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Residual Stress in Glass Components

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Architectural trends have tended towards curved glass envelopes and maximised transparency by reducing solid fixing areas. One approach towards transparent glass connections is a heat bonding process based on the principles of welding. This paper investigates the level of residual stress in soda lime silica and borosilicate glass caused by a heat-based connection or forming process. Nominal levels of residual stress prior to heat impact, directly after heat impact and after annealing will be measured on small-scale samples, utilizing a scattered light polariscope (SCALP). Material properties the large temperature range required for the heat bonding process have been identified to allow subsequent numerical modelling to verify the results obtained in this study.

Keywords: Structural glass, residual stress, welding, glass connections

1. Introduction

During the past decades a vast development in structural glass envelopes and enclosures could be observed, aiming to achieve a maximum amount of transparency.

The development from an infill material to a structural material enabled designers to develop buildings that are based on using a large amount of glass i.e. atriums skylights and structural glass enclosures.

These Glass structures feature the ability to merge with their surroundings and become invisible, nearly dematerialised if the connections are kept to a minimum. This requires a large amount of structural engineering, detailed analysis and precise detailing to achieve safe sufficient structures. Although significant amount of research in transparent bonding materials and bonded connections has been undertaken in recent years, solid metal connections are still commonly used to form structural glass connections.

With the development that can be observed in structural glass, tending to an optimisation of connections and production capabilities, leading to a reduction of the amount of fittings and an increase in the transparency of glass structures, however, to overcome the necessity of opaque connections, further research is required to innovate in this respect as opposed to improve existing technology. One experimental proposal is the heat bonding (welding) of borosilicate components to achieve mono-material transparent connections. Borosilicate is chosen in this case, due to its low coefficient of linear thermal expansion (3.3 for Borofloat 33, 8.4 for soda lime silica). The welded connections themselves shall not be discussed in this paper, the focus shall in fact be on the analysis of the heat impact itself on the glass and resulting residual stress from heat impact caused by heat bonding of two glass components.

2. Material Properties

To study the behaviour of a material in temperature range, it is essential to understand the material properties in relation to temperature. Commonly, material properties are established for a small temperature range only, however, to understand the behaviour of the material when heat bonded, larger temperature ranges need to be considered. Key thermal and mechanical properties have been obtained from manufacturer's literature [Schott Borofloat 33, 2013] and will be introduced in this chapter. Material properties are highly dependent on the chemical composition of the material, and even relatively small variations in composition might result in significantly different behaviour. The chemical composition of the investigated glass [Schott Borofloat 33, 2013] is shown in Figure 1.

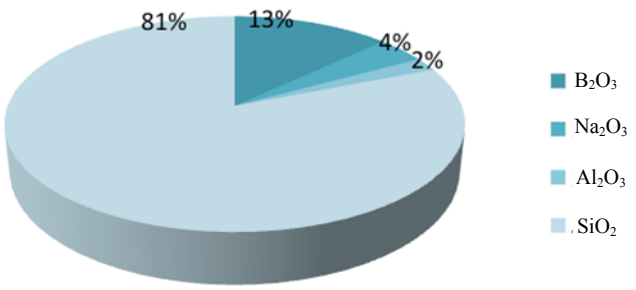

Fig. 1 Chemical composition of Borofloat 33

2.1. Borosilicate

Borosilicate glass is primarily used in chemical and pharmaceutical industries due to its high chemical resistance and low coefficient of linear thermal expansion, which is essential when substances are to be heated in test tubes.

For the same reason, borosilicate is commonly used as fire resistant glazing. However, due to a smaller production it is more expensive than soda lime. Until Schott developed a Microfloat process for borosilicate in 1993, it was produced in a drawing process resulting in larger surface deviations. Maximum available standard sizes here are smaller than for soda lime, however building relevant sizes can be achieved.

Thermal tempering of borosilicate is extensively more sophisticated than the thermal tempering of soda lime. However, by rapid quenching and a decrease of the quenching temperature, Schott have developed a process to overcome the problematic caused by the low thermal expansion and can produce thermally tempered borosilicate.

2.2. Material Properties Comparison at Ambient Temperature

Material properties of soda lime silicate glass and borosilicate glass are compared in Table 1.

Table 1: Material properties for soda lime silicate and borosilicate as obtained from (Petzold et al., 1990).

Property	Soda Lime Glass	Borosilicate Glass
Density [kg/m^3]	2490	2230
Scratch hardness on the Mohs hardness scale	6-7	4.5
Coefficient of mean linear expansion $\alpha \cdot 10^{-6}$ [K^{-1}] (20-300 °C)	8.4	3.3
Thermal Conductivity [W/m^{2}K]	450 × 30	4500
Softening point [°C]	710-735	825
Processing temperature [°C]	1015-1045	1260
Modulus of Elasticity E [N/mm^2]	70000	63000
Poisson Ratio [-]	0.2	0.2
Bending Strength [N/mm^2]	30	30
Compressive Strength [N/mm^2]	700-900	700-900
Tensile Strength [N/mm^2]	30-80	70
Maximal thermal shock resistance ΔT_{max} [K]	68.02	192.4

2.3. Thermal Shock

Whenever glass is rapidly cooled, thermal shock is one of the immediately resulting issues causing a high breakage potential. During the process of tempering the cooling rate creates differing temperatures on the surface and in the core of the glass, leading to a stress differential, however, also temporary stress is induced in the glass though a temperature gradient Although the stress formed during cooling is temporary, failure can occur caused by the differential of surface and core temperature. The maximum possible stress will occur if the surface is instantaneously cooled while the core remains at the higher temperature. Under these conditions stress is given by (Shelby, 2005):

$$\sigma = E\alpha\Delta T/(1-\vartheta) \quad (1)$$

ΔT: difference between surface and core temperature
α: thermal expansion coefficient of the material

2.4. Specific heat capacity

The heat capacity of a material is the amount of energy required to alter (increase or decrease) the temperature of an object by one degree Kelvin. The specific heat capacity for the borosilicate used in the tests described in this paper is shown in Figure 2 as obtained from manufacturer's literature [Schott Borofloat 33, 2013] up to 500 °C.

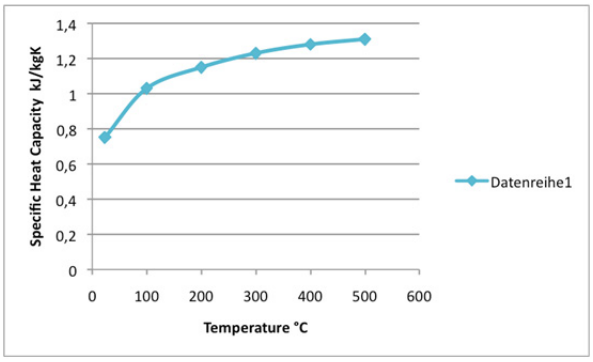

Fig. 2 specific heat capacity of Borofloat 33.

2.5. Thermal Expansion

Thermal expansion (linear or volumetric), is the tendency of the material to change the volume due to the temperature increase. Typical measurements of the thermal elongations are up to the transition temperature as shown in Figure 3 provided by Schott [Schott Borofloat 33, 2013].

To describe the thermal expansion of a glass, three main factors are to be considered: the thermal expansion coefficient, the glass transformation temperature and the softening temperature. While the thermal expansion coefficient indicates the relation between the volume of the glass and its temperature, the glass transformation temperature indicates the begin of the viscoelastic behaviour and the softening temperature (dilatometric) indicates the begin of flow under modest load [Shelby, 2005].

The thermal elongation as provided by the glass manufacturer [Schott, 2013] is shown in Figure 3 comparing the utilised Borofloat 33 with Pyrex borosilicate 3.3 and pure Silicon.

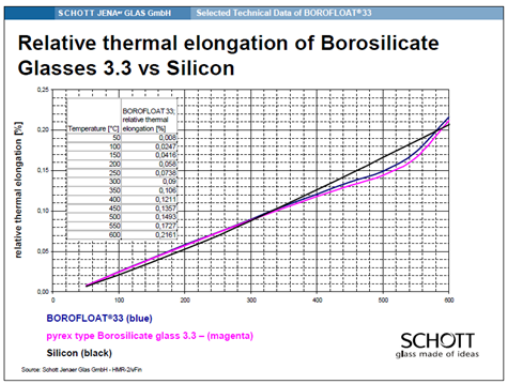

Fig. 3 Thermal elongation as obtained from [Schott, 2013].

The thermal elongation data provided was utilised to calculate the coefficient of linear thermal expansion for a temperature range up to approximately 600 °C (Figure 4). Unfortunately values above these temperatures could not be obtained, although these would be required to verify test results with a viscoelastic numerical model.

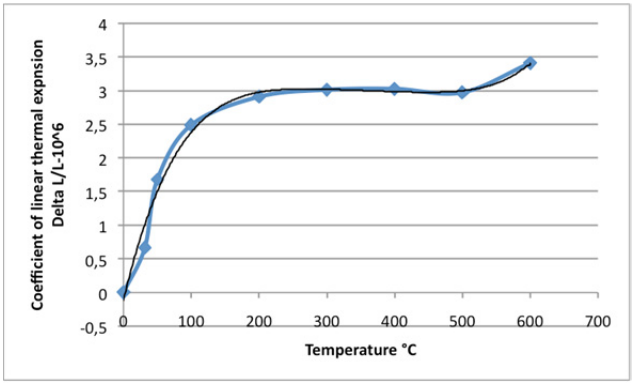

Fig. 4 Coefficient of linear thermal expansion over a temperature range up to 600°C.

2.6. Refractive Index

Glass is a solid that transmits light in the visible spectrum, which has not only made it to be a great building material as it transmits light into the building while forming a protecting layer, but this also allows to measure stress in the material with the help of a visual light polariscope.

The interaction of light with the electrons of the individual atoms of a glass determines the refractive index. If either electron density or polarizability are increased, the refractive index increases, too. The RI of the borosilicate measured in this study is 1.473 according to data provided by the manufacturer, compared to 1.51 for soda lime glass.

3. Test setup

3.1. Heat impact on material structure and residual stress after heat bonding (welding)

To understand the local heat impact of a welding process on borosilicate components, a physical test has been carried out. Although connections between two glass components have been established, these shall not be discussed in this paper, as the focus shall be on the influence of the temperature on the residual stress of the glass component irrespective of the geometry. To represent the heat bonding process while achieving a neutral geometry that would not influence residual stress measurements, flat specimen with a size of 100mm x100mm have been heated locally to working temperature and then undergone a controlled cooling process. Residual stress measurements have been carried out on these borosilicate specimens that have undergone a local heat treatment using temperatures high enough to achieve a chemical bond between two specimens. Temperatures required to achieve a chemical bond have been established in previous experimental tests (Rammig, Direct Glass Fabrication, 2010) where specimens where heat bonded and underwent fracture mechanical testing to proof that the bond is stronger than the parent material. These tests were established on an experimental basis and further mechanical testing will be required to obtain significant results, however the results suggest, that chemical bonds can be achieved in a heat bonding process.

To achieve a heat bond between two borosilicate specimens, the glass requires local heating to working temperature. This is only possible if the entire specimen requires heating to Tg to avoid thermal shock related breakage. To minimise visual distortion locally, where the specimens have been heated to working temperature, it has been experimented with temperature exposure prior to the manufacture of the specimens that were used for the stress measurements. For these experimental trials two components were bonded to understand temperature ranges required to achieve an atomic bond. Insufficient heating of the entire specimen easily leads to breakage, while the local temperature impact could be optimised for a short duration, reducing visual distortion and still achieving sufficient bond between two specimens.

10 specimens of 50mm x 50mm and 4mm thickness have been tested, 5 of which have undergone the heat treatment and 5 of have been measured untreated. Residual stress has been measured on 5 points on the specimen as shown in Figure 5. Each point has been measured in two directions, x and y at 90 degrees to each other and perpendicular to the edges of the specimen. Initial measurements were taken prior to the heat treatment to monitor residua stress levels of the fabricated glass, the second measurements were taken directly after the welding process, when the specimen had only undergone a controlled cooling process, but have not been annealed, and the final measurements were obtained after the annealing process, to understand whether the stress induced can be fully released.

A fracture mechanical evaluation of the influence of the temperature on the strength of the material is anticipated through ring-on ring tests, however this paper only discusses the impact of the process on residual stress.

Fig. 5 test specimen with measuring points.

The specimens have been heated with a burner utilising earth gas and oxygen in adjustable quantities. The air –gas mixture was adjusted manually, to gradually increase the temperature of the flame and heat up the specimen. Two different nozzles have been used, a larger opening for a big, low temperature flame (up to 600-800°C) and a smaller nozzle to achieve a slim focused flame with temperatures up to 1500 °C to locally heat a small are of the glass (∅ 10mm). As no heat gauge could be fitted on the nozzle of the burner or the specimen, a thermal camera (FLIR-T 640) has been used in the heat range mode of 300°C-3000°C to monitor temperatures of the specimens. The camera offers three temperature ranges for operation; -40-100°C, 150°-600°C and 330°-2000°C. Given that required temperatures are significantly larger than 600°C, the highest temperature range was chosen for the measurements, despite the expectation of inaccuracies of measurements in the temperature range below 300°C.

To assure that the heating process is repeatable in the most similar way despite using a manual process, the specimens were retained in position in a welding jig (Fig. 6). Two steel clamps keep the glass in position allowing expansion as a non-combustible tape with low friction is used to separate the glass from the steel clamp and allow expansion while retaining the specimen in position.

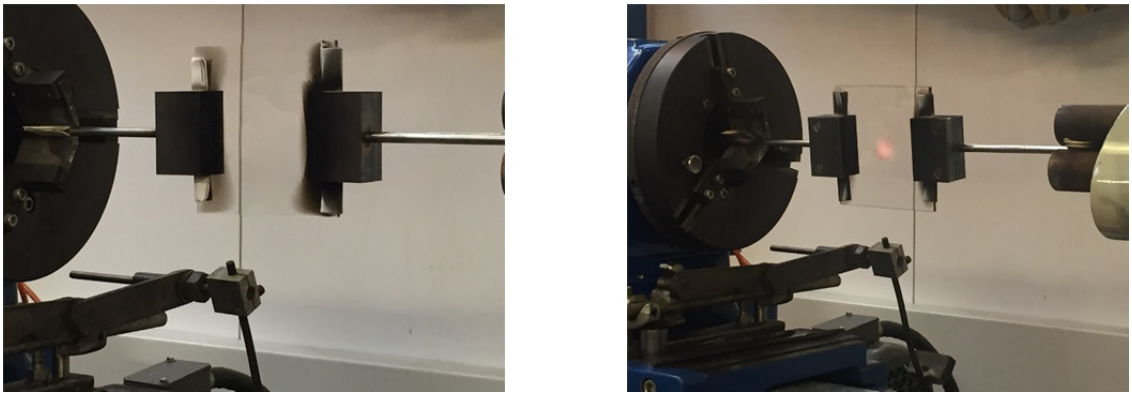

Fig. 6 Specimen in welding jig.

To avoid breakage due to thermal shock during the process of temperature impact, the entire specimen requires heating up to temperatures 600-700°C prior to local heating of the specimen centres to working temperature (~1200°C). After the heat impact that typically was held for 35 seconds. Temperatures have been monitored with a thermal camera to understand heat impact over time and max temperatures are shown in Figure 9. After the heat impact in the centre of the specimen, the entire specimen requires controlled cooling to room temperature before the glass needs to be annealed in a full annealing cycle over 20 hours (Fig 8) to remove locked in residual stress from the temperature impact.

3.2. Annealing

The annealing of glass is a method to ‘relax’ stresses that have been locked in through a heat impact i.e. in this case a bonding process. Annealing is equally important to commonly used glass processing techniques such as heat gravity bending (‘slumping’). After the heat impact the glass requires an additional cooling process in which controlled temperature drop leads to a relaxation of locked-in stress. The exact adherence to maximum temperature and different heating and cooling rates during the annealing cycle is of high importance to achieve a continuous annealing result with a homogeneous stress distribution in the component [Greil, E., 1964].

The relaxation temperature of the annealing cycle is defined by the glass composition. Fig. 7 shows annealing temperatures for several glass compositions, including the Borofloat 33 utilised in the tests.

Glass type	Relaxation Temp.°C	Glass type	Relaxation Temp.°C	Glass type	Relaxation Temp.°C
Schott, Mainz:		Kolbenglas	562	**Quickfit:**	
DURAN 50	575	Leuchstoffroehrenglas	530	Laborglas	565
Geraeteglas 20	575	Leuchtstoffroehren-Farbglas	510	**Philips:**	
Therm Gl 16	544	**Ruhrglas**		Gluehlampenglas 01	435
Therm. Gl 2954	596	Apparateglas	520	Gluehlampenglas 03	510
Supremax 56	750	Roehrenglas	496	Fernsehkolbenglas 162	465
Supremax -	722	Ampullenglas	538	**Sovirel:**	
Supremax	573	Leuchtroehrenglas	506	Pyrex	545
Fiolax clear	571	**Osram:**		**Borosilikatglas 73201**	**562**
Fiolax brown	566	Bleiglas-M	435	Borosilikatglas 74001	550
Bleiglas	429	Bleiglas	425	Borosilikatglas 74644 et al	480
Uvioglas	452	Roehren-Normalglas	505	Borosilikatglas 75001	508
Fernsehkolbenglas	445	Magnesiaglas	515	Bleiglas	438
Glaswerk Wertheim		Wolframglas	528	**Tschecjische Glaeser**	
Geraeteglas	538	Hartglas	743	SIMAX Glas	536
Roehrenglas fuer Automaten	530	**Thueringer Glaeser:**		SIAL Glas	562
Resistenzglas	582	Rasotherm	570	Ampullen Neutral Glas	581
Sterilisatiosglas	566	Gerateglas G52	605	ThermometerGlas PN	550
Bleiglas	550	Geraeteglas 399	540	Einfaches Fenster und Behaelterglas	530
Molybdaen-and Kovar Glas	428	Gegeef	560		
	512	Fischer Prima	530		

Fig. 7 Annealing temperatures for several glass compositions [Greil, E., 1964].

To achieve a relaxation of stresses, the glass requires a homogeneous heating of the entire component so that stress can be released through the plastic behaviour of the heated material. The cooling through the transformation phase of the glass has to be slow enough to avoid further stress being locked in. This means that the cooling rate is the crucial factor in the annealing process. It is dependant on the thickness of the material as well as the composition, which cooling rate is chosen. The annealing cycle used for the specimens tested is shown in Figure 8 and is based on material properties obtained from literature [Schott Borofloat 33, 2013] and [Greil, E., 1964].

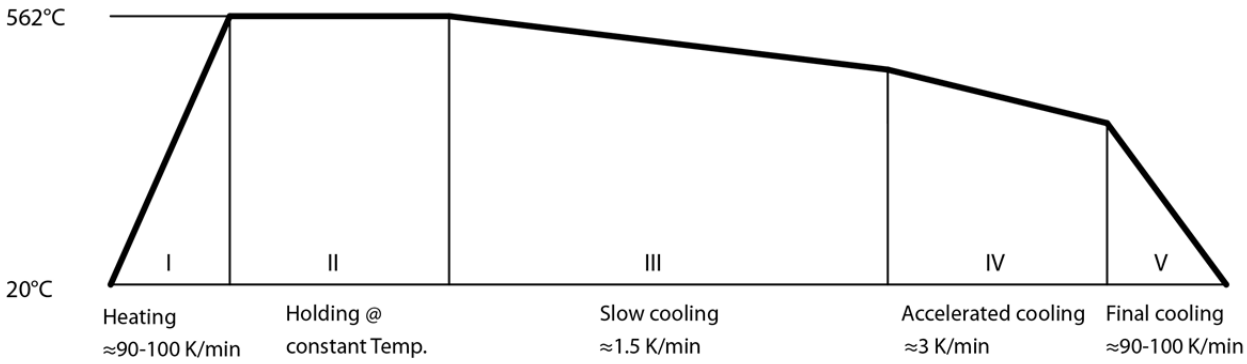

Fig. 8 annealing cycle for borosilicate specimen

4. Test results

It was expected that residual stress would be relatively consistent on small specimens prior to the heat treatment and deviations and measurement errors were predicted to be low. Similarly it was expected that residual stress levels on the annealed specimens would be low, however deviations were expected to be larger. Directly after the heat impact, larger variation on residual stress within the specimens was predicted as well as significant stress in the glass due to the high Temperatures applied.

4.1. Visual assessment of residual stress

Residual stress was visually monitored under a polarisation filter prior to heat impact, just after heat impact and after annealing. All visual assessments were carried out at a consistent room temperature of 24°C.

Residual stress at the three stages mentioned is shown in Fig 9.

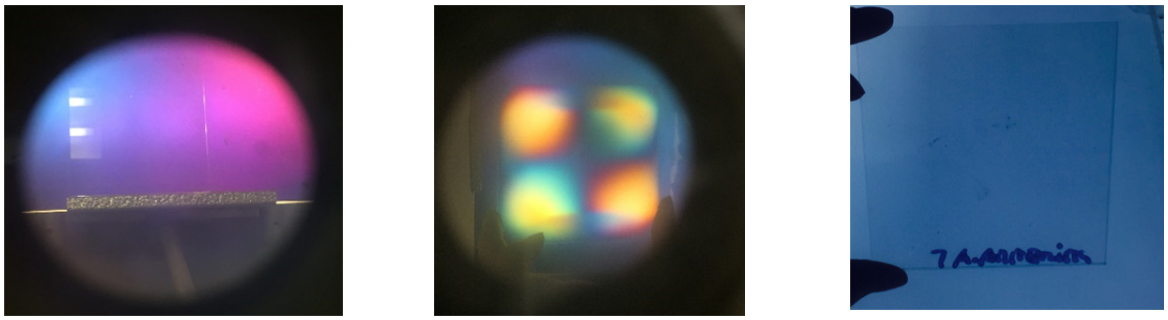

Fig 9a), b) and c): residual stress under polarisation filter before and after welding and after annealing.

Prior to the heat impact, no stress could be observed under the polarisation filter, the specimen appears to be evenly annealed with residual stress levels to small to be visible under a polarisation filter. Figure 14b shows the distribution of residual stress immediately after the heat impact. The glass has been cooled to room temperature in a controlled way (Figure 12), however, the fast cooling process leads to stress being locked in. With the centre of the specimen having been heated, the stress distribution appears relatively evenly in the opposite corners of the specimen, with slight non-uniformities in the areas the specimen was clamped to the jig.

After the annealing process, again, no visual stress could be observed in the visual assessment through polarised light (Fig 9b).

4.2. Residual stress measurements

Non-destructive testing on the surface stress has been carried out using a scattered light polariscope (SCALP 5) to understand the impact of heat induction and annealing on the residual stress of the glass.

The SCALP operates by sending a polarised laser through the thickness of the glass. The laser beam scatters on the particles of the glass and the intensity of this scattering is recorded through the thickness of the glass, the device obtains the absolute optical retardation at every point of the beam, which is then converted to stress values [GlasStress, 2013].

Residual stress measurements on the heat treated specimen have first been carried out immediately after the heat treatment, just allowing the specimen to cool down to room temperature (24°C) and have then been repeated after the annealing process.

The annealing cycle used is optimised for the borosilicate used (Borofloat 33) and is shown in Figure 8.

Ideally residual stress levels prior to heat induction and after annealing should be identical, however, previous tests carried out on slumped annealed glass showed that this might not be the case and the annealing process might not release locked- in stress evenly.

The general effects of thermal history on thermal and mechanical properties are well understood and as such should be taken into consideration.

4.3. SCALP calibration

As the refractive index (RI) of borosilicates differs from the RI of soda lime silicates, the polariscope requires adjustment to be able to measure borosilicates accurately. The RI of the borosilicates used is 1.48 as previously described, which according to the polariscope manufacturer requires a laser angle <75°.

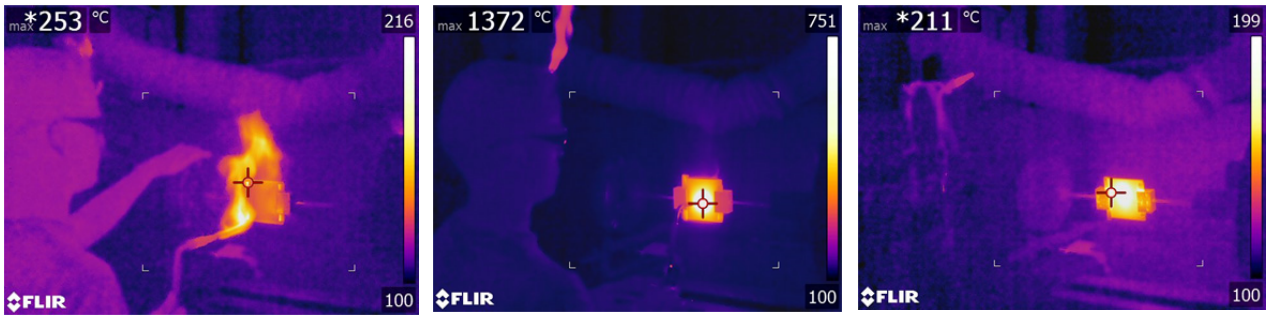

Fig. 10 temperature at 45, 405, and 675 seconds.

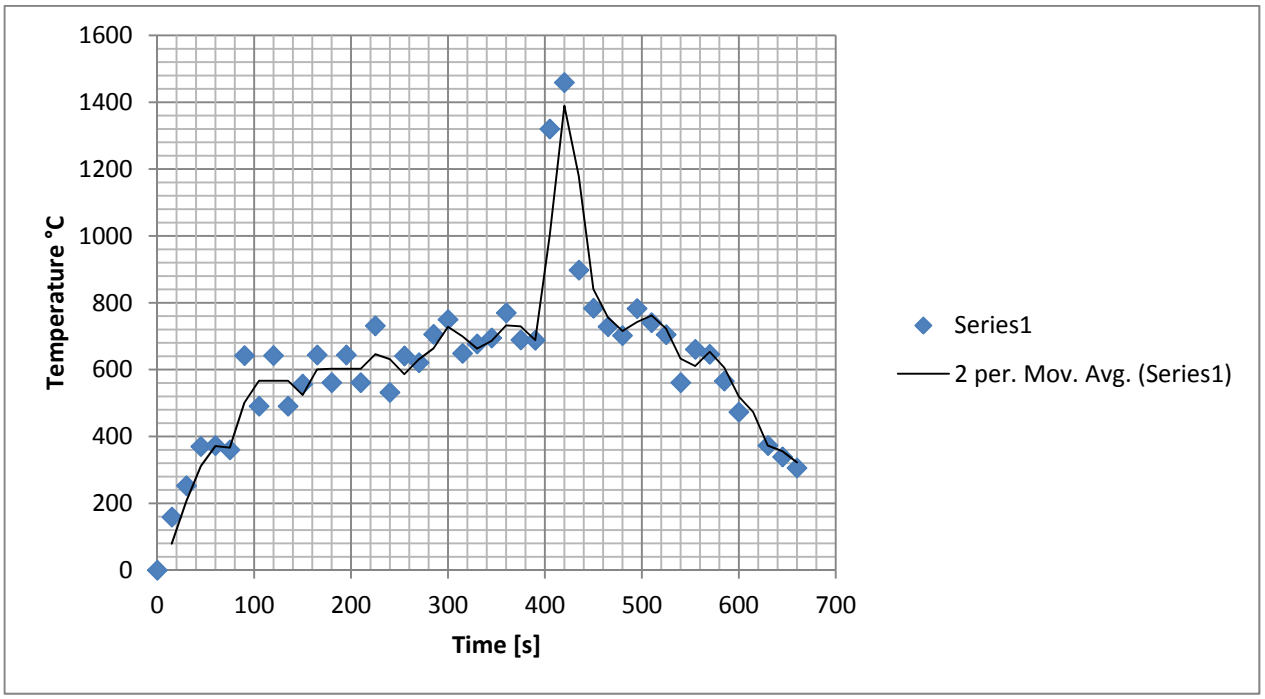

Fig.11 temperature profile for 5 tested specimen.

5. Measurement results

To understand the impact of heat on the glass through a heat bonding or -deforming process, the same three conditions have been analysed: prior to heat impact, directly after a local heat impact and after annealing of the specimen. The specimens were measured on 5 points (Figure 7) in x and y direction.

5.1. Data analysis

The data was analysed using the manufacturer's software (GlasStress SCALP Software version 5.8.1.4). Processing of the results was based on the fit error and the amount of pixels that were excluded from the measurement. The fit error refers to the root-mean-square error of the fitting curve that is used to smoothen retardation results and allow stress calculation. Large values indicate that measurement data is invalid. The manufacturer suggests that acceptable values are between 5 and 15% [GlasStress, 2013].

Pixels not sufficiently reliable for stress calculation are referred to as excluded pixels. Parasitic scattering of the light beam saturates the sensor and leads to an exclusion. Approximately 5 to 15% of the data is lost due to parasitic scattering preliminary at entrance and exit points of the laser [GlasStress, 2013].

Readings with a fit error >10% and excluded pixels of >20% were not considered in the data processing.

Results were separated based on the direction of stress measurement, average stress values were calculated for each direction and finally overall results were transferred in Excel format.

5 points were measured for each specimen in x and y direction. Then principal stress was calculated to:

$$\sigma\ 1, \sigma\ 2 = (\sigma\ x + \sigma\ y)/2 \pm \sqrt{((\sigma\ x - \sigma\ y)^2/2 + 4\ tau,xy^2)} \quad (3)$$

assuming that the shear stress = 0. The SCALP is not capable of measuring shear stress; hence this assumption has been made, as shear stress could not be verified.

Results are shown in Table 2. Measurements of residual stress prior to heat treatment are very consistently in the acceptable range for annealed glass [Haldimann et al, 2008].

Table 2: principle residual stress on specimen.

Measurement point	Prior to heat impact		After heat impact		After annealing	
	Mean principal residual stress (MPa)	SD (MPa)	Mean principal residual stress (MPa)	SD (MPa)	Mean principal residual stress (MPa)	SD (MPa)
1	-3.51	0.68	5.39	1.30	-1.72	1.52
2	-3.24	0.14	-0.6	0.58	-1.10	0.50
3	-3.27	0.31	-0.08	1.76	-1.45	0.96
4	-3.39	0.45	2.51	1.96	-0.90	1.09
5	-3.35	0.51	1.34	0.97	-0.86	0.58

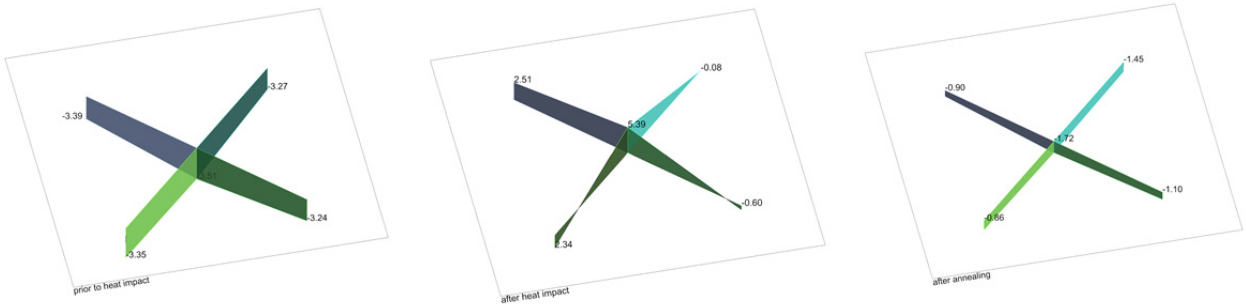

Fig. 12 principle residual stress on specimen prior to welding, after welding at 24° C and after annealing at 20° C.

While the distribution of residual stress prior to heat impact is very even throughout the specimens (Fig. 12), after welding larger differentials can be observed, with compressive values in the corners of the specimen, surrounding the centre point that was exposed to localised heat impact. This matches results previously described under a polarisation filter shown in Fig. 9 although distribution of stress after welding appears to be not as homogeneous as indicated in the polarisation image. However, stresses measured are much lower than expected; The significance of the results obtained is questionable though, as the measurement device has a tolerance of +/- 4 MPa for measurements <20 MPa [Aben, H., Anton, J., et al., 2010], which, given the small values measured, might have a significant influence on the results and suggests that the device is not reliable for measurements of the pre-annealed and annealed glass. After annealing, the stress distribution is more homogeneous again, with a higher standard deviation in results. Deviation in results directly after heat impact is comparably high. The experimental results shall be verified in a numerical model, which will, however require reliable material properties over the large temperature range the glass is exposed to. After annealing, the stress distribution is more homogeneous again, with a higher standard deviation in results. Deviation in results directly after heat impact is comparably high.

6. Conclusion

Temperature impact on the residual stress of glass specimen is shown in the results obtained through experimental testing of borosilicate specimen. The importance of an additional annealing process can be observed, as residual stress is clearly reduced. However, measurement results obtained in areas of large heat impact, where surface deformation of the specimen can be observed, show larger deviations and error rates, so further testing might be required to verify the results obtained in this study. Residual stress levels measured after the heat impact were significantly lower than expected and might not be reliable, as stress is below the threshold where the scattered light polariscope used (SCALP 5) can obtain reliable measurements. Through the process however, it could be verified that a controlled process needs to be followed to heat the glass to T_g, as thermal shock breakages where observed when specimen were heated too quickly. This will require further studies to explore optimised exposure temperatures. The visual assessment of stress shows, that a controlled annealing process, reduces stress induced through a welding process/ heat impact can be relaxed and components do not show significant residual stress or stress differentials that would make the glass unemployable as an annealed glass component for a building application. It is assumed that stress is sufficiently eased out for the glass to undergo further thermal treatment processes, however, this shall be verified in further tests. For application in an industrial process, controlled heating, temperature exposure at T_{max} and controlled cooling would require further optimisation and study.

Advanced numerical analysis utilizing viscoelastic material models will have to be carried out to verify the current results. These rely on the availability of material properties in the temperature range explored, which could not be obtained for this study; hence further research is required. In practice, apart from the use shown in this study, the analysis methodology as well as material properties obtained for large temperatures have a wide variety of applications, including the study of fire resistant glazing or gravity curved glass. The latter is commonly in use, however stress levels are not very well understood so further studies on residual stress of gravity formed glass shall be carried out.

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