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Fused Silica Windows for Solar Receiver Applications

Johannes Hertel[1, a)], Ralf Uhlig[2, b)], Matthias Söhn[3], Christian Schenk[3], Gundula Helsch[4] and Hansjörg Bornhöft[4]

[1] *German Aerospace Center (DLR), Institute of Solar Research, Pfaffenwaldring 38-40, 70569 Stuttgart, Germany, phone: +49 711 6862-786*
[2] *German Aerospace Center (DLR), Institute of Solar Research, Pfaffenwaldring 38-40, 70569 Stuttgart, Germany*
[3] *Heraeus Quarzglas GmbH & Co. KG, Reinhard-Heraeus-Ring 29, 63801 Kleinostheim, Germany*
[4] *Clausthal University of Technology, Institute of Non-Metallic Materials, Zehntnerstraße 2a, 38678 Clausthal-Zellerfeld, Germany*

[a)]Corresponding author: Johannes.Hertel@dlr.de
[b)]Ralf.Uhlig@dlr.de

Abstract. A comprehensive study of optical and mechanical properties of quartz glass (fused silica) with regard to application in high temperature solar receivers is presented. The dependence of rupture strength on different surface conditions as well as high temperature is analyzed, focussing particularly on damage by devitrification and sandblasting. The influence of typical types of contamination in combination with thermal cycling on the optical properties of fused silica is determined. Cleaning methods are compared regarding effectiveness on contamination-induced degradation for samples with and without antireflective coating. The FEM-aided design of different types of receiver windows and their support structure is presented. A large-scale production process has been developed for producing fused silica dome shaped windows (pressurized window) up to a diameter of 816 mm. Prototypes were successfully pressure-tested in a test bench and certified according to the European Pressure Vessel Directive.

INTRODUCTION

Solar Tower systems operate at high process temperatures, thus enabling high conversion efficiencies especially when using combined cycles. A high overall efficiency for the conversion of solar radiation can be achieved by introducing solar energy into a Brayton cycle. Hence, pressurized air receivers are a promising technology. They enable direct solar preheating of the compressor discharge air of a gas turbine before it enters the combustor of the gas turbine. Figures 1a and 1b show schematics of two air receiver types that have been successfully tested under solar conditions, the pressurized volumetric receiver (PVR) and the tubular cavity receiver (TCR).

A window made of fused silica (quartz glass) is a key component of the PVR. The PVR absorber is located in an insulated pressure vessel closed by a domed window to maintain pressure (Fig. 1a). The solar radiation passes through the window and is absorbed in the volumetric absorber which transfers the heat by forced convection to the air stream flowing through it. The PVR was tested successfully within the projects REFOS [1] and SOLGATE [2].

Another application of fused silica windows is as a cover of a TCR aperture in order to reduce thermal losses which are mainly caused by convection (Fig. 1b). Uhlig et al. [3] showed that such a window could increase the efficiency of an 8 MW_{th} tubular cavity receiver with an outlet temperature of 800 °C by 8 percentage points. Since the receiver operates under atmospheric pressure, tightness is not necessary and so a segmented approach can be chosen. The advantages are avoiding large parts, easier manufacturing and maintenance and reduced failure risk. A prototype of a segmented pressureless window has been developed and successfully tested within the ARTRANS project [4]. Although the windows are not exposed to pressure, they are mechanically stressed due to fixtures, wind loads, thermal gradients and other effects.

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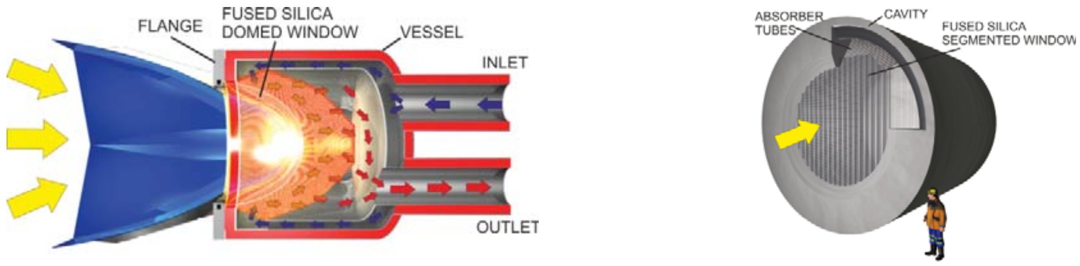
Fused silica is highly transparent for solar radiation and allows operation temperatures above 1000 °C. Mechanical properties (rupture strength) and necessary safety factors must be known for proper layout and design.



(a) (b)

FIGURE 1. (a) Pressurized Volumetric Receiver, (b) Cavity receiver with segmented pressureless window

MATERIAL EXPERIMENTS

Mechanical Strength

Like any type of glass, fused silica is very sensitive to surface defects that influence strongly the mechanical strength. It is important to know how rupture stress changes at elevated process temperatures. Above all, fused silica surfaces are prone to surface devitrification, i.e. crystal formation, when exposed to environmental contaminants at elevated temperatures.

Material testing with focus on the effect of surface defects on the ultimate strength was performed. For every defect a batch of samples has been subjected to a coaxial double ring test according to DIN EN 1288-5.

Four test series have been prepared from one lot of machined plate samples (size 100x100x5 mm³), with a standard mechanical polish. Series #1 shows the original polished surface condition. It is to be noted that generally the grade of mechanical polish has a strong influence on rupture strength. Subsequently, series #2 and #3 have been treated with NaCl and heat to generate devitrifications. Series #4 has been sandblasted on a 9 mm concentric spot with high purity natural quartz grain.

In order to remove any influences from edge conditions, coaxial double ring tests (18 mm / 90 mm) have been used to investigate the strength of each series.

The results of these measurements are summarized in Table 1:

TABLE 1. Summary of the results of the ring-on-ring test series

	Test series			
	#1	**#2**	**#3**	**#4**
Number of samples	33	34	34	29
Average failure stress in MPa	91.0	104.7	109.5	52.5
Max. measured failure stress in MPa	166.0	121.4	139.0	58.3
Min. measured failure stress in MPa	47.0	84.9	72.5	38.4
Weibull modulus m	4.5	13.5	6.5	13.5
99% calculated survival probability in MPa @	26	75	54	40

As can be seen from the table, each surface condition yields different strength levels and different scatters of the individual fracture stresses within the series. The scatter of a series is described by the so-called Weibull modulus m. A high Weibull modulus stands for a lower scatter of values – and for a more reliable and predictable material strength. Since the only variation for each test series is the surface condition, any difference can be related to this parameter. The lowest strength values have been measured for the samples with sandblasted surfaces (#4). In comparison, the samples from the original (polished) surface series (#1) are significantly stronger, but the individual strength values in this series are far more scattered than any of the three others.

Remarkably, the sample series #2 and #3 with devitrified surfaces yield higher average strengths than #1 with original surface condition. Above all, the slightly devitrified samples (#2) reach the same high Weibull modulus of 13.5 as the sandblasted series #4. Series #3 with the significantly devitrified surfaces reaches $m_3 = 6.5$, a value still being higher than series #1 with the polished surface condition.

In order to estimate the reliability, i.e. the probability of surviving a certain stress level, linear regression has been applied to the experimentally determined stress distributions. From Table 1, the 99th percentile of the four reliability distributions can be taken for all four test series. These values quantify the stress level that can be survived by each series with a probability of 99 %. As can be expected from the different scatter of values and the resulting Weibull moduli, the highest 99th percentile value is 75 MPa for the slightly devitrified series #2, followed by the significantly devitrified series #3 (54 MPa). In comparison, the original polished series #1 has a 99th percentile value of 26 MPa which is even lower than the weaker - but less scattered - series #4 with sandblasted surfaces (40 MPa).

It has to be noted that the fused silica windows have been designed by FEM calculations for a maximum tensile stress level of 6 MPa. Even the lowest 99th percentile stress of series #1 with polished surfaces is at 26 MPa, still well above the design stress. On the other hand, the calculated survival probability at the design stress of 6 MPa is above 99.99 % for all series.

In order to determine whether there is a significant temperature dependence of the material strength, two sets of identical rod samples (45 x 4 x 3 mm³) have been tested in two series at room temperature (#5) and elevated temperature (800 °C - #6). Four point flexural tests have been performed by Bundesanstalt für Materialprüfung, Berlin. Note that due to different geometries and mechanical preparation of the samples, the absolute values obtained for series #5 (room temperature) do not match with those from series #1, although both have polished surfaces. Therefore, the absolute numbers from test series #1 to #4 cannot be compared directly with those from #5 and #6.

TABLE 2. Summary of the results of the four-point test series

	Test series	
	#5	**#6**
Number of samples	32	30
Average failure stress in MPa	155.6	293.5
Max. measured failure stress in MPa	210	374
Min. measured failure stress in MPa	60	163
Weibull modulus m	4.5	6
99% calculated survival probability in MPa @	51	134

As a remarkable result, the samples tested at 800 °C (series #6) proved to be significantly stronger than those tested at room temperature (series #5). The statistical scatter as indicated by the Weibull modulus is slightly less for the high temperature series #6 (m = 6) than for the room temperature series (#5; m = 4.5).

Therefore, it can be concluded from these results that pressure proof testing of the windows at elevated temperatures is not necessary. The lower mechanical strength of the material at room temperature allows a more conservative – and simpler – proof testing for the intended application.

Crystallization

The crystallization of fused silica to cristobalite caused by high temperature treatment is widely investigated [5], [6], [7], [8], [9]. The long term behavior of crystallization has been analyzed by samples with typical temperatures and environmental contamination. Therefore, segments of fused silica tubes were heat treated in a gradient furnace between 850 and 1100 °C, in cycles of 55 times for 6 h. Each heating step was followed by a cool-down step to less than 140 °C within 16 h. Afterwards the samples have been heated up again within 2 h. Investigating the samples by optical microscopy after 55 heating cycles in clean atmosphere, no general crystallization could be observed except for a few isolated spots.

Furthermore, the influence of test contaminants on the crystallization of fused silica has been investigated. A defined contamination has been applied to the surface of the fused silica samples with NaCl, Arizona dust (name:

SAE J 726 Rev. June 93) or Chlorophyllin. The samples were then heat-treated between 600 and 900 °C for 6 h. At 700 °C, growth of cristobalite was detected by XRD (x-ray diffractometer) for samples contaminated with Chlorophyllin or NaCl. The contamination with Arizona dust resulted in sintering of particles on the sample surface at 800 °C. Both, the sintered particles from the Arizona dust as well as the crystallization of the glass surface lead to a reduced transmittance of the fused silica.

For studying the effect of repeated heat treatment on the crystal growth, a fused silica sample has been contaminated one-time with NaCl, followed by six subsequent heat treatment cycles at 900 °C for 6 h. After each heating cycle the crystal formation has been investigated by optical microscopy. After six heating steps at 900 °C the crystallized silica surface was identical with the condition after the first heating step, so no continued crystal growth could be observed. This means that isolated crystallized areas do not necessarily trigger a general crystal growth over the whole silica glass surface.

DESIGN OF FUSED SILICA WINDOWS

Design Criteria

The low thermal expansion coefficient of fused silica leads to very low thermal stresses in case of location and time dependent thermal gradients. As the window has to be supported by another structure, the difference in thermal strain of this support structure and the glass is the most critical design issue. The thermal expansion coefficient of metals is about 20 times higher than that of fused silica. If the window would be supported by a bounded metallic support structure, the difference in thermal strain would lead to unacceptable high stresses. Therefore, the glass parts and the support structure need to have the flexibility to compensate the difference in thermal expansion.

Detailed FEM simulations had to be carried out to analyze the stresses caused by thermal gradients, different thermal expansions of glass and support structures and additional forces like e.g. pressure or wind load. As fused silica can tolerate higher compressive stresses compared to tensile stresses, the design should try to guide the forces in a way that mostly compressive stresses occur. Aside from manufacturing aspects, the scale up of the size is also limited due to the pressure load. The pressure load increases squarely with increasing surface.

A segmented design of pressureless windows is a good way to handle these critical issues. The segmented pressureless window of the ARTANS project follows this approach using several glass segments supported by an insulated frame.

Segmented Pressureless Window

There are several approaches to reduce the convection losses of cavity receivers by the use of window segments. The segments are arranged to cover the receiver aperture either partially or completely depending on the aperture size. Complete cover of the aperture can be achieved by arranging the segments next to one another (Fig. 1b). The segments are stuck at their ends in a groove of an insulation material. This insulation material has to keep the window in position, has to allow a soft bearing and has to prevent that the segments touch each other. The segments and their support are held by a frame for an easier handling and mounting. Tube diameter and section angle (e.g. quarter or half tube) can be specified depending on the aperture area. For large aperture areas, half tube sections are advantageous due to the higher stiffness. A window of 3x3 m² with a tube diameter of 197 mm as a typical size was CAD-designed.

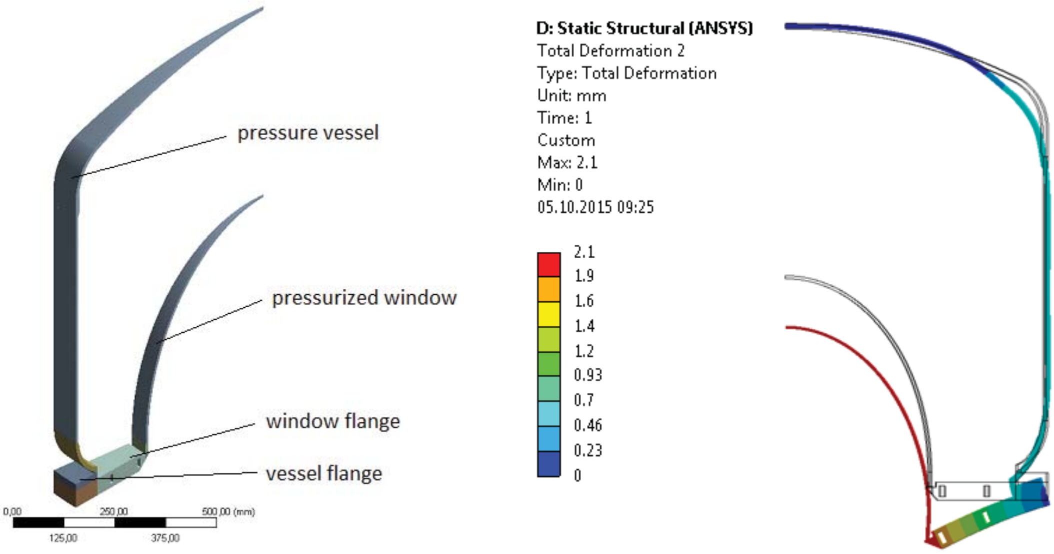
For that design, FEM simulations were performed in order to identify the (maximum principle) stresses in the window. The simulation model consists of one window segment. The calculation considers the temperature distribution, Earth gravity and wind forces. A frictional contact definition was used to model the contact between the window and the insulation material.

The highest stresses are caused by the wind pressure. Due to the small thermal expansion of the fused silica window, the temperature does not result in any significant stress in the window. The highest tensile stress was calculated to 4 MPa, below the design strength of 6 MPa.

Dome Shaped Pressurized Window

As depicted in Fig. 2a, the pressurized window of the PVR (Fig. 1a) is placed on the window flange. The window is pressed on the window flange due to the pressure in the receiver. The different thermal strain of the flange and the window is compensated by a graphite gasket which also seals the receiver (located between the window flange and the window). The domed design of the pressurized window was chosen to reduce bending stresses.

An FEM model of the metallic vessel parts, the sealing and the window was used to analyze the stresses for several load situations (Table 3). The simulation considers pressure, temperature distribution and Earth gravity. The model uses nonlinear frictional contact definition between the components window, graphite sealing and flange. This is necessary as the relative movement between these parts is a key functionality in the receiver design. Modelling the complete vessel is necessary in order to consider the bending of the window flange which is influenced by the vessel as Fig. 2b shows. Maximum principle stresses have been determined. It was found that for a pressure of 10 bar the design value of 6 MPa is not exceeded even with a wall thickness as small as 8 mm.



(a) (b)

FIGURE 2. (a) FEM model, (b) bending of the window flange and deformation of the pressure vessel (55-times magnification)

TABLE 3. Load cases considered in the FEM calculations

Load cases	Value
Start-up	Operation pressure: 0.9 MPa; Gravity Cooling channel temperature (window flange): 10 °C
Solar operation	Pressure: 0.9 MPa; Gravity; Temperature window: gradient 900-800 °C Cooling channel temperature (window flange): 50 °C
Sudden shading (due to passing clouds)	Pressure: 0.9MPa; Gravity; Temperature window: gradient 900-800 °C Cooling channel temperature (window flange): 10 °C
Pressure test	Test pressure: 1.42 MPa; Gravity

MANUFACTURING AND OPERATION

Prototype

Pressurized Window

Commonly, dome-shaped windows for solar thermal applications with 600 mm diameter are manufactured by stepwisely closing a 600 mm diameter fused silica tube on a turning lathe. An improved production route was established by Heraeus Quarzglas for the production of large dome shaped solar windows. This mold-based process allows the formation of large-size rotational-symmetric blanks which are close to final shape, thus reducing the number of hot forming steps and rework processing efforts. The process is capable of handling large production quantities and allows a high reproducibility. The final size is scalable, yet limited by available molds and machine capability. For the first test, a window series with the standard diameter of 636 mm was produced. Later, the potential of the improved manufacturing route was demonstrated by the production of clear fused ellipsoid windows with an outer diameter of 816 mm (see Fig. 3b). OH content and impurity levels are within HSQ300 material quality (semiconductor-grade natural fused silica).

Pressureless Window

An array of 15 halved tube segments with 3400 mm length has been produced. An electrically fused direct drawn fused silica tube with 197 mm outer diameter and 9 mm wall thickness, material quality HSQ 300 has been used. The tubes were laser-cut in two pieces along their long side. Edges have been ground and polished to decrease the risk of cracks. Finally, thermal treatment was used to remove internal stresses.

Pressure Tests

For the pressurized volumetric receiver, the fused silica window is an essential part of the pressure vessel and has to be certified according to the European Pressure Vessel Directive (PED) to use it commercially. Fused silica is not listed as a specified material for pressure equipment.
Therefore, each window has to be proof-tested at pressures 1.43 times higher than the nominal application pressure for at least one hour. So a pressure test bench for the windows has been constructed and built.

(a) (b)

FIGURE 3. (a) Drawing of the pressure test bench, (b) Prototypes of pressurized window

The test bench is shown in Fig. 3a. The pressure window is placed with the open end onto a graphite gasket located in the notch of the window flange. After filling pressure chamber 1 with water, an isostatic pressure is applied to the convex shape of the window. The pressure history is recorded and documented.

Two pieces of the 636 mm windows and 5 pieces of the 816 mm windows have been successfully tested with at least 15 bars for more than one hour. No pressure-induced damage has been observed. This proves the validity of the DLR design as well as the quality of the produced windows. Both production process and quality assurance have been successfully audited by TÜV Thüringen, a Notified Body for pressure vessels. Therefore, Heraeus Quarzglas is enabled to produce, test and certify pressurized solar windows out of fused silica up to a diameter of 850 mm.

Surface Treatment

Although a transparent fused silica window improves the efficiency of a solar receiver by reducing heat losses, a new source of losses is hereby introduced: Due to the different refractive indices of air and fused silica, a part (~7 %) of the radiation is reflected. The impact of the reflections can be reduced by the surface curvature of the window: Concave surfaces allow a second contact of the reflected light with the glass, thus increasing the amount of light which reaches the absorber. However, these losses are significant for the segmented large area windows.

Therefore, an antireflective coating (AR-coating) is applied onto both glass surfaces. This can be done by the principle of destructive interference (so-called lambda/4 layers). A sol-gel based layer of nanoscaled SiO_2 particles is applied to the surface by dip coating. This has been successfully demonstrated in the previous project ARTRANS [4] for 1 m segments of fused silica. In order to realize the coating for large segments (>3000 mm), an improved process has been developed.

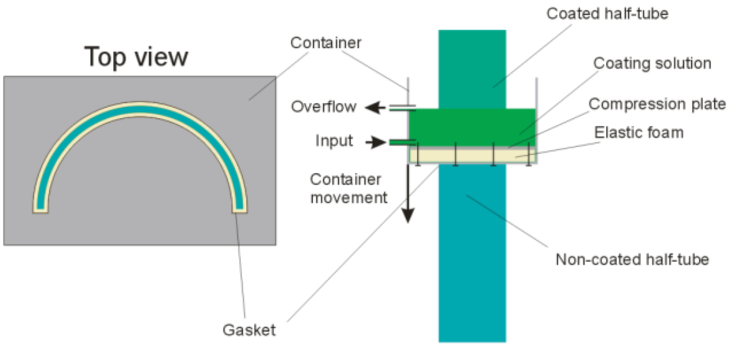


FIGURE 4. Scheme for AR-coating

The setup is shown in Fig. 5: A container, partly filled with sol-gel solution, is moved vertically down along the fixed glass segment. The window passes through an opening at the bottom which is adapted to the cross-section of the half tube. The gap between glass and container is sealed by a flexible foam. Hence, a controlled moving boundary between solvent and glass with constant evaporation atmosphere can be established.

Measure to Reduce Crystallization and Degradation

A daily cleaning procedure will be necessary to avoid surface crystallization, because the daily temperature stress in combination with organic and inorganic contaminants will result in the crystallization of the silica cover glass. However, the cleaning procedure can also contaminate the surface of the cover glasses: especially the AR-coating can be infiltrated due to its high porosity. Therefore the effect of alternating cleaning and heating procedures was studied. Deionized water, water with surfactant, a water/ethanol-mixture as well as tap water were used as cleaning agent. After cleaning procedure the samples were dried in air followed by thermal treatment at 900 °C for 8 h. These alternating heating and cleaning processes were repeated 20 times.

Solar transmittance was measured according to DIN EN 410 with a Perkin Elmer Lambda 950 before and after cleaning and heating cycles. The effect of cleaning and heating was different for the uncoated and AR-coated silica glass. The solar transmittance of the uncoated silica glass was 93 % before and after treatment, independent on the cleaning agent. The solar transmittance of the AR-coated samples was reduced from 97 to 95 % using tap water or water with surfactant. But using deionized water or a water/ethanol-mixture, solar transmittance was 97 % even after 20 cleaning and heating cycles.

In summary, the silica cover glass should be cleaned every morning before starting the power plant to avoid surface crystallization. In the case of AR-coated fused silica the use of deionized water for cleaning is mandatory.

CONCLUSION

The paper describes the material investigation of fused silica windows for high temperature solar receivers. Material testing has been performed investigating the effect of different surface defects (crystallization and sandblasting) on the mechanical strength. The rupture strength at room temperatures and at elevated temperatures (800 °C) has been investigated. It could be shown that a design strength of 6 MPa allows a survival probability of 99,99 % even for samples with surface defects. The samples tested at 800 °C proved to be significantly stronger than those tested at room temperature. Furthermore, the optical transmittance of contaminated and thermally cycled fused silica samples has been examined.

Designs for segmented pressureless and dome-shaped pressurized windows including their integration into high temperature volumetric solar receivers are presented. FEM simulations have been performed to quantify thermo-mechanical stress conditions.

It could be shown that the principal stresses for both the pressurized and the pressure less window design do not exceed the design strength.

An improved process for applying AR-coatings onto large scale segments of the pressure less window has been introduced. Additionally, several cleaning agents and their influence on preventing surface devitrification have been tested. Deionized water reached the best results for AR-coated fused silica.

A large-scale production process for dome-shaped pressurized windows has successfully been developed and realized up to a diameter of 816 mm. Prototype windows have been successfully tested with 15 bar for one hour on a pressure test bench. A Notified Body enabled Heraeus Quarzglas to produce, test and certify pressurized fused silica solar windows according to the European pressure vessel directive up to a diameter of 850 mm.

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