

IMI-NFG Course on Processing in Glass Spring 2015

(available online www.lehigh.edu/imi)

<u>Lecture 9</u>: Annealing and Tempering

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Outline of this lecture

1. Annealing of glass

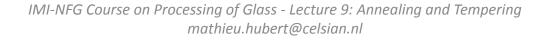
- Introduction Principles
- Annealing in industrial glass production

Photo: Erik Skaar

2. Tempering

- Principles
- Tempering in industrial glass production
- Tempered vs. Heat strengthened glass

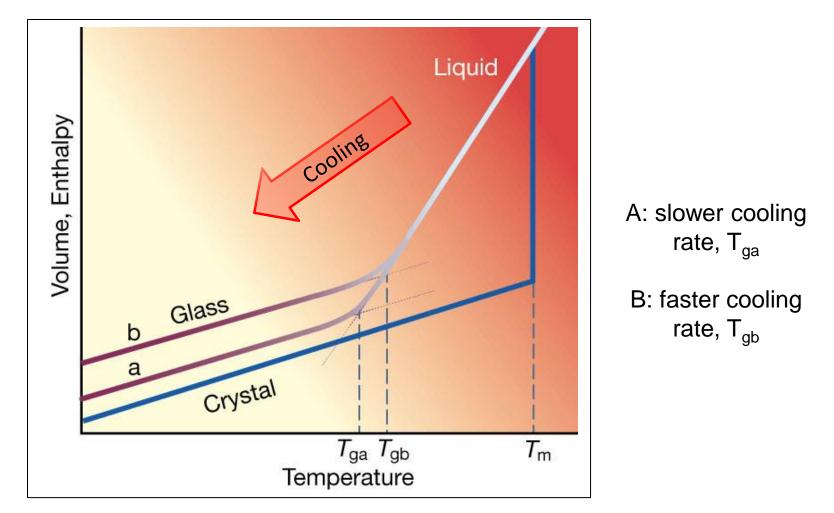






Introduction

Just a word on glass science – The glassy state



From P. Debenedetti and F. Stillinger, "Supercooled liquids and the glass transition", Nature 410, 259-267(March 2001)

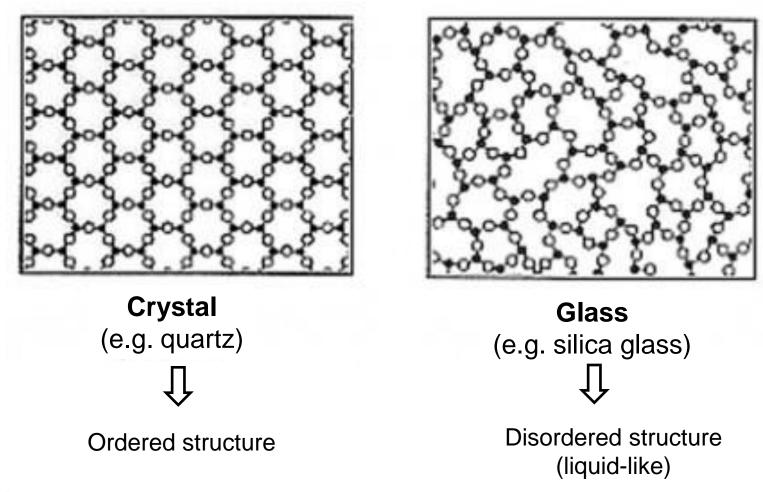


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Introduction

Just a word on glass science – The glassy state



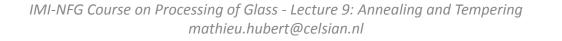


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Introduction

- **Rapid cooling** of the melt is necessary to obtain a glass (avoid crystallization)
- This rapid cooling will generate **constraints** within the glass, which will are detrimental for the mechanical properties
- This constraints can be **relaxed by careful thermal treatment**
- This relaxation of constraints is called **annealing** of the glass
- **Good annealing** is extremely important to produce good commercial glasses and for their durability
- Non-annealed or poorly annealed glasses will be subject to low resistance to cracks/failure under small thermal or mechanical shocks



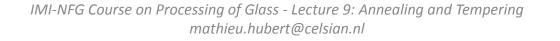




Annealing

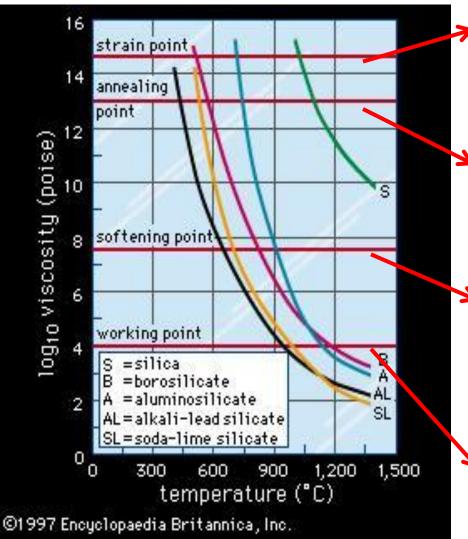
- All along the cooling process, the viscosity of the glass increases, from a low-viscosity melt, to a rigid material with a higher viscosity
- A certain "degree of freedom" is necessary for the glass to relax the constraints caused by the rapid cooling (re-arrangements in the glass structure)
- Good annealing can only be obtained in a relatively narrow range of temperatures (thus of viscosity)
- For a good annealing of the constraints, the viscosity of the glass should be:
 - ✓ Not too high (constraints cannot be released anymore)
 - \checkmark Not too low (the glass will not retain its shape)







Characteristic temperatures vs. viscosity



Strain point

 $\eta = 10^{14.5}$ Poise (10^{13.5} Pa.s)

Internal stresses are relieved in ~ 15 h

- Annealing point
 - $\eta = 10^{13.4}$ Poise (10^{12.4} Pa.s)

Internal stresses are relieved in ~ 15 min

Softening point

 $\eta = 10^{7.65}$ Poise (10^{6.65} Pa.s)

Glass deforms under its own weight at a rate of 1mm/min

• Working point $\eta = 10^4$ Poise (10³ Pa.s)





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Annealing point and stress point

Annealing point $\eta = 10^{12.4} \text{ Pa-s}$

• At this temperature, the internal thermal stresses present in the glass are relieved by **viscous relaxation** within 15 minutes. In order to relieve a glass product from its internal stresses the glass has to be heated to just above the annealing point and subsequently cooled down slowly.

Strain point $\eta = 10^{13.5}$ Pa·s.

- Below this temperature relieving the internal stresses is practically impossible (at the strain point it may last about 15 hours)
- Between the annealing and the strain point glass products should be cooled down gradually, slowly and uniformly in order to avoid the formation of internal stresses, due to temperature gradients

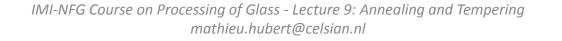




Temperature profile and stresses

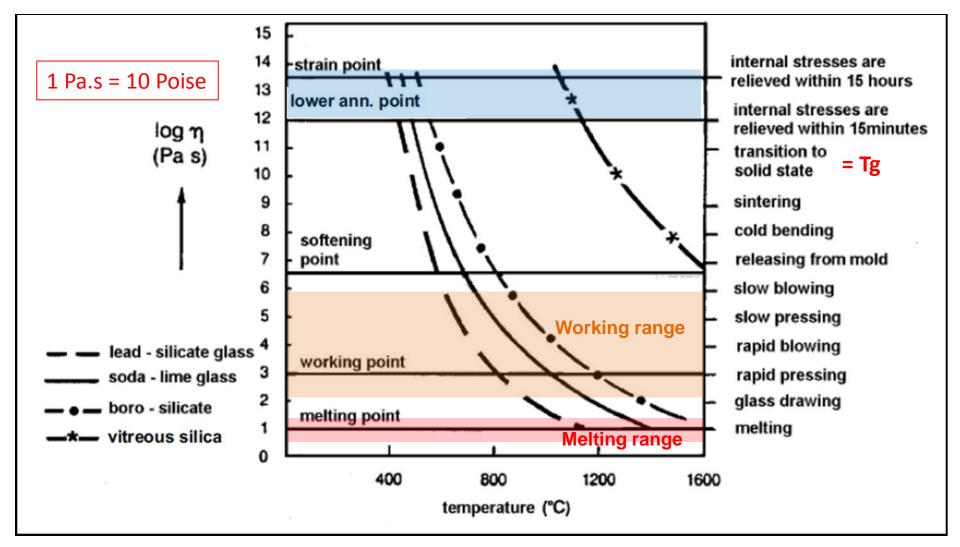
- Stresses acquired during cooling and remaining from temperatures above the **strain point** are **permanent stresses** (unless annealed)
- Stresses acquired during cooling below the strain point are considered temporary stresses (but can still lead to failure in case of a too important thermal shock)
- The goal of the **annealing process** is to **relieve the permanent stresses** created by the fast cooling below the strain point which occurred during the forming process of the glass
- To avoid creation of permanent stresses, the cooling of the glass should be slow in the temperature (viscosity) range between the annealing point and the strain point







Viscosity – Temperature profile of glasses



In blue: Critical temperature range for annealing



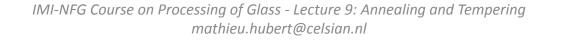
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Annealing in continuous glass furnaces

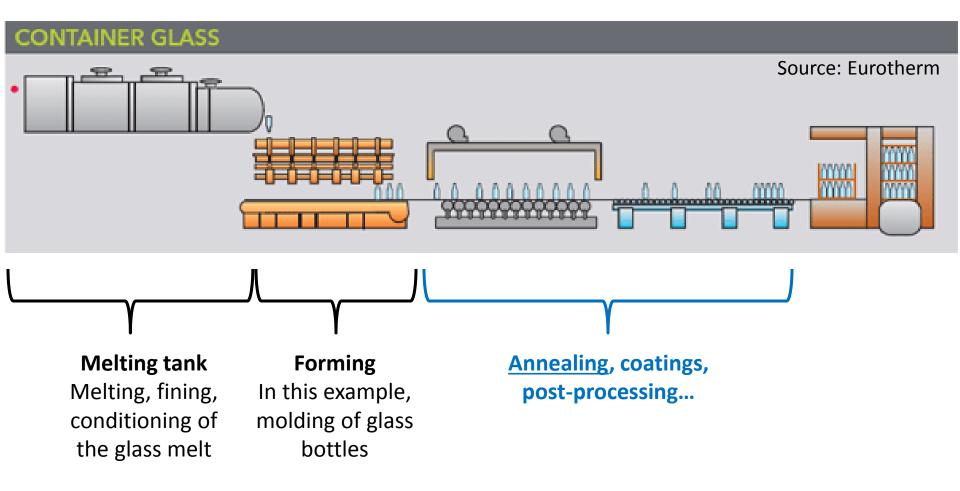
- Right after the forming process (e.g. molding for container glasses), a rigid glass article is obtained
- The article did not experience a homogeneous cooling and a lot of stresses are generated
- To reduce these stresses, the articles are brought to a temperaturecontrolled kiln, or Lehr, for annealing
- The process from the forming of the article to the annealing Lehr is **continuous**, the articles are conveyed on belts or rollers
- The **temperature profile** in the lehr must be **controlled** for an efficient annealing
- After annealing (at the end of the lehr), the articles are continuously conveyed to further processing steps (coatings, cutting,...)



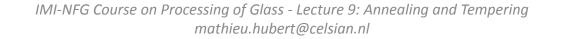




Example: forming of glass bottles



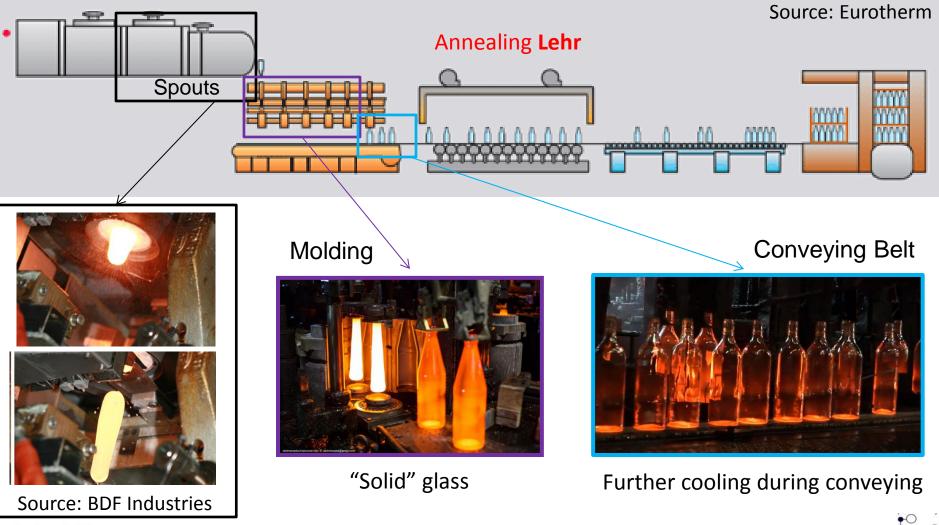






Example: forming of glass bottles

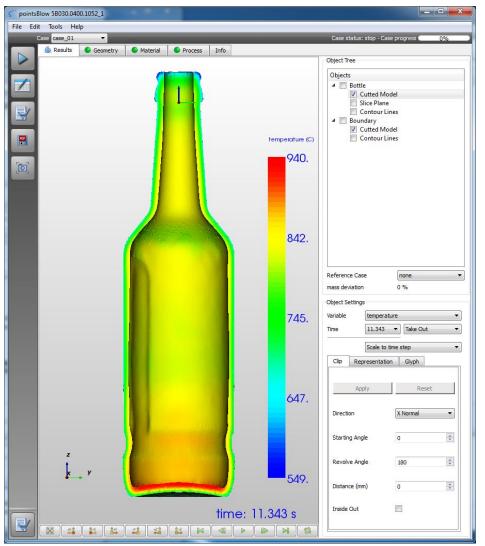
CONTAINER GLASS





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Temperature distribution right after forming



- Right after forming (and before annealing), the temperature distribution is not uniform throughout the glass article
- Different parts cooled down at different rates, which can result in constraints and residual stresses

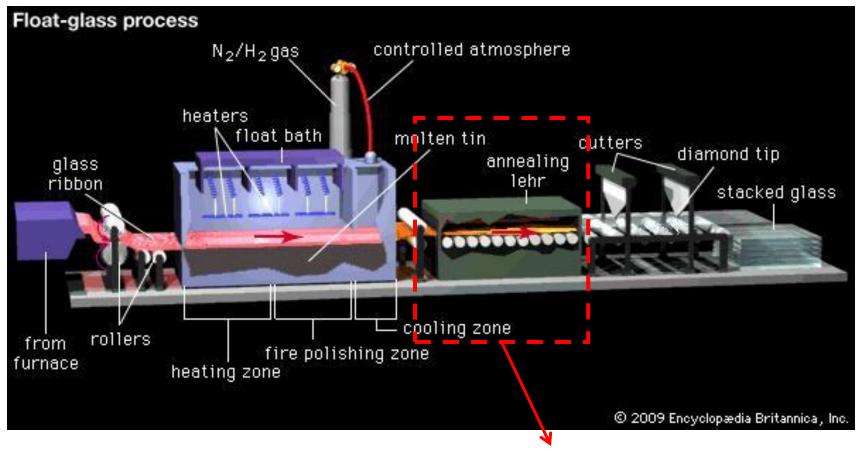
Source: NOGRID, www.nogrid.com/index.php/en/product/nogrid-points-blow1



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Illustration of the continuous process for flat glass

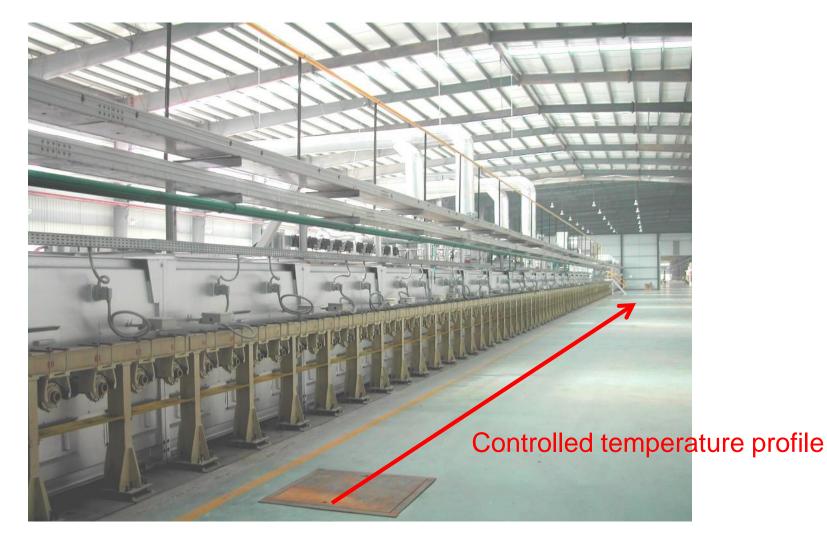


Source: http://www.britannica.com

Controlled temperature profile

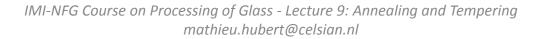


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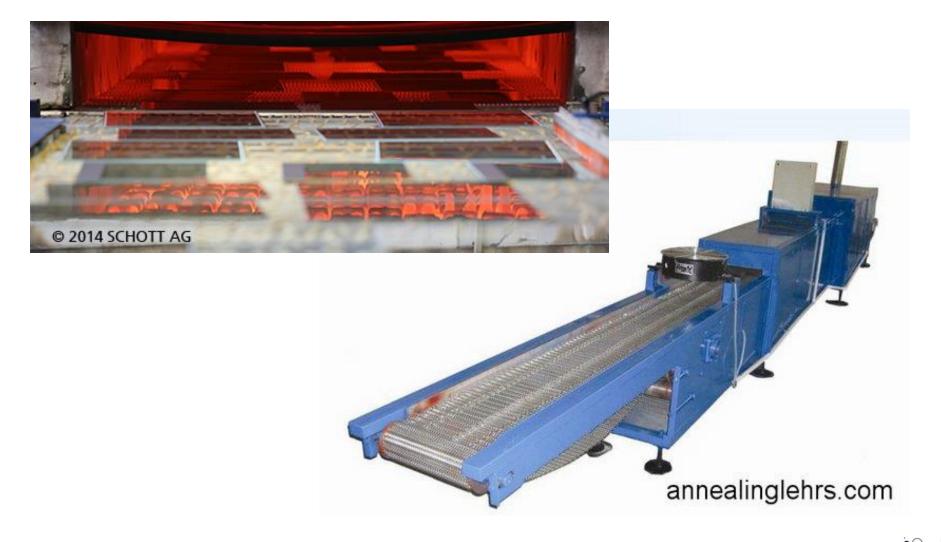


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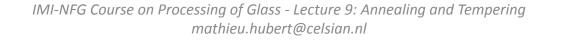


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- The viscosity-temperature profile for a glass depends on its composition
- Thus, the annealing point and strain point depend on the type of glass produced
- Different articles with different shapes (e.g. bottles, tubes, plates...) and different characteristics (e.g. thickness, diameter, ...) will have different thermal behavior
- All these parameters have to be taken into account when designing the annealing lehr
- The goal for an annealing lehr: it should be as short as possible while guaranteeing an efficient annealing
- Equations exist to calculate the best temperature profile for the lehr







Temperature profiles and stresses in glass

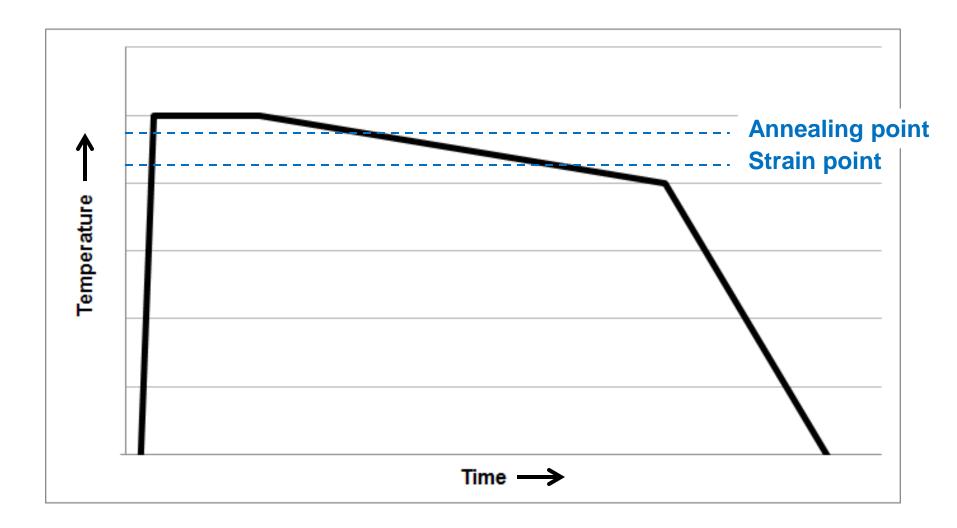
- During cooling of glass, internal temperature gradients develop, depending on cooling rate & internal thermal equalization within the glass
- The internal temperature gradients will eventually lead to stresses
- The stress in the glass can be calculated from the cooling rate, properties of the glass and shape of the article
- In return, the "best" cooling rate can be calculated for a maximum allowable residual stress in the glass article
- Keep in mind: the goal of (industrial) annealing is to minimize the stresses in the glass article in a duration as short as possible (annealing = heating = energy consumption = costs)







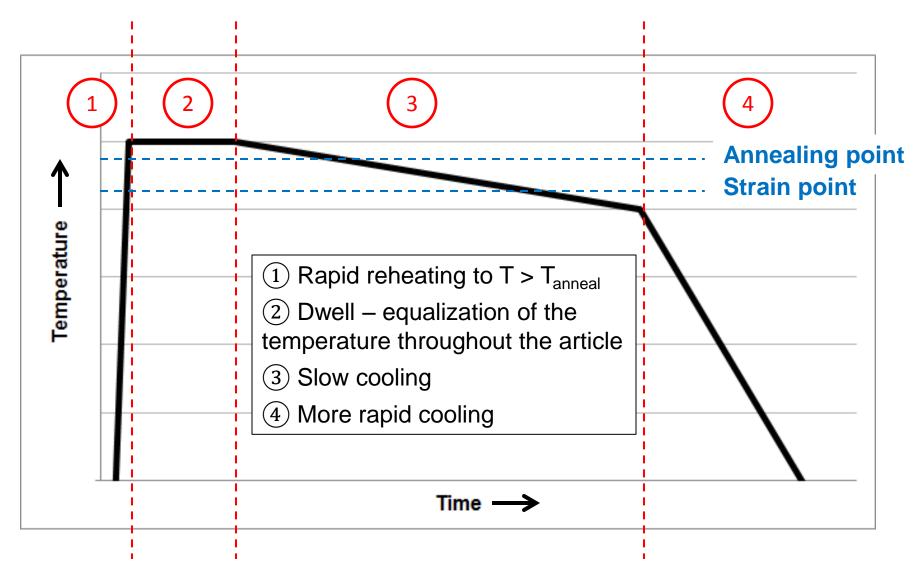
Temperature profile in annealing Lehr







Temperature profile in annealing Lehr

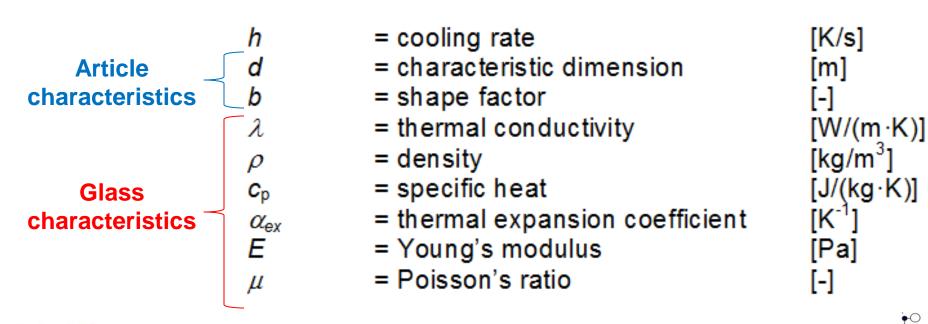




Temperature profiles and stresses in glass

The relation for cooling rate h from above the annealing point to below strain point and generated permanent thermal stress is given by:

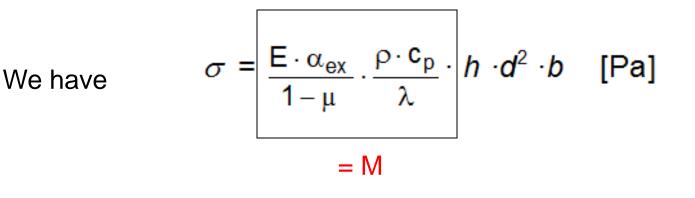
$$\sigma = \frac{\mathbf{E} \cdot \alpha_{\text{ex}}}{1 - \mu} \cdot \frac{\rho \cdot \mathbf{c}_{p}}{\lambda} \cdot \mathbf{h} \cdot \mathbf{d}^{2} \cdot \mathbf{b} \quad [\text{Pa}]$$





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Temperature profiles and stresses in glass



With

$$M = \frac{\mathbf{E} \cdot \alpha_{ex}}{1 - \mu} \cdot \frac{\rho \cdot \mathbf{C}_{p}}{\lambda} \quad \text{in MPa·s·K-1·m-2}$$

(σ in MPa)

Thus

$$\sigma = M.h.d^2.b \qquad \Longrightarrow \qquad h = \frac{\sigma}{M.d^2.b}$$



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(h in K/s)

MI NFG

Glass characteristics

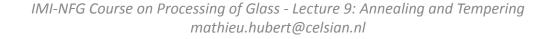
Survey of the expansion coefficient α_{ex} of some familiar glass types

	α _{ex 0-300°C} [K ⁻¹]	T _g [°C]
Soda-lime-silica glass	92 x 10 ⁻⁷	520-580
Pyrex borosilicate	33 x 10 ⁻⁷	565
E-glass	60 x 10 ⁻⁷	670
Vycor (97 SiO ₂ , 3 B ₂ O ₃)	8 x 10 ⁻⁷	910
Vitreous Silica	5 x 10 ⁻⁷	1100

For a soda-lime-silica glass, the factor M is equal to 1.2x10⁶ MPa.s.K⁻¹.m⁻² which gives:

 σ = 1.2 x 10⁶ h.d².b [MPa] with d in m and σ in MPa







Characteristic dimensions and shape factor

$$\sigma = M.h.d^2.b$$
 With d: characteristic dimension

Characteristic dimension "d"

- d = thickness for one-sided cooled plate
- d = 0.5 thickness for double-sided cooled plate
- d = radius for spheres and cylinders
- d = $\sqrt{(d.L)}$ for pots and bottles with wall thickness d and bottom thickness L
- d = L for pots and bottles with thick bottoms (L = bottom thickness)





Characteristic dimensions and shape factor

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$$\sigma = M.h.d^2.b$$
With d: characteristic dimension
b: shape factor
$$\frac{Shape factor "b"}{b = 0.336 \text{ for flat plates}}$$

$$b = 0.126 \text{ for massive cylinders}$$

$$b = 0.066 \text{ for spheres}$$

$$b = 0.3 \text{ for hollow products}$$

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Example annealing (cooling) rate

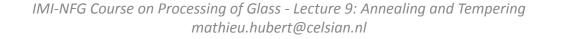
When a maximal permanent stress of 1 MPa is permitted after cooling, the limit for the cooling rate h for a glass with $M = 0.8 \text{ MPa} \cdot \text{s} \cdot \text{K}^{-1} \cdot \text{m}^{-2}$ becomes:

h
$$\leq \frac{1}{0.8 \times 10^6 . d^2 . b}$$
 [K/s] $h = \frac{\sigma}{M. d^2. b}$

h ≤ 6 K/min. for 10 mm plate glass cooled double-sided

- h ≤ 36 K/min. for 4 mm plate glass cooled double-sided
- h \leq 36 K/min. for 2 mm hollow glass cooled one-sided



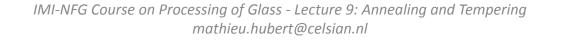




Example annealing (cooling) rate

- The **cooling rate** h must be maintained (should not exceed the above given limits) in the critical annealing range between T_{ap} and T_{str} , because this is the determining range for the build-up of a permanent stress.
- Below this range a faster cooling rate is allowed, because this will cause only a temporary stress
- However fracture or crack formation during cooling, caused by too large value of ΔT still to be prevented
- The limitations on the cooling rates will determine the needed length and temperature profiles in the annealing lehr
- As an example: the annealing range for soda-lime-silica glass is about 20-30°C (± 515 - 545°C).

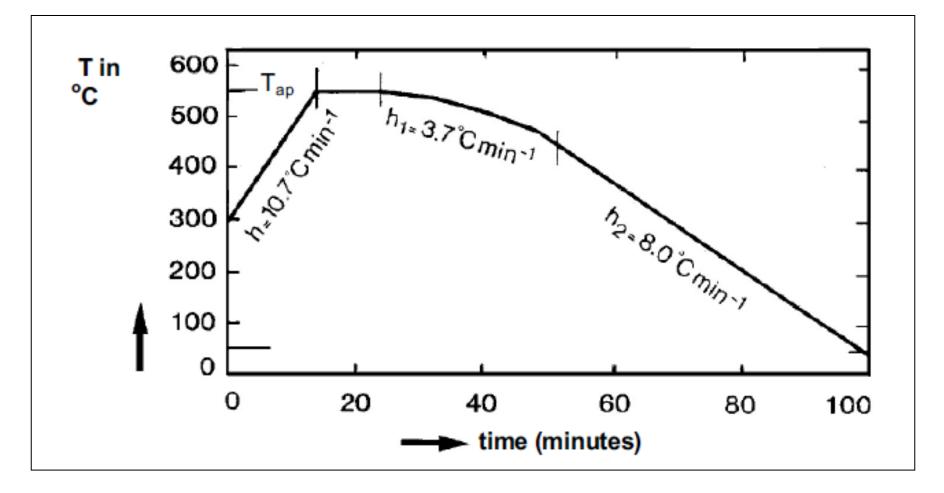






Temperature profile in annealing Lehr

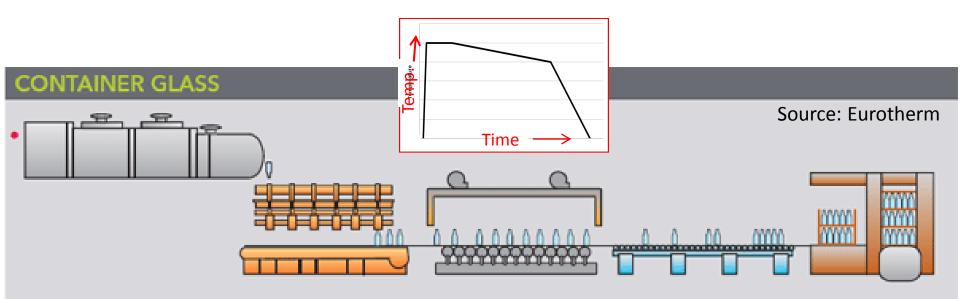
Example: annealing curve for tube glass, 10 cm diam. & 1 cm wall thickness





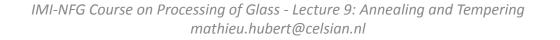
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Example: forming of glass bottles



• At the exit of the annealing lehr, the glass article is (continuously) conveyed to further steps, including coatings, cutting, inspection...







Inspection – residual stresses in the glass

- Perfect glass is **optically isotropic**, its refractive index is the same in all directions
- Mechanical stresses causes deformations in the glass that lead to (local) changes in the refractive index within the material
- A difference in the refractive index within the glass article will lead to birefringence
- This birefringence can be analyzed and quantified with a **polarimeter** (or polariscope), in which the angle of rotation of the polarization direction of linearly polarized light passing through the sample is determined
- Thus, polariscopes can be used to determine the presence of residual stresses within the glass article
- Automated devices based on this principle are used in the glass industry for systematic inspection of the articles produced

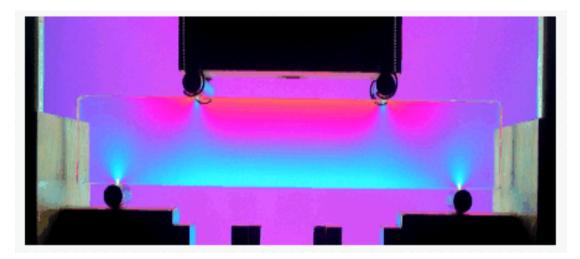




Polariscopes and stresses in glass

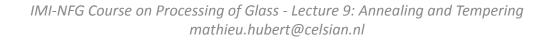






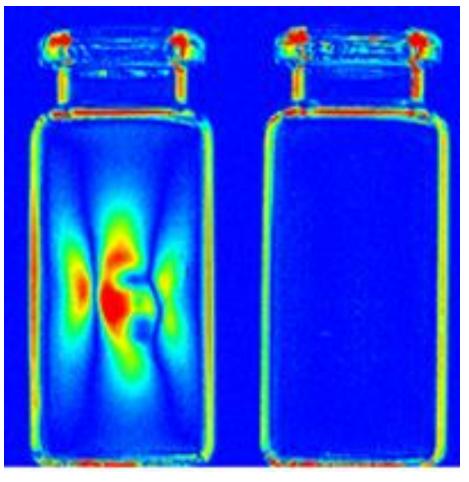
Example of stress distribution in glass (university Erlangen, Germany







Polariscopes and stresses in glass



- Articles with a too high amount of residual stress (e.g. jar on the left of the picture) are rejected (automatic process)
- The rejected product are (often) collected and re-injected in the furnace as raw material (internal cullet)

http://www.vision-systems.com









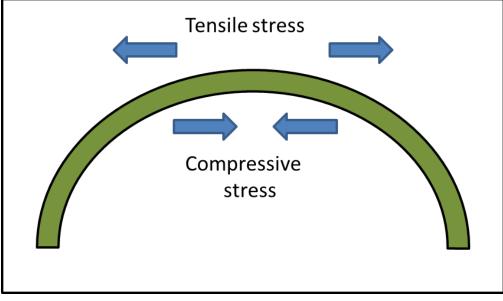


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Introduction – what is tempered glass?

- Glass is stronger in compression than is tension (compressive strength ~10 times higher than tensile strength)
- Glass failure almost invariably originates from flaws at the surface (stress multipliers for local tensile stresses)
- A compressive stress at the surface of the glass can thus increase glass strength

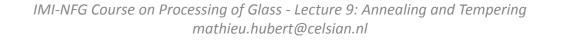




Introduction – what is tempered glass?

- Tempered glass is a glass that has been subjected to an additional heat treatment after annealing in order to increase its mechanical strength
- The tempering process lies on the controlled creation of permanent stresses in the glass
- The surface is under compressive stress while the core is under tensile stress
- Tempered glass can be as much as 4 to 5 times stronger than annealed glass (without tempering)
- When fracturing, tempered glass breaks into small fragments. It is often referred to as "**safety glass**"



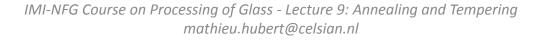




Introduction – what is tempered glass?

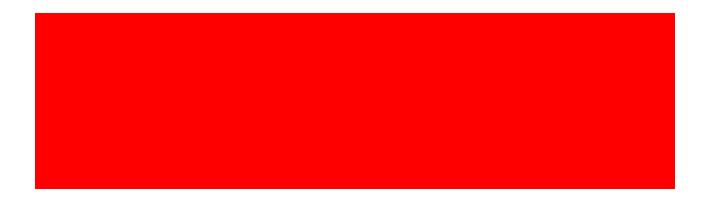
- Tempering of glass is mostly applied to articles with relatively simple geometries, e.g. windows, windshields...
- The tempering process involves reheating of the glass article to a critical temperature (typically above 600-650°C) and subsequent rapid cooling of the surface to create a desired stress profile within the material
- NB: tempering of the glass is performed on an already well annealed glass
- The following slides will illustrate the principle of tempering for a flat glass plate





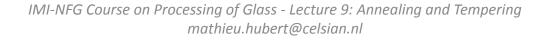
t₀: Temperature T > Tg

Uniform temperature throughout the sample



<u>NB</u>: The temperature T_0 (at t_0) should not be too high to avoid deformation of the glass plate

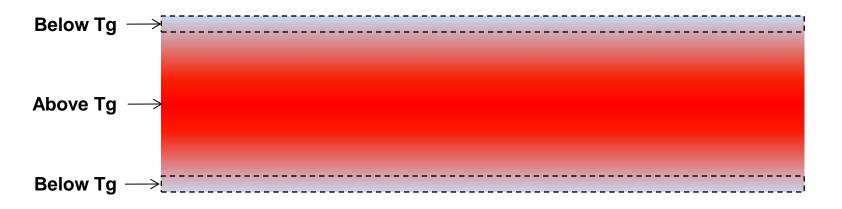






• **t**₁: **surface** of the glass piece **cooled down rapidly to a T < Tg**

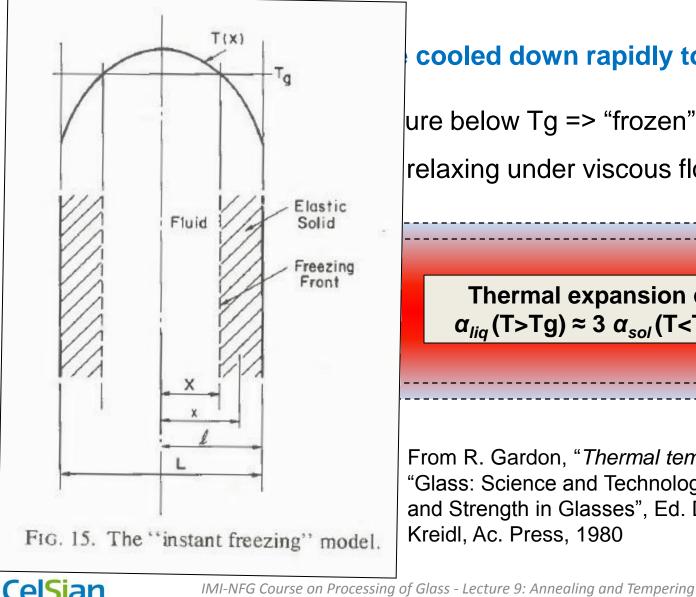
Surface temperature below Tg => "frozen" Core still above Tg, relaxing under viscous flow



At t_1 : Surface tries to shrink while the inner part acts as a counterforce: \Rightarrow Surface under tensile stress \Rightarrow Inner part under compressive stress







cooled down rapidly to a T < Tg

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ure below Tg => "frozen"
relaxing under viscous flow
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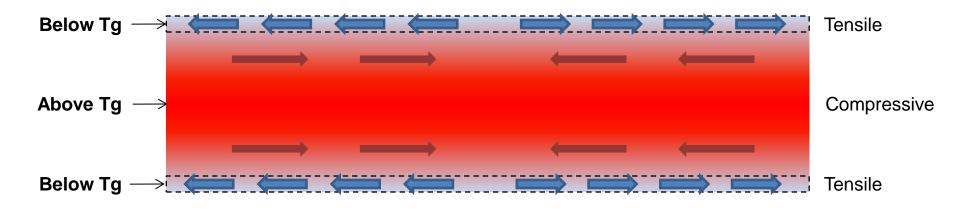
Thermal expansion α α_{lig} (T>Tg) \approx 3 α_{sol} (T<Tg)

From R. Gardon, "Thermal tempering of glass", in "Glass: Science and Technology, Vol.5, Elasticity and Strength in Glasses", Ed. D. Uhlmann and N. Kreidl, Ac. Press, 1980



t₁: surface of the glass piece cooled down rapidly to a T < Tg

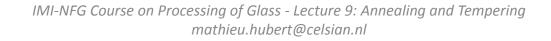
Surface temperature below Tg => "frozen" Core still above Tg, relaxing under viscous flow



At t_1 : Surface tries to shrink while the inner part acts as a counterforce:

- \Rightarrow Surface under tensile stress
- \Rightarrow Inner part under compressive stress

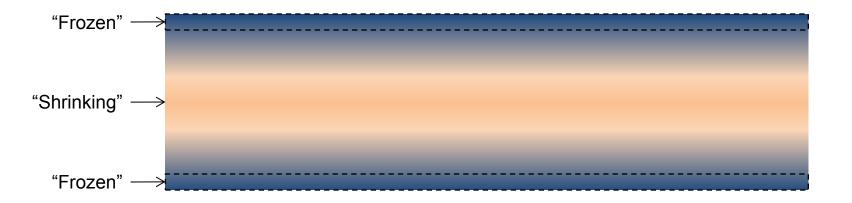






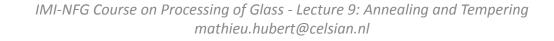
• **t₂**: **further cooling**, inner part cooled down to a temperature T < Tg

Inner part of the glass piece contracting ("shrinking") Surface temperature already "frozen", shrinking less



At t_2 : Inner part tries to shrink while the surface acts as a counterforce: \Rightarrow Surface under compressive stress \Rightarrow Inner part under tensile stress

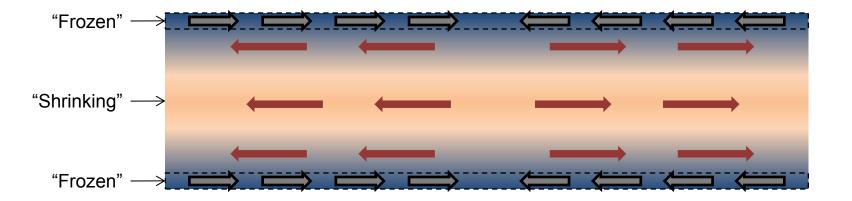






• **t₂**: **further cooling**, inner part cooled down to a temperature T < Tg

Inner part of the glass piece contracting ("shrinking") Surface temperature already "frozen", shrinking less

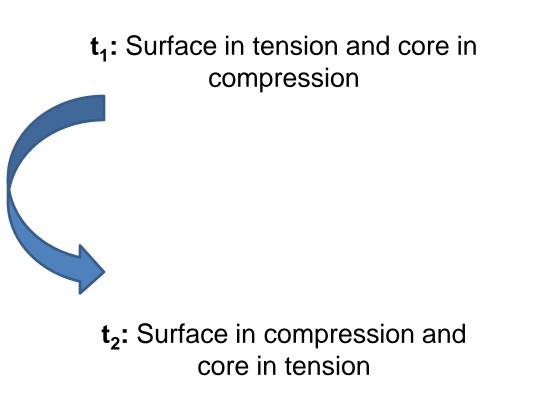


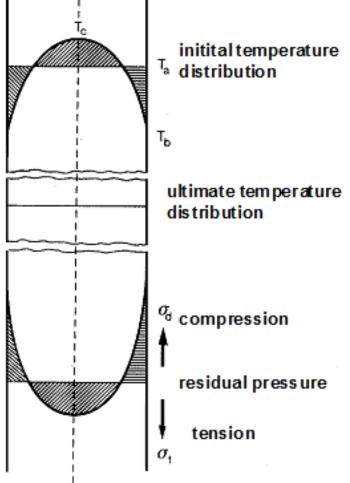
At t_2 : Inner part tries to shrink while the surface acts as a counterforce:

- \Rightarrow Surface under compressive stress
- \Rightarrow Inner part under tensile stress





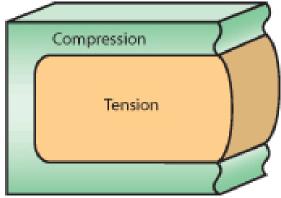






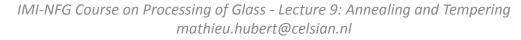
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- After further cooling, the glass article is left with a permanent stress profile with:
 - ✓ Surface in compressive stress
 - ✓ Core in tensile stress



- For a crack to propagate from the surface of the glass article, it must overcome the usable strength of the material + the extra compressive force at the surface
- For this reason, tempered glass is more resistant to failure than a glass which is simply annealed (without compressive layer at the surface)

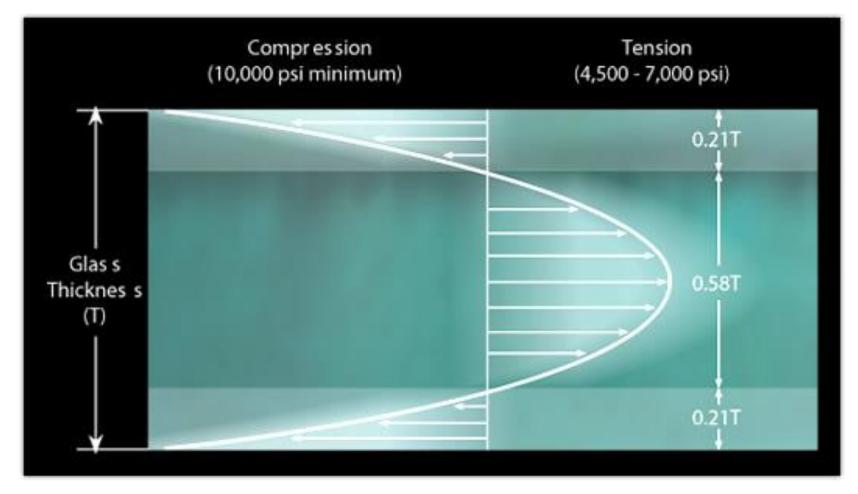








Example stress distribution in solar (flat) glass



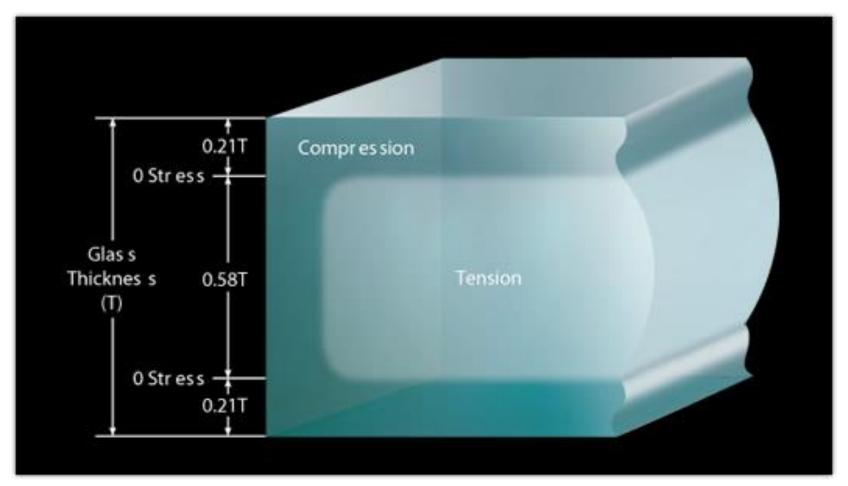
Source: http://www.cardinalst.com/products/solartemp/



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Example stress distribution in solar (flat) glass



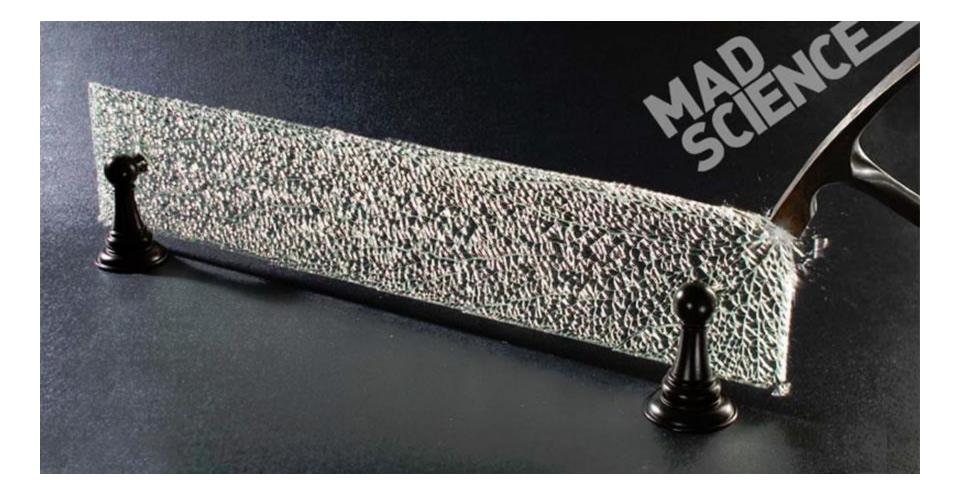
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Fracture pattern of tempered glass



Source: http://www.graysci.com/chapter-seven/shattering-the-strongest-glass/



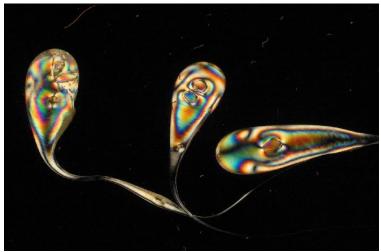
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Taken from: www.bbc.co.uk







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Dropping molten glass into cold water creates a tadpole-like shape called Prince Rupert's Drop.



http://www.youtube.com/watch?v=xe-f4gokRBs#t=29

The head is very strong and can withstand blows from a hammer, but if the tail is damaged at all the whole structure will disintegrate explosively.

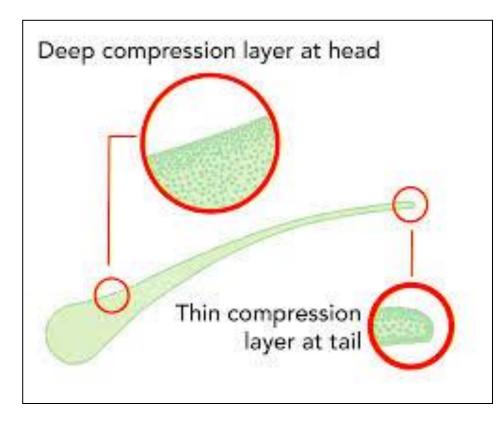
RandomInterestingFacts.com



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• What does it mean?





Pictures © Smarter everyday



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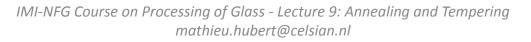
- When damaging the tail => creation of a flaw which propagates to the core, in tensile stress
- All the strain energy stored in the glass (stress-strain relationship) is released, leading to the catastrophic failure of the glass article
- This is similar to what is observed in tempered glass





- More on Prince Rupert's drops? Check out these videos:
 - ✓ Video "Smarter everyday Mystery of Prince Rupert's Drop at 130,000 fps"
 - ✓ Video "Corning- <u>The glass Age, Part 2: Strong, Durable Glass</u> "



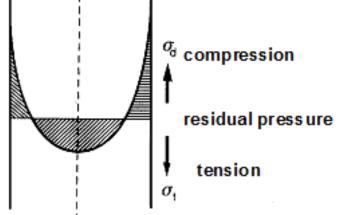




Important parameters for glass tempering

Permanent stress profile generated σ_p

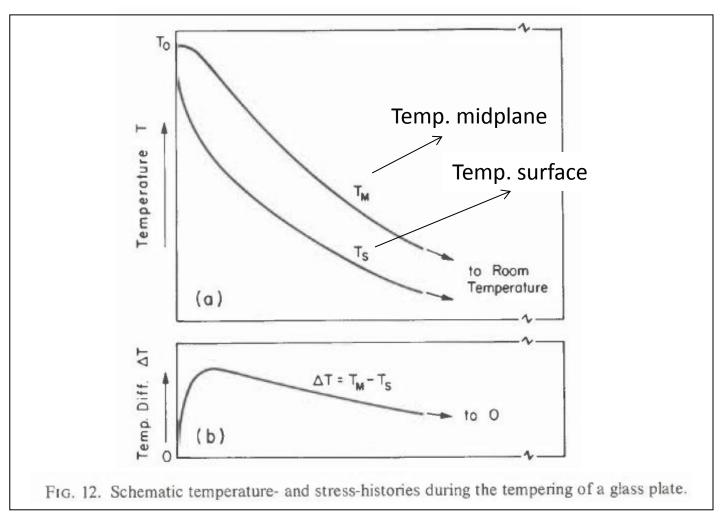
$$\sigma_p = \frac{\alpha.E.\Delta T_{MS}}{1-\mu} = \frac{\alpha.E}{1-\mu} x \left(1 + \frac{2\lambda}{hd}\right)^{-1} x T_E$$



With: α = thermal expansion coefficient [K⁻¹] E = Young's modulus [MPa] Glass μ = Poisson's ratio characteristics λ = thermal conductivity [W/m².K] h = heat transfer coefficient [W/m².K] $\Delta T_{MS} = T_M - T_S = Temp.$ middleplane – Temp. surface [K] **Process** related $T_F =$ "freezing temperature" \approx Tg [K] d = thickness of the glass plate [m] IMI-NFG Course on Processing of Glass - Lecture 9: Annealing and Tempering mathieu.hubert@celsian.nl



Thermal history in a tempered glass plate



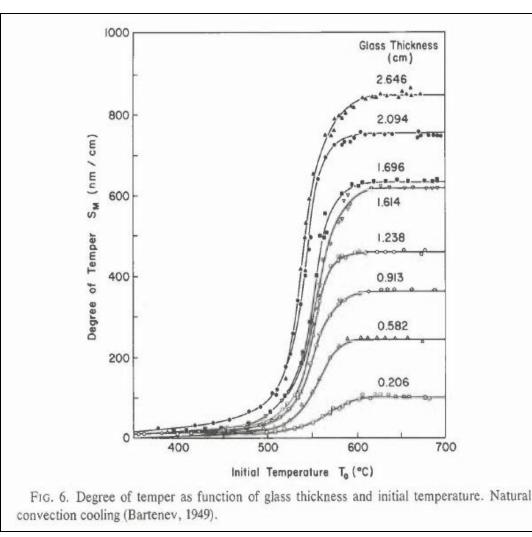
Adapted from R. Gardon, "*Thermal tempering of glass*", in "Glass: Science and Technology, Vol.5, Elasticity and Strength in Glasses", Ed. D. Uhlmann and N. Kreidl, Ac. Press, 1980



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Effect of glass thickness



- With thicker plates, higher degrees of temper achieved (larger stress profiles)
- Above a certain temperature, a plateau is reached
- Below a certain thickness, tempering becomes inefficient

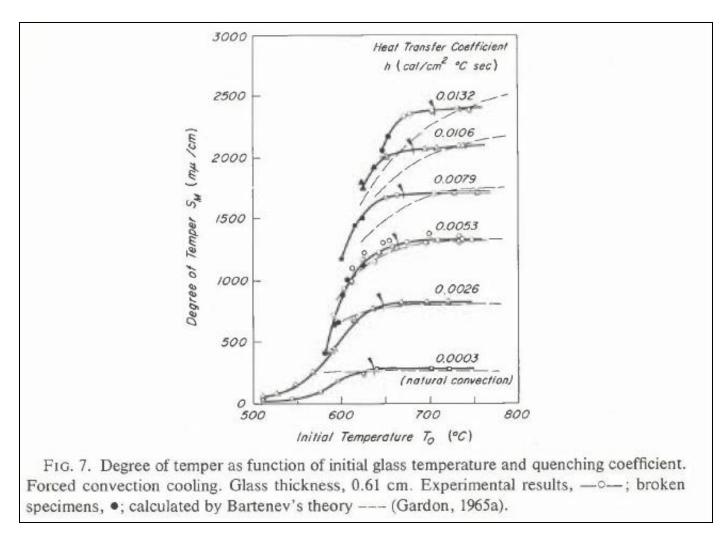
From R. Gardon, "*Thermal tempering of glass*", in "Glass: Science and Technology, Vol.5, Elasticity and Strength in Glasses", Ed. D. Uhlmann and N. Kreidl, Ac. Press, 1980



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Effect of temperature and quenching coefficient



From R. Gardon, "*Thermal tempering of glass*", in "Glass: Science and Technology, Vol.5, Elasticity and Strength in Glasses", Ed. D. Uhlmann and N. Kreidl, Ac. Press, 1980



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Examples for a glass plate of thickness = 8mm

• Initial viscosity of the glass $\eta_i = 10^8$ Pa.s

	H _{max} (W/m².K)	Initial temp. T _i (°C)	σ _{max} midplane (MPa)	σ _{max} surface (MPa)
Soda-lime-silicate α = 9.10 ⁻⁶ K ⁻¹	4500	650	105	235
Borosilicate α = 3.10 ⁻⁶ K ⁻¹	>5000	730	32	70

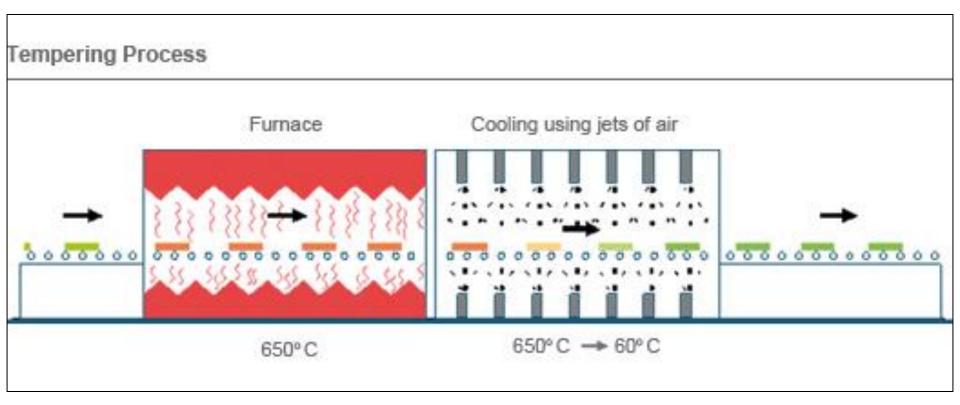
• Initial viscosity of the glass $\eta_i = 10^9$ Pa.s

	H _{max} (W/m².K)	Initial temp. T _i (°C)	σ _{max} midplane (MPa)	σ _{max} surface (MPa)
Soda-lime-silicate α = 9.10 ⁻⁶ K ⁻¹	470	630	65	135
Borosilicate α = 3.10 ⁻⁶ K ⁻¹	1500	680	25	50



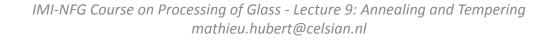


Illustration of the a glass tempering unit for a soda-lime-silica glass

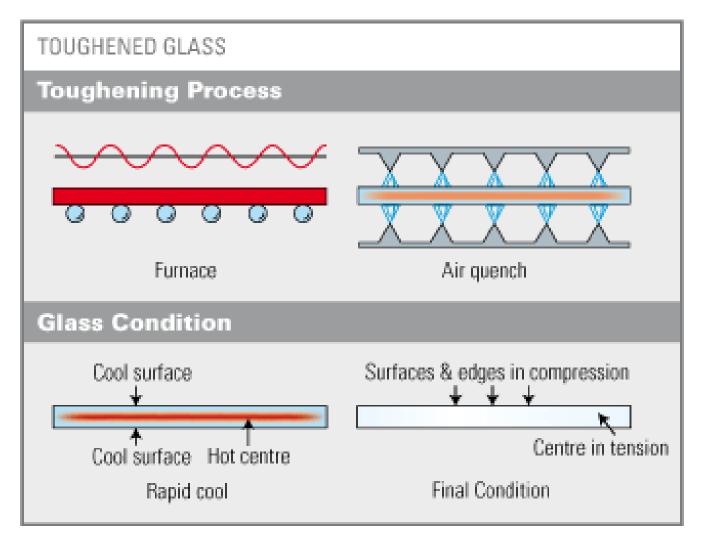


From: http://us.agc.com





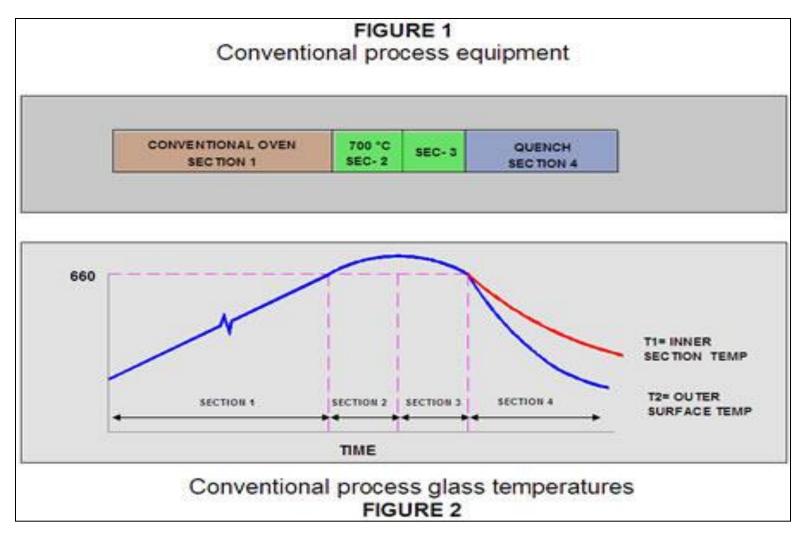




Source: http://www.metroglasstech.co.nz/catalogue/038.aspx



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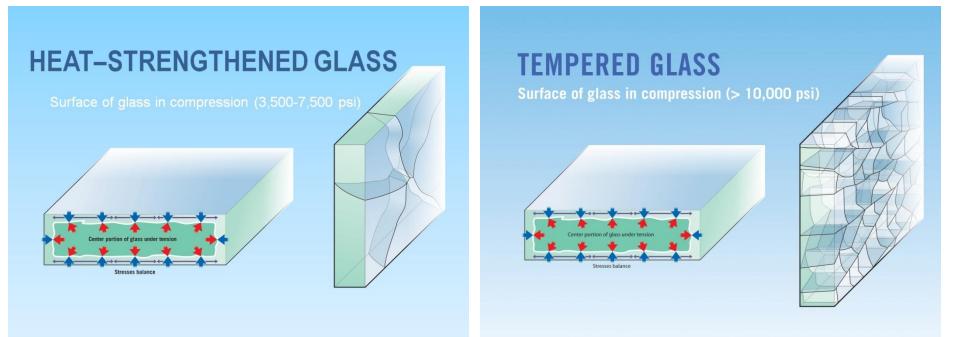
From P. Boaz "Thin glass processing with radio wave assist", www.glassonweb.com/articles/article/561/





Heat-strengthened vs tempered glass?

- Heat-strengthened glass: the cooling process is slower, which means the compression stress is lower
- In the end, heat-strengthened glass is approximately twice as strong as annealed glass but less strong than tempered glass



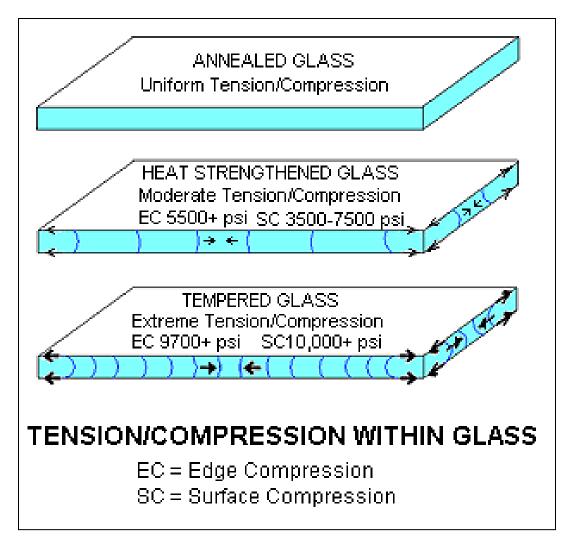


Pictures from: http://educationcenter.ppg.com

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Heat-strengthened vs tempered glass?



Source: www.chicagowindowexpert.com



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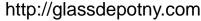


Processing of tempered glass

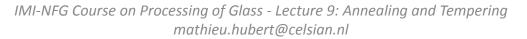
- **Tempered glass cannot be cut nor drilled!** It would lead to release of the strain energy and thus catastrophic failure of the glass
- How are the tempered glass articles made (for instance the windshields?)



http://www.grandsportautobody.com

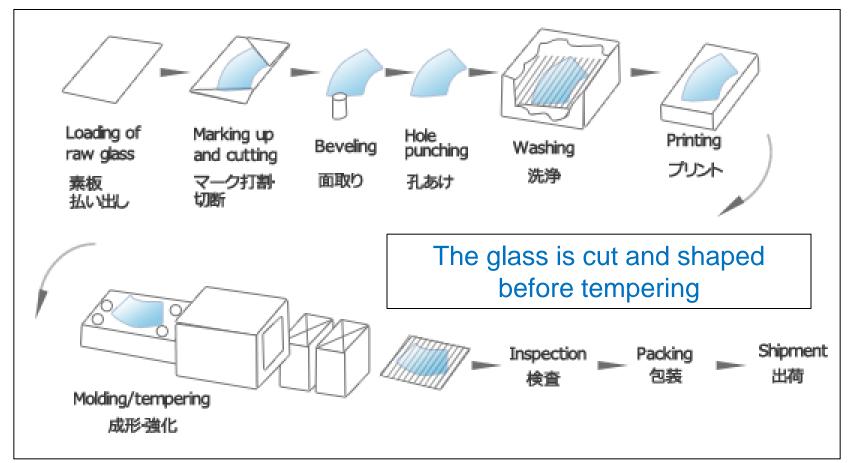








Processing of tempered glass



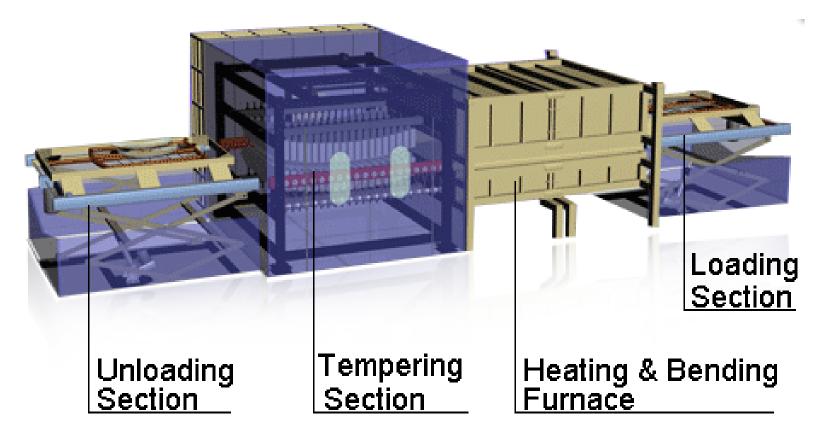
• Example of fabrication of tempered windshields

From: www.agc-automotive.com/english/products/temper.html



64 MIG

Example of a glass tempering furnace



Picture from www.xinology.com/Glass-Processing-Equipments-Supplies-Consumables/glass-tempering/glass-tempering-furnace/feature/bending-tempering.html



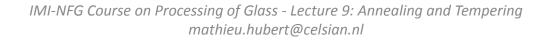
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Other technical considerations

- A certain thickness is necessary for obtaining an efficient strengthening of the glass by tempering
- For thin glass articles with a thickness below 2mm (typically), thermal tempering becomes much less efficient
- Also, tempering requires a uniform cooling from both sides of the glass article (e.g. both surfaces of a glass plate)
- It is thus complicated to temper glass articles with complex or uneven geometries (such as bottles)
- For these types of products, strengthening (when applied) can be performed using ion-exchange technique (chemical strengthening)

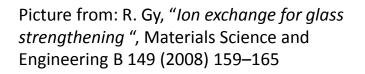






Chemical strengthening

- Chemical strengthening of glass also relies on the formation of a compressive stress on the surface, with the core in tensile stress
- The way to achieve this stress profile is however very different (ionexchange instead of thermal treatment)
- Chemical strengthening of glass will be presented by A. Varshneya in this series of IMI-NFG lectures



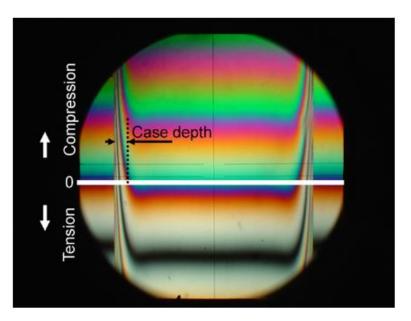


Fig. 3. Stress pattern in a chemically tempered glass: side view in a polariscope equipped with a Babinet compensator.

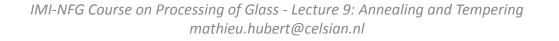


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Conclusions – 1/2 - Annealing

- Annealing of the glass articles after the forming process is crucial for relaxing the stresses due to inhomogeneous, rapid cooling
- The annealing consists in reheating the glass above the annealing temperature and perform a **controlled**, **slow cooling** between the annealing point and the strain point
- The **cooling rate between T_{anneal} and T_{strain}** is crucial and will depend on the type of glass (composition) and the type of article produced (shape, thickness...)
- At industrial scale, annealing is a continuous process, and is performed in annealing lehrs
- The **temperature profile** in the lehr should be optimized to obtain a well-annealed product in the shortest possible time



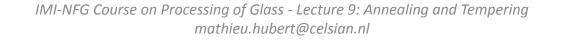




Conclusions – 2/2 - Tempering

- **Tempering** of glass is a thermal treatment performed on annealed glasses to create **controlled** stresses in the glass
- The glass is reheated at a critical temperature and then rapidly cooled, leaving the surface in compressive stress and the core in tensile stress
- Tempered glasses can be **5 times stronger** than annealed glass
- If broken, tempered glass will shatter in small fragments (securit glass)
- Tempered glass cannot be cut or drilled, and the glass article must be shaped before the tempering process
- Tempering is limited to relatively thick products (> 2mm) and relatively simple geometries (windows, windshields...)



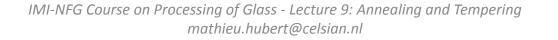




Home assignment

- A multiple choice questionnaire (MCQ) including questions on industrial glass annealing and tempering processes is provided with this lecture
- The MCQ will be available online on IMI's website



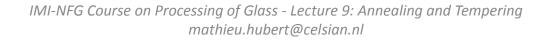




References and further reading

- "The glass tempering handbook" by Jonathan Barr, available online (free) at <u>https://dl.orangedox.com/IOM4ukrFcunESCW2Yh/TheGlassTemperingHa</u> <u>ndbook.pdf</u>
- Book "Strength of Inorganic Glass", Ed. C. Kurkjian (Plenum, 1985)
- R. Gardon, "Thermal tempering of glass", in "Glass: Science and Technology, Vol.5, Elasticity and Strength in Glasses", Ed. D. Uhlmann and N. Kreidl (Academic Press, 1980)
- CelSian's glass course e-learning trailer: <u>https://www.youtube.com/watch?v=pID0PYsBlbQ&feature=youtu.be</u>







Thank you for your attention



Questions ?

Visit us in Eindhoven

Contact me via email:

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