the crucial phase of prestressing, a temperature gradient of $\Delta T = 100\text{K}$ (precisely: average temperature minus surface temperature) is induced in the glass sheet. Let the thermal expansion coefficient α be 8.4ppm/K , Young's modulus be 70 GPa , and Poisson's ratio be 0.2 (all typical values for soda-lime-glass). Which compressive stress do you expect for the surface?
2. Consider a glass plate, thickness 10mm , density 2500kg/m^3 , specific heat 1000J/(kgK) . The sheet has 600°C . It has to be cooled at 10K/s . Which heat transfer coefficient is required if the cooling medium has 0° ?
3. The above "crucial phase" is reached when the core temperature runs through T_G (roughly speaking; we assume the very simple model of an infinitely fast relaxation at $T > T_G$ and no relaxation at $T < T_G$ here). The thermal conductivity shall be $\kappa = 1\text{W/mK}$, the heat transfer coefficient $h = 200\text{W/(m}^2\text{K)}$, the temperature of the cooling medium 0°C . T_G is 550°C . What is the ΔT between the core and the surface in this crucial phase? If you do not distinguish between this ΔT and the quantity "average temperature minus surface temperature", what is the resulting compressive stress at the surface?

Generation and relaxation of stress

Starting point: for elongational viscoelasticity, the formulas look the same as for shear viscoelasticity (why: there are immediate response, delayed elasticity, and creep; stresses will completely relax for $t \rightarrow \infty$ because all intermediary compressions/dilatations are removed via viscous flow)

$$\sigma(x, t) = E_0 \cdot \int_0^t e^{-((t-t')/\tau)^b} \cdot \frac{\partial \varepsilon(x, t')}{\partial t'} \cdot dt'$$

starting point

$$\rightarrow \sigma(x, t) = E_0 \cdot \int_0^t e^{-(t-t')/\tau} \cdot \frac{\partial \varepsilon(x, t')}{\partial t'} \cdot dt'$$

simplification: single Maxwell instead of Kohlrausch

$$\rightarrow \frac{\partial \sigma(x, t)}{\partial t} = E_0 \cdot \frac{\partial \varepsilon(x, t)}{\partial t} - \frac{\sigma(x, t)}{\tau}$$

from integral to differential equation to have an equation suited for non-isothermal processes

$$\rightarrow \frac{\partial \sigma(x, t)}{\partial t} + \frac{\sigma(x, t)}{\tau} = E_0 \cdot \left(\frac{\partial \varepsilon(x, t)}{\partial t} - \alpha \frac{\partial \Delta T(x, t)}{\partial t} \right)$$

again to prepare for non-isothermal processes: no stress from strain due to local thermal expansion*)

$$\rightarrow \frac{\partial \sigma(x, t)}{\partial t} + \frac{\sigma(x, t)}{\tau} = E_0 \cdot \left(\frac{d\varepsilon(t)}{dt} - \alpha \frac{\partial \Delta T(x, t)}{\partial t} \right)$$

assumption of plate with infinite dimensions: x-dependence of ε would lead to infinite dislocations

$$\rightarrow \frac{\partial \sigma(x, t)}{\partial t} + \frac{\sigma(x, t)}{\tau} = \frac{E_0}{1-\nu} \cdot \left(\frac{d\varepsilon(t)}{dt} - \alpha \frac{\partial \Delta T(x, t)}{\partial t} \right)$$

to take into account the effect arising from strain in the other dimension of the plate

$$\int_{-d/2}^{d/2} \sigma(x, t) \cdot dx = 0$$

results also from the infinite dimensions of the plate

***) however, stress will be caused by imposed strain that results from thermal expansion elsewhere; ΔT is the difference to the homogeneous initial temperature.**

Generation and relaxation of stress (continued)

$$\frac{\partial \sigma(x,t)}{\partial t} + \frac{\sigma(x,t)}{\tau} = \frac{E_0}{1-\nu} \cdot \left(\frac{d\varepsilon(t)}{dt} - \alpha \frac{\partial \Delta T(x,t)}{\partial t} \right), \quad \int_{-d/2}^{d/2} \sigma(x,t) \cdot dx = 0$$

In general, $\tau = \tau(x,t)$.

Consider the above equations for the elastic case ($\tau = \infty$). In this case, one can apply the integral on both sides of the equation, exchange integration and differentiation and obtains:

$$\frac{d\varepsilon(t)}{dt} = \alpha \cdot \frac{d\overline{\Delta T}(t)}{dt} \quad \text{with the last expression giving the derivative of the temperature average at } t.$$

Consider now the viscous case, i.e. $\frac{\partial \sigma(x,t)}{\partial t} \ll \frac{\sigma(x,t)}{\tau}$

With $\tau = \eta_e / E_0$ one gets (neglecting $1-\nu$): $\sigma(x,t) = \eta_e \cdot \left(\frac{d\varepsilon(t)}{dt} - \alpha \frac{\partial \Delta T(x,t)}{\partial t} \right)$

Consider now the start of a prestressing process when the temperature and, consequently, the

viscosity are homogeneous. In this case $d\varepsilon(t)/dt = \alpha \cdot d\overline{\Delta T}(t)/dt$ holds also and one has:

$$\sigma(x,t) = \eta_e \cdot \alpha \cdot \left(\frac{d\overline{\Delta T}(x,t)}{dt} - \frac{\partial \Delta T(x,t)}{\partial t} \right)$$

This explains why the start of a prestressing process at too low a temperature leads to breakage.

Some exercises:

1. The prestressing starts at $T_G + \delta T$. T_G is 550°C . At T_G , the elongational viscosity η_e shall be $3 \cdot 10^{12} \text{Pa}\cdot\text{s}$. Assume that η_e decreases by a factor of "10" if the temperature is raised by 20K. The thermal expansion coefficient is 8.4ppm/K. At the start, the average cooling rate shall be 5K/s. The surface cooling rate shall be 100K/s. Which tensile stress is to be expected at the beginning of the process, at the surface, if the cooling starts at T_G ? Which is the minimum dT that will keep the initial tensile stress at the surface below 20MPa?

Example: prestressed borosilicate glass for fire protection in EU

Feature 1: low thermal expansion coefficient. In case of fire, there is less deformation because of temperature gradients than for a high thermal expansion glass.

Feature 2: prestressing. In case of fire, the glass can stand the above deformation (less than for a high thermal expansion glass, but non-zero) to a certain extent because of the prestressing.

Feature 3: prestressing. In case of no fire, the glass acts like an “ordinary” safety glass.



Even in simple frame constructions borosilicate glass resists high temperatures and has long staying power.

Source: various brochures from SCHOTT AG on Pyran (available via internet).



PYRAN® S meets the requirements for single-pane safety glass according to DIN 1249-12 and/or DIN EN 13024-1. If PYRAN® S is broken under heavy impact it shatters into small and relatively harmless granules, conforming to regulation for accident insurance and for health and safety at work.

← DIN: Deutsche Industrie Norm (Germany). Note again that fire protection regulations and, consequently, fire protection glazings differ substantially from country to country! Fire protection glazings for the US look different! Also, in one country or region different systems may be available which offer different levels of fire protection. This is not subject of the course so that no liability is assumed concerning any scientific or technical use of the material presented here. See above disclaimer.

