



Stress relaxation in tempered glass caused by heat soak testing



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ARTICLE INFO

Article history:

Received 24 June 2014

Revised 13 January 2016

Accepted 11 April 2016

Keywords:

Tempered glass

Heat soak test

Stress relaxation

Tool-Narayanaswamy-Model

Fracture pattern

Photoelastic measurements

ABSTRACT

Heat soak testing of tempered glass is a thermal process required after the tempering process itself to bring glasses of commercial soda-lime-silica-glass to failure that are contaminated with nickel sulphide inclusions, diameter 50 μm to 500 μm typically. Thus, the tests avoid a so-called “spontaneous” breakage of the glass in building elements at ambient temperatures months or years later. According to industry standards, the duration of the tests typically differs between 1 h and 4 h at temperatures of 290 ± 10 °C. Although this temperature is well below the transformation temperature of commercial soda-lime-silica glass, it causes stress relaxation in tempered glass and the fracture pattern of the glass changes accordingly, especially thin glasses are affected. Based on the Tool-Narayanaswamy-Model, this paper comprises the theoretical background of the stress-relaxation-process and the results of a parameter study for its most influential technical parameters. Results are compared to photoelastic measurements of temper stresses and fracture patterns of tempered glass before and after a heat treatment similar to heat soak testing.

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1. Introduction

Heat soak testing (HST) of tempered glass today is a common thermal process for tempered glass after the tempering process itself. According to industry standards, the duration of the tests typically differs between 1 h and 4 h at temperatures of 290 ± 10 °C [1]. The test is used to reduce the failure probability of tempered glasses affected by nickel sulphide (NiS-) inclusions. The reason for the glass failure is a phase change of microscopically small NiS-inclusions, diameter 50–500 μm typically, that induce high local stress peaks and related stress intensities at cracks tips around the inclusions that finally cause fracture of the glass pane if in or near the tension zone of the temper stress [2–6]. The phase change strongly depends on the temperature and so a HST-temperature is chosen to bring glasses to a failure as quickly as technically possible and at the same time to avoid a new phase change. Typically the phase change between α -phase and β -phase happens at above 379 °C, but also lower values (280 °C) are possible depending on the stoichiometric composition of the NiS [7]. A significant reduction of the failure probability from NiS-inclusions for tempered glass can be reached by the HST [5,8]. Recent research shows that

also heat strengthened glass is affected, although the fracture pattern is different compared to tempered glass [8]. As a reliable optical detection of the inclusions during the glass production is not yet possible due to the small size of the inclusions and their rather small probability of occurrence compared to other imperfections (typically one inclusion on 8–10 tons of glass), the HST today is an important part of the production process. Standards [1] and stricter building regulations from different countries (e.g. in Germany) specify the required HST time and temperature. Fig. 1 shows a typical time–temperature-diagram of the glass surfaces during HST in a commercial heat soak oven. The glass is typically heated to about 290 °C during 2.5 h, the holding phase duration last about 2–4 h (depending on regulations) and the cooling of the glass to ambient temperature takes about 1.5 h.

Although the temperatures that initiate the desired phase change is well below the transition temperature of commercial soda-lime-silica glass (above 525 °C), the process causes stress relaxation in the glass and the fracture pattern of the glass changes to a more coarse pattern due to a reduction of the tensile stress in the glass core. This is an undesired effect as the fracture pattern is often related to safety issues of tempered glass. As the bending strength of the glass is governed by the surface compression stress, the related relaxation of the surface compression stress also affects the apparent bending strength. Therefore, it is important to study the most influential parameters on the stress relaxation from

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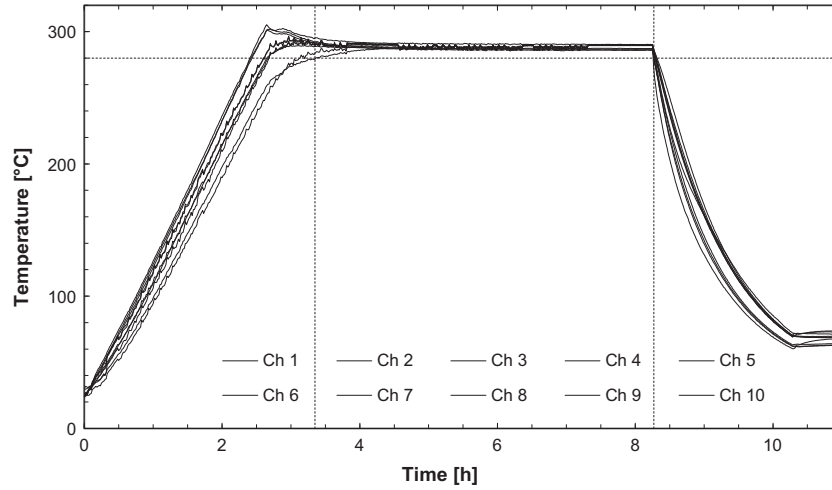


Fig. 1. Typical time–temperature–diagram from thermocouples on glass surfaces during commercial heat soak testing.

HST for different temper scenarios and glass thicknesses to allow a prediction based on simulations and experimental data.

2. Stress-relaxation in tempered glass – theoretical background

To study the stress relaxation in tempered glass the whole stress and strain history of tempering process should be known. In the tempering process at high temperature above the transition temperature, all the stresses can be assumed to relax and strain and stress changes after that should be taken into account on modelling. The strains and stresses can be divided into volumetric and deviatoric parts and the hereditary integral should be used due to the time-dependency of bulk and shear moduli [9]:

$$\sigma_{ij}(t) = \delta_{ij} \int_0^t K(t-t') \frac{\partial(\varepsilon_{ii} - 3\varepsilon^{th})}{\partial t'} dt' + 2 \int_0^t G(t-t') \frac{\partial(e_{ij})}{\partial t'} dt' \quad (1)$$

where σ_{ij} is the stress tensor, K is the bulk modulus, G is the shear modulus, ε_{ii} is the sum of normal strain components ($\varepsilon_{ii} = \varepsilon_{xx} + \varepsilon_{yy} + \varepsilon_{zz}$), ε^{th} is the thermal strain and e_{ij} is the deviatoric strain ($e_{ij} = \varepsilon_{ij} - \delta_{ij} \frac{1}{3} \varepsilon_{ii}$).

The bulk and shear relaxation moduli for the generalised Maxwell model can be presented with Prony's series by dividing the relaxation curve by weighting factors w and relaxation times τ :

$$K(t) = K_\infty + (K_0 - K_\infty) \sum_{p=1}^P w_{2p} \exp\left(-\frac{t}{\tau_{2p}}\right) \quad (2)$$

$$G(t) = G_0 \sum_{q=1}^Q w_{1q} \exp\left(-\frac{t}{\tau_{1q}}\right) \quad (3)$$

where subscript 0 is the initial value and ∞ is the value at infinite time. In the weighting factors and relaxation times the subscript 1 is for the shear relaxation and 2 for the bulk relaxation.

The relaxation times are temperature dependent. Because glass is assumed to be a thermorheologically simple material, the relaxation time at different temperatures can be calculated using a shift function ϕ . For the shift function the Arrhenius equation can be used where the structural relaxation of glass has been taken into account [10]:

$$\begin{aligned} \phi(t) &= \frac{\tau_{ref}}{\tau} = \exp\left[\frac{H_g}{R} \left(\frac{1}{T_{ref}} - \frac{1}{T(t)}\right) + \frac{H_s}{R} \left(\frac{1}{T_{ref}} - \frac{1}{T_f(t)}\right)\right] \\ &= \exp\left[\frac{H}{R} \left(\frac{1}{T_{ref}} - \frac{x}{T(t)} - \frac{1-x}{T_f(t)}\right)\right] \end{aligned} \quad (4)$$

In Eq. (4) the activation energy $H = H_g + H_s$ and $x = H_g/H$, where H_g is related to the stress relaxation and H_s to the structure. Furthermore R is the universal gas constant, T_{ref} is the reference temperature (the temperature where the reference relaxation times are chosen), T is real temperature and T_f is the fictive temperature.

The relaxation speed of stresses depends on the time used since the change of strain. Due to the change of temperature during the tempering and heat soak test the original time in Eqs. (1)–(3), should be change to the reduced time ξ , which is the time integral of a shift function.

$$\xi(t) = \int_0^t \phi(t') dt' \quad (5)$$

By using the reduced time the stress–strain relation (Eq. (1)) can be presented as a function of reduced time instead of original time.

$$\begin{aligned} \sigma_{ij}(t) &= \delta_{ij} \int_0^t K(\xi(t) - \xi(t')) \frac{\partial(\varepsilon_{ii} - 3\varepsilon^{th})}{\partial t'} dt' + 2 \int_0^t G(\xi(t) \\ &\quad - \xi(t')) \frac{\partial e_{ij}}{\partial t'} dt' \end{aligned} \quad (6)$$

The fictive temperature in Eq. (4) presents the behaviour of structural relaxation. The structural relaxation refers to the rearrangement of microstructure components of material, which is changing with time [11]. At a temperature below the transition temperature glass is in an unstable state. A stabilized glass is one that has been held at given temperature until its properties have stopped changing with time [12]. The response function for material properties M_p can be presented with temperature and fictive temperature:

$$M_p(t) = \frac{p(t) - p_2(\infty)}{p_2(0) - p_2(\infty)} = \frac{T_f(t) - T_2}{T_1 - T_2} \quad (7)$$

In the response function M_p above p is a property and subscript 1 is the state before temperature change and 2 is the state after the temperature change. Using Eq. (7) the fictive temperature can be calculated. The whole time history has to be taken into account, because the temperature change is time-dependent.

$$T_f(t) = T(t) - \int_0^t M_p(t-t') \frac{\partial T}{\partial t'} dt' \quad (8)$$

The response function of the material property M_p can be expressed by analogy with the bulk or shear relaxation function:

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