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Experimental investigations on the fracture strength of soda-lime silica glass at elevated temperatures

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The surface strength of glass plays a significant role when dimensioning glass components in structural facades. Commonly soda-lime silica glass is used as material in architectural glass design. Different environmental conditions for example the load rate, relative humidity, surface flaws etc., but also the ambient temperature can have a strong influence on the resulting surface strength. The relationship between temperature, especially elevated temperatures, and surface strength of soda-lime silica glass has so far been rarely investigated. Therefore, this paper methodically presents an experimental test setup for the determination of bending strength at elevated temperatures as well as first results. To determine the bending tensile strength of soda-lime silica glass as float glass, the double ring bending test was conducted on specimens with small test surfaces according to EN 1288-5. The double ring bending tests were performed using a tensile fatigue testing machine with furnace on circular glass plates of soda-lime silica glass in a temperature range from room temperature to 550 °C. Before testing, the specimens were first pre-damaged (inducing crack with diamond), stored and heat treated as the final pre-treatment step. To determine the load rate for the experiments at elevated temperatures, the experiment was simulated in advance for the various temperatures using the finite element method with consideration of structural relaxation. To account for the structural relaxation, the Narayanaswamy model was utilized.

Keywords: soda-lime silica glass, pre-damaging, heat treatment, glass-transition temperature, double ring bending test, fracture strength

1 Introduction and previous research

In the production of flat glass and the further treatment of these glasses by the thermal toughening processes, glass breakage may occur due to cooling [10]. Narayanaswamy [20] studied, within the framework of thermal toughening process, the transient stresses occurring in the glass during the toughening technique. He could show that during cooling tensile stresses occur on the glass surfaces. Pourmoghaddam and Schneider [4] showed in their studies via finite element analyses (FEA) that on the glass surface and at unfavorable cutout geometries high tensile stresses develop which may lead to fracture during cooling in the thermal toughening process. Knowledge of the glass strength at elevated temperatures thus plays a decisive role in controlling the cooling process. In the past, several studies [22, 19, 18, 17, 15, 8, 5] were performed with different approaches

Engineered Transparency 2021. Glass in Architecture and Structural Engineering, First Edition.
Edited by Christian Louter, Jens Schneider, Silke Tasche, Bernhard Weller.

for pre-treatment of the specimens, test setups and also analyses of the results e.g. fracture strength, stress intensity factors and fracture surface energy. Investigations on the bending strength of reproducible pre-damaged soda-lime silica glass at elevated temperatures with the background of the construction industry have been little studied so far. For the investigations carried out within this research, in contrast to the previous investigations, an own method was developed to determine the bending strength of pre-damaged soda-lime silica glass as float glass.

2 Theory of the axisymmetric plate

The stresses in the glass specimen which arise in the double ring bending test can be calculated with the theory of the axisymmetric plate assuming a linear elastic material law. The geometrical non-linearity and effects from friction at the load and support ring may be neglected due to the small deformations ($w / 2r_2 \ll 1$). EN 1288-1 [11] gives the analytical equation (2.1) to calculate the tensile stresses arising in the double ring bending test for the given geometry in Figures 2-1a and 2-1b.

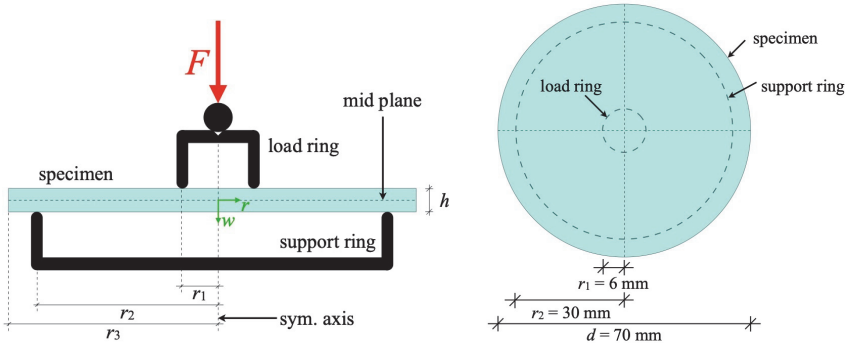

Figure 2-1 a) Sketch of the test setup: double ring bending test according to EN 1288-5 [12] and b) Top view of the specimen.

In EN 1288-1 [11] the influence of geometry (r_1, r_2, r_3) and *Poisson*'s ratio υ is summarized with the factor K_1. For the test setup R30 according to EN 1288-5 [12] with consideration of *Poisson*'s ratio $\upsilon = 0.23$, K_1 results to 1.09 and equation (2.1) may then be simplified to equation (2.2). The specimens used in this study had a nominal diameter of 70 mm and deviate from the normative specification ($d = 66$ mm). If 70 mm diameter and a *Poisson*'s ratio of 0.22 (see below for explanation) are used to determine K_1, a value of 1.069 results for K_1, which is 2 % lower than 1.09. A disadvantage in the notation of equation (2.2) (dependencies of K_1 are not displayed) is, that with regard to the specimens tested at different temperatures, the temperature dependency of the *Poisson*'s ratio υ and also the thermal strains of the test setup (r_1, r_2, r_3) may be forgotten. Manns and Brückner [18] and [16] have investigated the influence of the *Poisson*'s ratio on the fracture stress calculated with equation (2.1) by choosing theoretical curves of the *Poisson*'s ratio as a

function of temperature. They concluded that neglecting the temperature dependency of the *Poisson*'s ratio results in errors of maximum 11 %. By taking the *Poisson*'s ratio at room temperature (υ = 0.22) into account, lower fracture stresses are determined, which finally result in conservative values of the fracture stress. Shinkai et al. [19] and Duffrène et al. [14] investigated the temperature dependency of the *Poisson*'s ratio of soda-lime silica glass experimentally. The results of Shinkai et al. [19] allow for a linear relationship between temperature and *Poisson*'s ratio, where a value of υ = 0.29 was measured at approximately 500 °C. The results of Duffrène et al. [14] also show a linear relationship between temperature and *Poisson*'s ratio, but the gradient from room temperature up to 600 °C is very small, starting with υ = 0.218 at 0 °C resulting in υ = 0.223 at around 600 °C. The results of Shinkai et al. [19] and Duffrène et al. [14] differ and may be caused by different measurement techniques. Duffrène et al. [14] used Brillouin scattering, an optical method, whereas Shinkai et al. [19] used resonance techniques with the help on an acoustic spectrometer. From the investigations of Shinkai et al. [19], Duffrène et al. [14] and Manns and Brückner [18], it is assumed for the own studies presented here that the *Poisson*'s ratio may be set to a constant value of υ = 0.22 for the temperature ranges investigated. The thermal expansion of the sample and the experimental setup was also neglected in accordance to Manns and Brückner [18]. It can be shown that the influence of the thermal strains (specimen, load ring and support ring) at a temperature difference of 527 K between room temperature and 550 °C only results in a difference of 1 % in the calculated fracture stress by equation (2.1), assuming υ = 0.22.

$$\sigma_{\mathrm{rad}} = \sigma_{\mathrm{tan}} = \sigma(r_1, r_2, r_3, \upsilon) = \frac{3(1+\upsilon)}{2\pi}\left[\ln\left(\frac{r_2}{r_1}\right) + \frac{1-\upsilon}{1+\upsilon}\frac{r_2^2 - r_1^2}{2r_3^2}\right]\frac{F}{t^2} \tag{2.1}$$

$$\sigma = K_1 \frac{F}{t^2} = 1.09\,\frac{F}{t^2} \tag{2.2}$$

3 Numerical model to determine the load rate for testing at elevated temperatures

The load rate to obtain a stress rate of 2 MPa/s (EN 1288-5 [12]) on the bending tensile side of the specimen at elevated temperatures was determined using a finite element model (see Figure 3-1a). To determine the temperature at which the FE model is required to calculate the load rate for the experiment, a numerical study was carried out. For this purpose, the double ring bending test under the same loading was numerically simulated for different temperatures. In Figure 3-1b the applied force and the resulting displacement at the bottom center of the specimen are presented for different temperatures. As it can be seen in Figure 3-1b, first non-linear effects appear at 350 °C and above. For this reason, the load rates for the experiments at temperatures of 350 °C and higher were determined using the numerical model. If the relationship between force and stress is known, equation (3.1) can be used to calculate the load rates required for the tests (see Table 4-1). For the experiments at lower temperatures the load rates were determined using equation (2.1) and equation (3.1). The numerical model is based on rotational symmetry in such a way

that a quarter of the specimen is represented. Figure 3-1a exemplary shows the total deformation of the model (specimen with 3.85 mm thickness) under 800 N load at 550 °C.

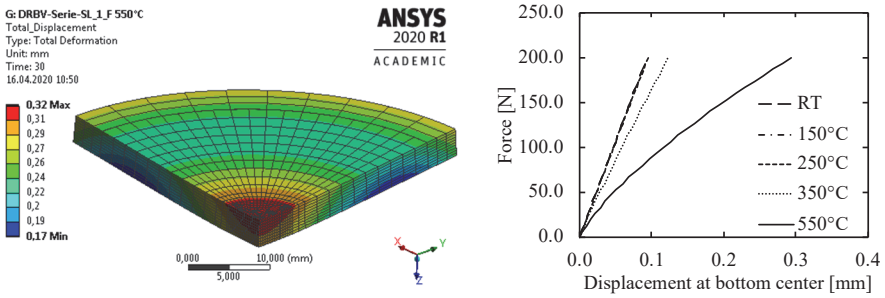

Figure 3-1 a) FE-Model in deformed state under a loading of 800 N at 550 °C and b) Results of the numerical study for different temperatures.

$$\frac{\Delta F}{\Delta t} = 2\ \mathrm{MPa/s}\frac{\Delta F}{\Delta \sigma} \tag{3.1}$$

In the numerical model, the area within the load ring was meshed more dense than the outer areas of the material sample. An extensive mesh study was omitted. The load and support ring were only represented numerically by boundary conditions. A detailed modelling including the components load ring and support ring, which enable the friction at these to be considered, was disregarded. For the numerical simulations the *Narayanaswamy* material model [21] was applied to take into account the effects of structural relaxation of soda-lime silica glass. To do so weights and relaxation times for viscous and structural relaxation were taken from Carré and Daudeville [13]. A detailed description of the material law is given in Pour-Moghaddam [2] and Schneider et al. [7].

4 Experimental investigations

4.1 Pre-damaging and storage of the samples

In the first step of the experimental investigations, the specimens (4 mm nominal thickness) were pre-damaged with the aid of the Universal Surface Tester (UST, see Figure 4-1a). For the investigations presented here, a conically milled diamond with an angle of 120° and a 5 µm radius of curvature at the tip of this cone was used as an indenter to induce a scratch on the tin side of the glass surface. This type of pre-damage was chosen to take into account surface defects in the glass, which can occur during flat glass production itself, transport and handling. With the presented settings in the list below the artificial flaw was induced in the glass surface [9]. This type of scratch causes, when performing double ring bending tests, fracture stresses which correspond to the 5 % quantile value of the fracture stress of float glass defined in EN 572-1 [6] ($f_{g,kk}$ = 45 MPa).

- Indenter: Diamond with an angle of 120° and 5 µm radius of curvature at the tip
- Force: 500 mN
- Scratch length: 2 mm
- Velocity of the indenter: 1 mm/s

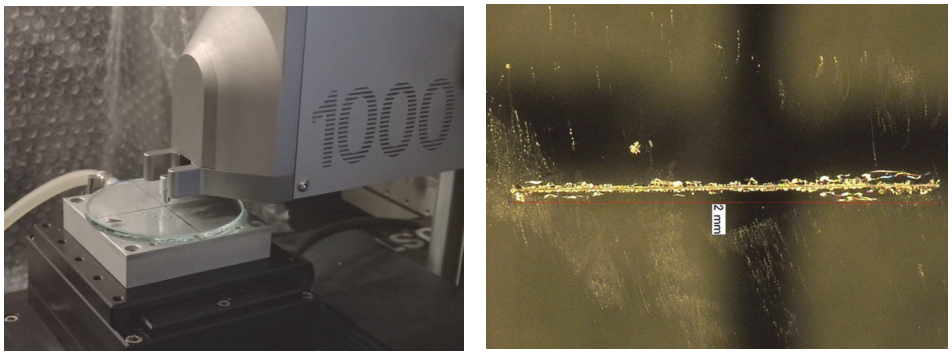

Figure 4-1 a) Universal Surface Tester with mounted indenter and specimen and b) Resulting scratch on the glass surface.

During crack initiation using UST, the specimen is fixed with the help of vacuum on its air side (furnace atmosphere side). The environmental conditions during pre-damaging were around 23 °C and 50 % RH. The resulting surface defect on the tin side can be seen exemplary in Figure 4-1b. The fracture origin of specimens scratched with the presented technique is typically located at this artificial flaw (assuming that the scratch lies within the load ring) when performing double ring bending tests. After pre-damaging the specimens were stored at least for one week in a standard climate (23 °C, 50 % RH) [9].

4.2 Heat treatment

In the last pre-treatment step, the specimens were heat treated in a radiation furnace with the setup shown in Figure 4-2a. The heat treatment is intended to harmonize the material samples in order to eliminate any residual stresses in the glass that may occur as a result of the induced scratch [1, 19, 22].

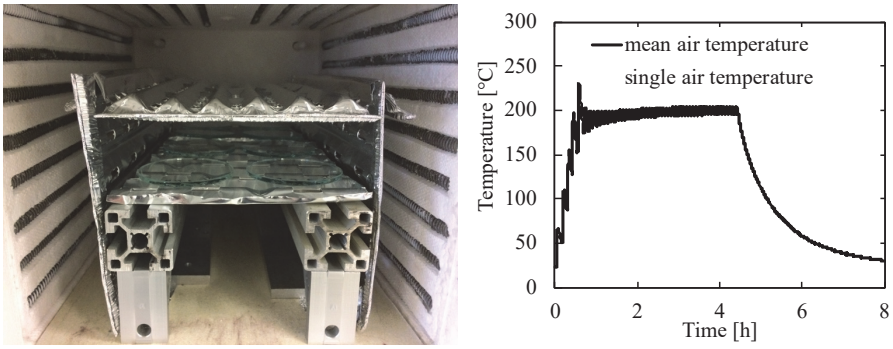

Figure 4-2 a) Arrangement of the specimens and aluminum radiation shield within the furnace chamber and b) Measured air temperature within the aluminum radiation shielding by four type K thermocouples.

The target temperature of the heat treatment was chosen according to Wiederhorn [22] at 300 °C. The dwell time of the specimens within the furnace was chosen to 4 h, so that the maximum temperature can be reached within the furnace due to thermal inertia and disturbance of free convection by the radiation shielding. In order to protect the specimens from overheating, aluminum sheets were used to shield the heat radiation. An independent air temperature measurement (see Figure 4-2b) within this enclosure showed, however, that only a temperature of about 200 °C was reached. The heat treatment therefore took place at around 200 °C. Unfortunately, the humidity inside the furnace chamber could not be measured during the heat treatment. It is assumed that at the initial situation the ambient conditions in the laboratory were around 23 °C and 50 % RH.

4.3 Test setup for double ring bending tests at elevated temperatures

The double ring bending tests at elevated temperatures have been performed on a Zwick/Roell Z100 electromechanical testing machine according to EN 1288-5 [12]. The temperature was measured by three calibrated type S thermocouples, which have been mounted to the grippings and the load ring. Figure 4-3a shows an overview of the test setup. The specimens were heated by a PID-controlled three-zone convection furnace which consists of two halves that are closed during testing.

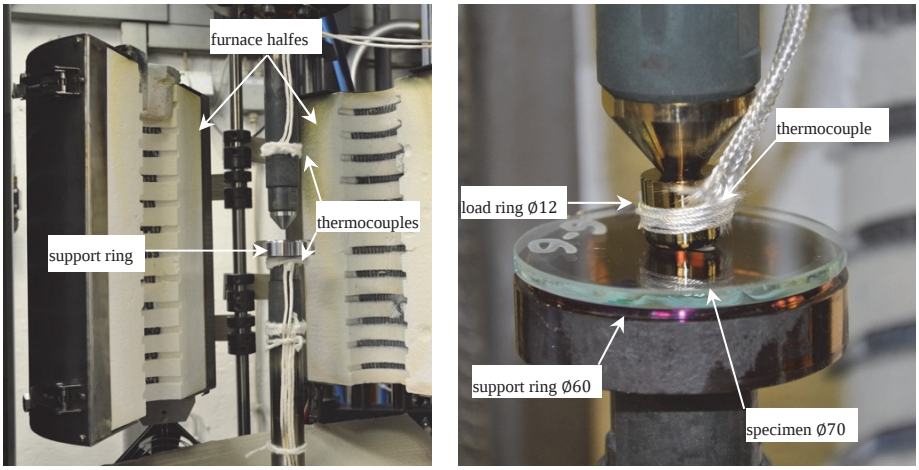

Figure 4-3 a) Overview of furnace and specimen gripping (load ring and specimen not mounted) and b) Specimen and third thermocouple mounted between load ring and support ring.

Depending on the test temperature, it took between 60 and 90 minutes to heat up the whole test setup. In order to ensure a homogenous temperature distribution in specimen and gripping (thermal steady state testing condition), the temperature was held for 30 minutes prior to starting the test. Force was measured by a load cell mounted in the load train on top of the specimen. The load cell was calibrated regularly according to EN ISO 7500-1 [3]. It is assumed that the humidity in the testing lab was around 30 to 40 % RH at room temperature. As shown in Figure 4-3b, the specimens were mounted between a support ring of 60 mm diameter and a load ring of 12 mm diameter. Contrary to EN 1288-5 [12], there was no rubber sheet between the support ring and the specimen, as rubber would not withstand the elevated temperatures during the experimental test campaign. Due to the fact that the induced scratch on the glass surface represents a relatively strong surface flaw, it was assumed that soft interlayers between the specimen and the load or support ring are not required. The specimens (4 mm nominal thickness) were pre-damaged on the tin side and stored as described in Section 4.1. They were then heat treated for 4 h at 200 °C as described in Section 4.2. The tin side of the specimens was placed on the support ring so that the tin side was exposed to bending tensile stress.

Before the oven halves were joined together and so prior to heating up the test setup, the specimen and the load ring incl. the third type S thermocouple were mounted to the grippings (see Figure 4-3b). Afterwards the force of the testing machine was set to a constant value of 15 N. This leads to contact between the load ring, specimen and support ring. This procedure is necessary to avoid tilting or even skewing of the load ring. It also ensures a centered setup. It is expected that the force of 15 N will not cause any damage to the specimen, as any crack growth and healing processes were considered to be completed by the previous storage and heat treatment. The force of 15 N was maintained during the entire heating process until the test was started. The tests have been performed load rate

controlled with the different load rates presented in Table 4-1 for each chosen temperature during testing to provide a stress rate of 2 MPa/s on the bending tension side (tin side). The load rate for testing at elevated temperatures was determined via the FE model presented in Section 3. The specimens were loaded until fracture.

Table 4-1 Load rates of the tested series (stress rate 2 MPa/s).

Series	Temperature during testing	Load rate
SL_1_A	23 °C	27.7 N/s
SL_1_E	500 °C	28.3 N/s
SL_1_F	550 °C	28.8 N/s

4.4 Test results

At the current state of this research, only three test temperatures are presented here, since the test setup and handling are comparatively complex. Figure 4-4 shows the fracture stresses calculated by equation (2.1) versus temperature during testing. In Figure 4-4 it can be seen that the mean values of fracture stresses increase slightly with increasing test temperature. When comparing the average values of the fracture stress between room temperature and 550 °C (see Table 4-2), an increase in strength of around 14 % can be observed. However, it should be noted that the results obtained so far must be statistically verified, as the sample size of the individual test series is very small. Based on the results presented here, only a rough estimate can be made that the bending strength of glass increases at higher temperatures. The results obtained so far are in good agreement with those from Manns and Brückner [18], so that it can be assumed that the presented method in this study provides reproducible results. On the basis of the increasing fracture stress with increasing temperature, it can be concluded that the scratch applied to the surface of the specimen heals slightly, resulting in an increase of fracture stresses. Manns and Brückner [18] also attributed the increase in bending strength at elevated temperatures to possible crack healing in the glass.

Table 4-2 Results of the tested series (stress rate 2 MPa/s).

Series	Temperature during testing	Mean fracture stress
SL_1_A (16 specimen)	23 °C	55.2 MPa
SL_1_E (7 specimen)	500 °C	61.1 MPa
SL_1_F (9 specimen)	550 °C	63.0 MPa

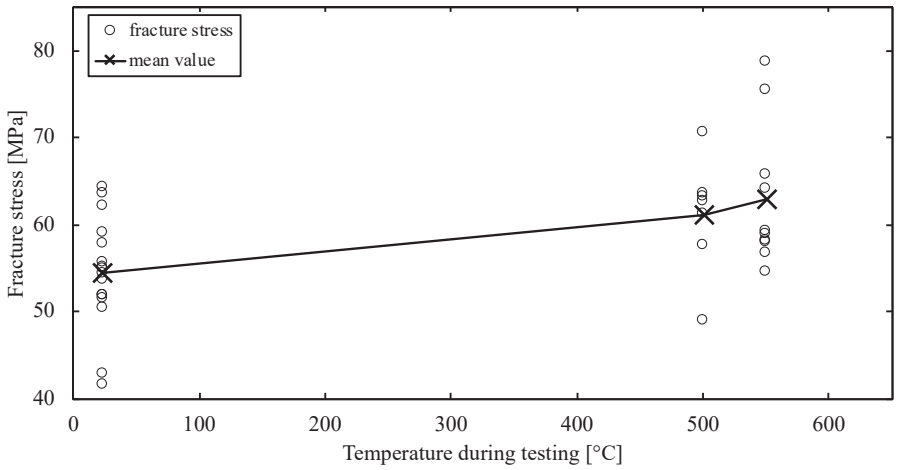

Figure 4-4 Fracture stress versus testing temperature.

5 Conclusions

In this publication an own method was presented for pre-damaging, heat treatment and testing, at elevated temperatures, of commercial soda-lime silica glass as float glass. Through the experimental investigations and the comparison to previous studies it could be shown that the presented method for determining the bending strength of soda-lime silica glass at elevated temperatures provides reproducible results. Although the sample size of the individual test series at different temperatures is comparatively very small, a trend is already apparent, namely increasing fracture strength with increasing test temperature. As already suspected by other researchers, the increasing fracture strength of the pre-damaged soda-lime silica glass could have its origin in a possible crack healing at elevated temperatures.

6 Outlook

Further experiments to determine the bending strength of soda lime silica glass as float glass are planned for the future. To check to what extent the test speed has an influence on the resulting fracture strength of the soda-lime silica glass, the testing speed will be increased to check the load rate dependency of the material at elevated temperatures. As a further type of pre-damage, centrically drilled specimens are to be tested with the help of the experimental setup presented here. Furthermore, borosilicate glasses are to be included in the investigations. If a larger sample size per test series can be realized, Weibull analyses are carried out in order to be able to make statistically sound statements about the relationship between test temperature and fracture strength.

7 Acknowledgements

Special thanks go to the Institute of Materials Technology in Darmstadt, which provided the experimental apparatus, designed and built the test setup and finally performed the double ring bending tests at elevated temperatures. Furthermore, we would like to thank the Deutsche Forschungsgemeinschaft (DFG, German Research Foundation) – Projektnummer 399287305, which provided financial support for this research project.

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