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Process parameters influence on mechanical strength of direct bonded surfaces for both materials: silica and Zerodur[®] glasses

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Direct bonding is of particular interest for optical system manufacturing for spatial application. This technology is already used in terrestrial optical system manufacturing because of the very high precision of the process and complex geometries are able to bond. However, even if a first prototype already passed with success space environment test, quantification and improvement of the mechanical strength of assemblies are essential to validate the assembly's life expectancy and to validate the European Space Agency standards. So, this work proposes to study the influence of process parameters, such as roughness, relative air humidity during room temperature bonding, annealing time and temperature, on mechanical strength of an elementary mechanical structure using a double shear test procedure and cleavage tests. At the same time, the performances of fused silica and Zerodur[®] glasses are compared. For the process considered in this study, a parallel is made between chemical phenomena, surface roughness and mechanical strength. In the end, cleavage tests confirm the choice of the optimal process parameters and highlight a damaging phenomenon of bonded interfaces with successive re-adhesion.

Keywords: direct bonding; mechanical strength; bonding energy; double shear test; annealing temperature; roughness; humidity; annealing time; silica glasses; Zerodur[®] glasses

1. Introduction

Direct bonding consists in joining two surfaces without the use of any adhesives or additional material.[1,2] Usually, by bringing two flat, well-polished surfaces into contact at room temperature, they locally attract to each other by Van der Waals or hydrogen bonds, and adhere or bond. Direct bonding is also known as wafer bonding or molecular bonding. Direct bonding is a process involved in micro-fabrication process on micro-electronics. The main applications are on silicon-on-insulator devices. Silicon-based sensor and actuators, electronics substrates are other examples of wafer bonding applications.[3–5] More recently, this process is used in the manufacturing of high-performance optical systems for terrestrial application such as Fabry–Perot

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interferometers, prism assemblies, etc. For instance, this bonding process has been used in the manufacture of the largest slicer ever used in the multi unit spectroscopic explorer (MUSE) for the very large telescope (VLT) [6] as related in Figure 1. Nowadays, they are of particular interest for spatial instruments applications. Indeed, this is a high-precision production process and assemblies obtained present a dimensional stability due to the fact that no mechanical part or glue is required. In addition, since no adhesive materials are used in those processes, the risks of contamination associated with degassing are avoided, which is another advantage in spatial context. A first prototype has already passed with success the mechanical and thermal environment of space [7] where the constraints involved (thermal fatigue, accelerations, vibrations, etc.) are very different from those encountered on Earth. However, a better understanding of the assemblies' mechanical strength behaviour is required to validate the system life expectancy and to meet the European Space Agency standard.

Mechanical strength of those bonded interfaces depends on the interface defects and the nature of bonds involved. Indeed, room temperature bonding needs flatness and roughness perfectly controlled, and no particles contaminations on surfaces.[8–12] Moreover, room temperature bonding is usually relatively weak; consequently, for some applications, the bonded assemblies undergo an annealing treatment causing changes in the nature of bonds responsible for adhesion and thus strengthen the bond across the interface.[13,14] Therefore, it is necessary to quantify the bonding strength and to improve the mechanical performance of adhesive bonds using the confidential Winlight Optics Company process of surface preparation and without degrading optical performances of the material used or damaging coating applied on bonded optical system. In this study, the Winlight Optics Company process is considered. Thus, it is proposed to improve the understanding of multi-physical couplings and multi-scale mechanical phenomena of direct bonding, by quantifying links and close interactions between chemical phenomena, process parameters (annealing temperature and time, humidity level, etc.), surface roughness and mechanical strength. The final aim consists to understand mechanisms at the interface of mechanical strength reinforcement and to find a way to improve treatment of the assemblies in order to obtain a better structural behaviour. So, in this work, the influence of some process parameters, such as roughness, relative air humidity during room temperature bonding, annealing time and temperature, on mechanical strength of an elementary mechanical structure is



Figure 1. Slicer developed in the MUSE for the VLT.[6]

investigated. First, fused silica glasses and Zerodur[®] glasses, which are specific materials used for spatial optical applications, are quickly presented as well as the sample preparation. At the beginning, the nature of both the materials surfaces is compared using wetting tests and X-ray spectroscopy analysis (XPS). Then, the mechanical strength of bonded interfaces of each kind of samples is also compared using double shear experiments. And performances of fused silica and Zerodur[®] glasses are compared. Afterwards, according to the results, the choice of the optimal process parameters is made in order to increase mechanical strength without degrading material properties. Lastly, a comparison between samples with initial parameters (bonding at ambient temperature) and optimal parameters is performed using cleavage tests.

2. Background on direct bonding process

2.1. Surface preparation

Direct bonding is possible as long as the two surfaces have similar geometries and require clean surfaces free of contaminants.[10,15] Therefore, first steps of the Winlight Optics Company process consist in physical and chemical preparation of surfaces. Several polishing of both surfaces to adhere were manually performed in order to obtain the exact surface roughness (here, roughness is always less than 1 nm RMS), flatness and deflection required. Indeed, a high level of roughness results in a weak contact zone and thus in the occurrence of defects during the bonding process. When the roughness is too great, bonding becomes impossible.[16–19] Then, surfaces undergo a chemical treatment in order to eliminate any contaminating particles.[20] With ambient water, free silicon surfaces are recovered by silanol (Si–OH) groups which are the precursor of the bonding [21,22]:



Then, silanol groups react with ambient humid air to form clusters of water [13,14] on surfaces as shown in Figure 3(a). Indeed, formation of clusters is energetically favourable compared to the formation of one silanol bond. The adsorption of water can be written as:



And the adsorption of an additional molecule of water can be written as:



Thus, the water form clusters on the surface before all the Si–OH groups have adsorbed water molecules. With this chemical treatment, we seek to have the more silanol groups possible on both surfaces. On Figure 2, one component of the roughness is represented, each line represents a bonding site, and obviously a lack of bonding sites causes incomplete bonding.

2.2. Room temperature bonding

Once physical preparation and surface treatment have been completed, surfaces are brought into contact and bonding occurs between clusters of the two surfaces. A process of capillarity occurs due to the humid air trapped between the surfaces.[8] By

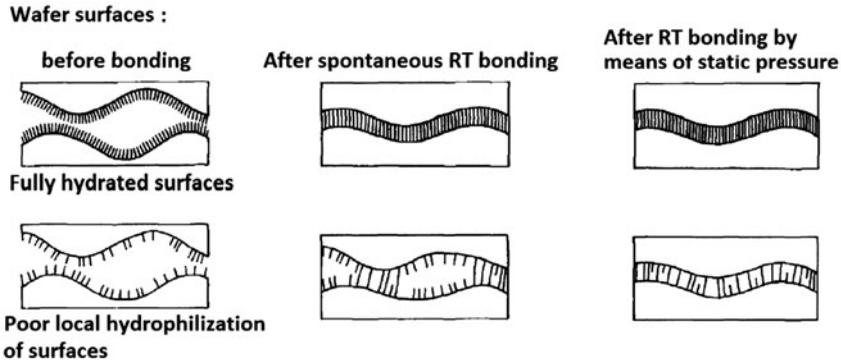


Figure 2. Behaviour of surfaces during room temperature bonding according to their hydroxylated rate (from [13]).

applying a slight local pressure, the surfaces are brought closer together, and hydrogen bonds developed between molecules of water present at the interface and the two surfaces. A bonding wave propagates from the contact point through the whole sample, resulting in the bonding of the surfaces. Molecules of water migrate from the bonding interface and/or diffuse at the interface, bringing the surfaces even closer together and initiating silanol-silanol covalent bonds.[14,23,24]

2.3. Evolution of bonded interfaces with temperature

In order to obtain a stronger or irreversible sealing by increasing the number of covalent bonds, it is necessary to change the nature of bonds at the interface by applying for instance a thermal treatment (Figure 3). Indeed, between 25 and 200 °C, surfaces are contacted via clusters of waters.[13,14] When temperature is lower than 110 °C, chemical reactions at the bonded interface are the same than during room temperature bonding i.e. formation of hydrogen bonds between molecules of water and surfaces.

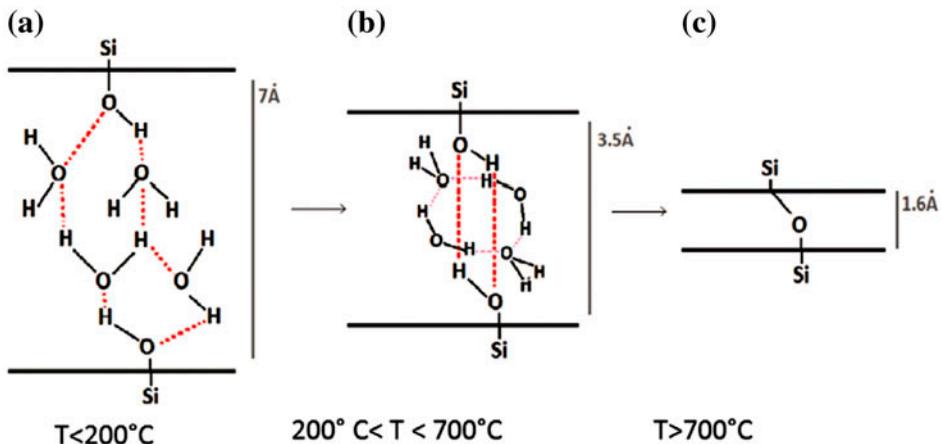
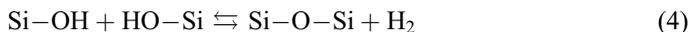


Figure 3. Evolution of the nature of bonds at the interface during thermal treatment.

- (1) Between 110 and 150 °C, molecules of water start to release (Phase I).
- (2) When temperature is between 200 and 700 °C, there is formation of cyclic tetramers of water; surfaces are directly contacted via double hydrogen bond (Phase II) as shown in Figure 3(b).[25]
- (3) Up to 700 °C, the polymerization reaction is triggered, resulting in the formation of covalent siloxane bonds (Phase III, Figure 3(c)) [13,14]:



3. Materials and test methods

3.1. Materials

In optical applications, three materials are mainly used: fused silica glasses, Zerodur[®] and Zinc Selenide (ZnSe). In this work, we chose to study two of them, the fused silica glasses and Zerodur[®] glasses, which are the most commonly used in spatial optical applications with a reasonable cost.

Silica is the main constituent of glass. Crystalline silica has a tetrahedral structure wherein each tetrahedral unit (SiO₄) contains a silicon atom at the centre and four oxygen atoms occupy the vertices of the tetrahedron. However, glass is an amorphous material, therefore, it shows major structural disorder. Fused silica glass can be seen as an anarchistic stacking of SiO₂ tetrahedra in which only short range order is maintained.

Zerodur[®] is a glass-ceramic type invented in 1968 by optical materials producer Schott AG[™] and it is specifically designed for astronomy observations in terrestrial and spatial fields. The special feature of this material comes from its thermal expansion coefficient which is almost nil and homogeneous in all directions. So, Zerodur[®] offers a very high thermal stability.[26] It is a non-porous glass ceramic composed of 78% of nanocrystals with a quartz structure trapped in residual vitreous phase. Crystals have a negative thermal expansion coefficient and the glass has a positive thermal expansion coefficient which offset, giving to Zerodur[®], its weak thermal expansion coefficient. Zerodur[®] is composed of 57.2% of silica (SiO₂), 25.3% of aluminium oxide (Al₂O₃), 6.5% of phosphorous pentoxide (P₂O₅), 3.4% of lithium oxide (Li₂O), 2.5% of titanium dioxide (TiO₂), 1.8% of zirconium dioxide (ZrO₂), 1% of magnesium oxide (MgO), 0.5% of arsenic trioxide (As₂O₃) and 0.2% of sodium oxide (NaO). Small changes can occur in coefficient of thermal expansion (CTE) with the annealing treatment and can affect the geometric accuracy and stability of the high-precision optical assemblies. So, it is interesting to note that for Zerodur[®], there are three temperature ranges where different behaviours of CTE are observed [26]:

- (1) When $T > 700$ °C (ceramising range).
- (2) Between 320 °C $< T < 700$ °C steady state.
- (3) Between 130 °C $< T < 320$ °C (superior relaxation range).
- (4) Between 40 °C $< T < 130$ °C steady state.
- (5) Between -70 °C $< T < 40$ °C (inferior relaxation range).

For temperature above 700 °C, the material ceramising continues in irreversible way, changes in material properties appear and the CTE is irreversibly changed. Figure 4 relates limits and temperature influence on dimensional stability for samples

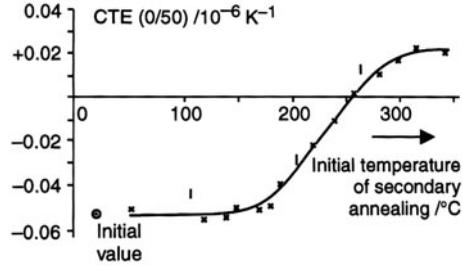


Figure 4. Variation of the CTE (0 °C; 50 °C) – first cooling at 0.1 K/min – as a function of the initial temperature of a secondary cooling in open air to room temperature.[26]

initially annealed at different temperatures and cooled at room temperature. Up to annealing temperature about 130 °C, there is no modification in CTE. Above this temperature, CTE increases to a specific constant value, in order to not degrade the thermal expansion parameter (CTE), the cooling speed has to be chosen between 1 and 6 °C/h (cooling procedure during Zerodur® manufacturing).[26]

3.2. Wetting tests and X-ray spectroscopy

To characterize samples surfaces, wetting tests and XPS were performed on fused silica glass and Zerodur® surfaces of two kinds: never bonded surfaces and bonded then debonded surfaces (called, respectively, in the following: non-bonded and debonded surfaces). In order to determine the surface energy of the both surfaces to adhere, wetting tests are performed. The angle of contact was determined by approximating the drop profile using the software Drop Shape Analysis V.1.80. Results summarized in Table 1 shown that surface energy is greater for Zerodur® samples. So, this material seems to be a better candidate for direct bonding. Indeed, the bonding will obviously be easier to perform when the surface energy of adhesion is high and furthermore when roughness is low.[9,12] The difference in surface energy between non-bonded and debonded surfaces was found to be approximately 20%. The results of the XPS tests presented in Figure 5 show that the spectra of debonded and non-bonded surfaces were identical a priori. The slight differences between fused silica and Zerodur® are attributable to the differences in the chemical composition of the two materials: the silicon atoms do not have the same environment in both cases. Surface wettability and XPS results confirm the reversibility – with damaging – of the process.[27] Indeed, one characteristic of the direct bonding process when no thermal treatments are applied is the reversibility of the bonding.[28]

Table 1. Surface energy for fused silica glasses and Zerodur® glasses.

Material	γ_s (mJ/m ²) surface energy	γ_s^d (mJ/m ²) dispersive component of surface energy	γ_s^p (mJ/m ²) polar component of surface energy
Zerodur®	51.59	27.69	23.91
Fused silica glasses	48.57	29.49	19.19
Debond Zerodur®	40.43	23.96	16.48
Debond fused silica glasses	41.52	26.11	15.41

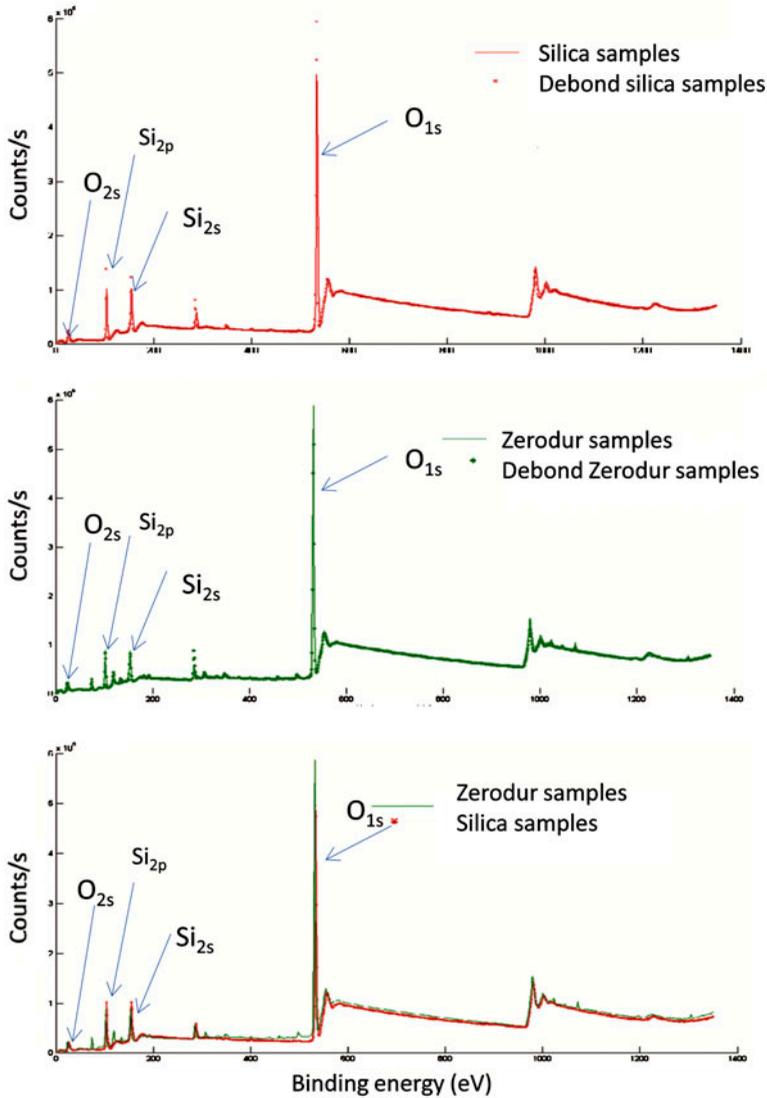


Figure 5. XPS spectra for silicon glasses and Zerodur[®] glasses.

3.3. Mechanical experiments

The objective of this part is to find the best candidate for direct bonding process between fused silica glasses and Zerodur[®] and to find the optimal parameters of the Winlight Optics Company process to increase mechanical strength of bonded assemblies. Therefore, we study the influence of some process parameters – roughness, relative air humidity during room temperature bonding, annealing time and temperature – on the mechanical strength of an elementary mechanical structure using a double shear test procedure and a cleavage test by comparing the both materials. Double shear test specimen was chosen for two main reasons: cylinder assemblies are classically used in some Winlight Optics Company interferometer, and it is easier to manufacture a large

number of small cylinders (150×3 cylinders) in glass with low roughness on each faces. The second test – the cleavage configuration – is the reference test for Winlight Optics Company and French National Centre for Spatial Studies (CNES). With these two mechanical tests, the influence of the surfaces treatment process on two failure modes (I & II) is observed.

3.3.1. Double shear tests

The first mechanical test has been set up on a large number (150) of samples in double shear configuration based on cylinder shape. With this configuration, we obtain a pure shear stress state and misalignments are controlled with a good adjustment between steel experimental device and sample geometry. The tools were beforehand mounted on a tensile testing machine. Samples are constituted with two cylinders of 5 mm thickness and 10 mm diameter, and one cylinder of 5 mm thickness and 15 mm diameter bonded together as shown in Figure 6. Materials used are fused silica glasses and Zerodur[®] glasses. The sample is inserted into the upper part of the tool as shown in Figure 6, and then fixed to the lower part using a flange. The loading rate of the test is fixed at 1 mm/min. We measured the ultimate force necessary to release the interfaces and compared results under different conditions. We assume that, in this case, the ultimate force is proportional to the bonding energy due to the brittle behaviour of molecular bonding. Table 2 shows the 150 experiments made (five samples per test).

3.3.2. Cleavage tests

The second mechanical test has been set up on five samples in classic cleavage configuration to validate the choice of the optimal parameters. Samples are constituted with two blades of 10 mm of thickness, 40 mm of width and 40 mm of length direct bonded together. Each sample has been bonded with glue on aluminium experimental device mounted in a tensile testing machine as related in Figure 7. A vertical load is applied, in the same way the loading rate of the test is fixed at 1 mm min^{-1} and the ultimate

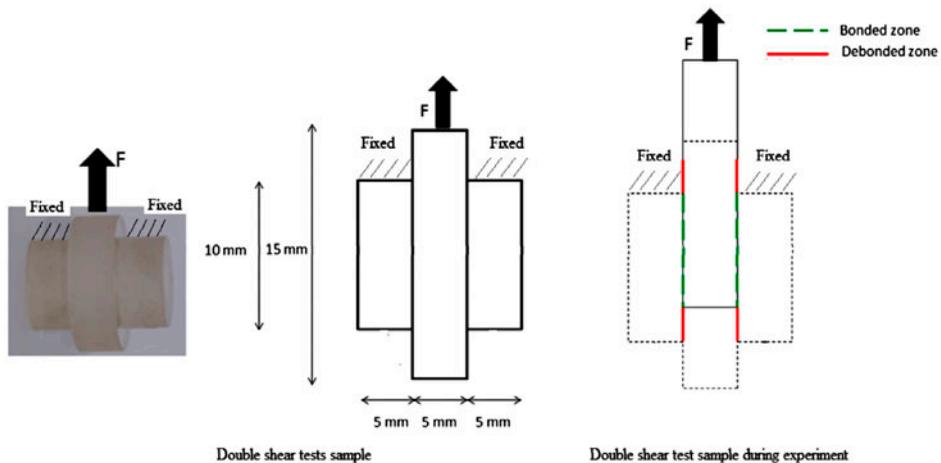


Figure 6. Sample and double shear experimental device.

Table 2. Summary of double shear tests performed (five samples per condition).

Material	Annealing temperature (°C)	Annealing time (h)	Roughness (nm RMS)	Humidity level (%)
Fused silica glass (Reference)	20	∞	0.4	55
Zerodur [®] (Reference)	20	∞	0.4	55
Fused silica glass	100, 200, 400, 700, 800, 900	15	0.4	55
Zerodur [®]	100, 150, 200, 400, 550	15	0.4	55
Fused silica glass	200	1, 15, 35, 120	0.4	55
Fused silica glass	200	120	0.2, 0.4, 0.6, 1	55
Zerodur [®]	130	120	0.2, 0.6, 1	55
Fused silica glass	X	X	0.4	32, 55, 82
Zerodur [®]	X	X	0.4	32, 55, 82

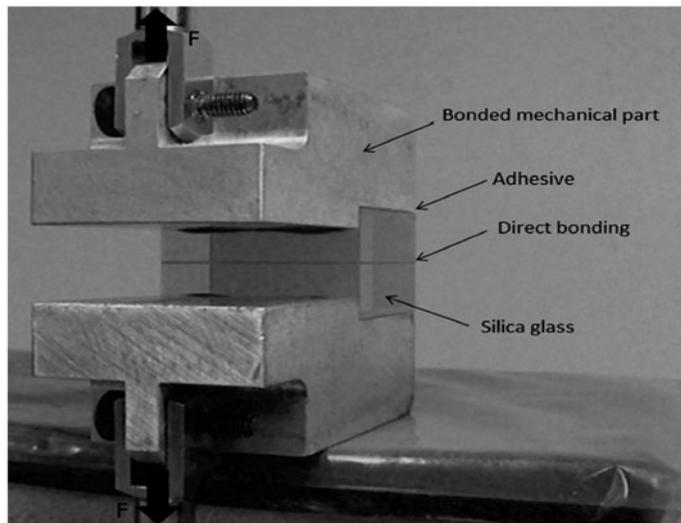


Figure 7. Sample and cleavage experimental device.

force is measured. With a finite element model considering an average stress value on a local zone, the maximal stress value is identified. Between each experiment on the same sample, surfaces are cleaned and re-adhered (without being polished anew).

4. Results and discussion

According to the adhesion mechanism presented in Section 2, parameters such as roughness, humidity level during room temperature bonding and annealing parameters are crucial for bonding. Indeed, bonding at room temperature is directly attached to the roughness of samples and to the presence of water cluster at the interface and therefore

to the rate of air humidity during room temperature bonding. Moreover, samples undergo a thermal treatment; the nature of bonds responsible for adhesion, therefore, depends on the temperature range and the kinetics of the reactions triggered at the interface. Thus, in this section, we study the influence of the previous process parameters: the annealing temperature and time, the roughness combined with thermal treatment, and to finish the influence of the relative air humidity during the room temperature bonding as related in Table 2. With Zerodur[®] samples, we had chosen to work at temperatures below 550 °C in order to preserve the thermal expansion coefficient.

4.1. Influence of annealing temperature

Firstly, we have studied the influence of the annealing temperature. Results, presented in Figures 8 and 9, show that the mechanical resistance increases non-linearly with annealing temperature. This can be explained by the chemical mechanism of direct bonding. Indeed, when temperature is below 200 °C, we have seen that silicon surfaces are contacted via water clusters. When temperature is lower than 110 °C, chemical reaction at the bonded interface are the same than during room temperature bonding (i.e. formation of hydrogen bonds between molecules of water and surfaces).

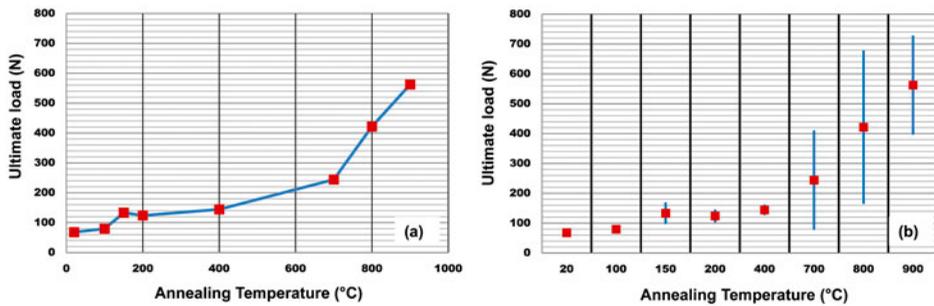


Figure 8. Annealing temperature influence on fused silica glass tested in double shear configuration: average values (a) and standard deviations (b) of measured ultimate loads.

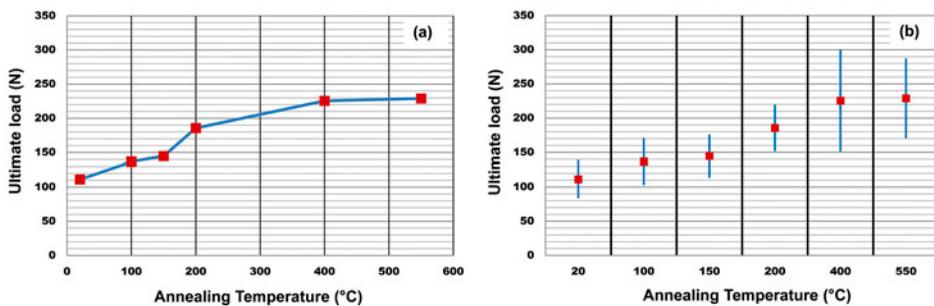


Figure 9. Annealing temperature influence on Zerodur[®] glass tested in double shear configuration: average values (a) and standard deviations (b) of measured ultimate loads.

The bonding energy is governed by the number of silanol group bonded with water.[14,29] Between 110 and 150 °C, the bonding energy increases due to the beginning of the releasing of molecular water. When temperature increases, there is formation of tetramers of water, surfaces are now directly contacted via double hydrogen bonds and the surfaces get closer. During this range of temperature, the bonding energy is limited by the area of contacted zone, the bonding energy slightly increases with the increasing of the contacted zone (Figure 10). Then, when temperature is up to 700 °C, there is polymerization of covalent siloxane (Si–O–Si) bonds.[13,14,30]

For thermal treatment at 900 °C, we do not observe a release along the interfaces but a fracture inside material of the bonded samples. It seems that the material is fused at the interface. Interface has the same behaviour that the pure crystal configuration and we can suppose that a single bloc of fused silica glass or Zerodur® is obtained as related on Figure 11. Moreover, the results confirm wetting tests results: the mechanical resistance is higher for Zerodur® samples.

4.2. Influence of annealing time

Then, we have studied the influence of annealing time. Figures 12–14 show an increase of the mechanical strength and the appearance of a plateau for the long annealing time (Figure 16). For low temperature, the bonding energy only depends on the number of hydrogen bonds. When temperature increases, the different reactions at the interface

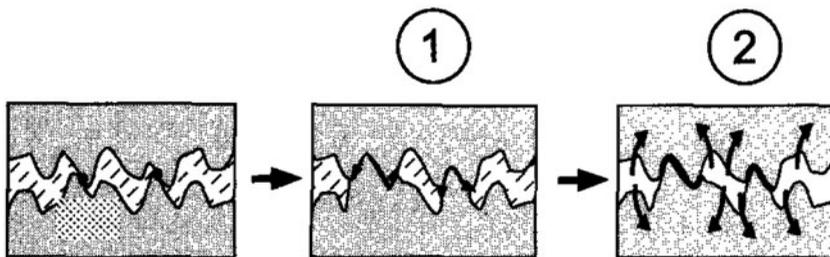


Figure 10. Evolution of the contacted zone (from [23]).

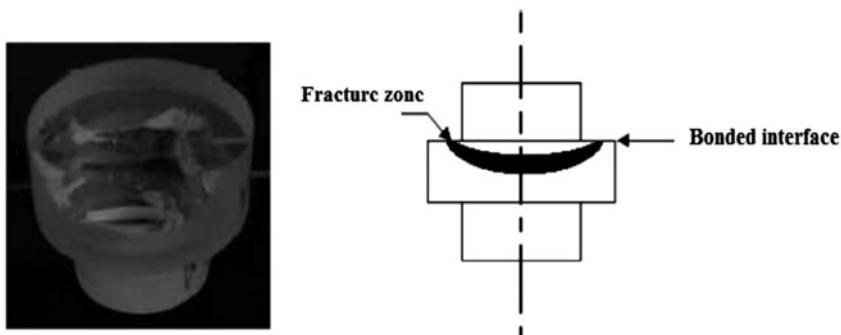


Figure 11. Fracture of fused silica glass sample annealed at 900 °C during 120 h.

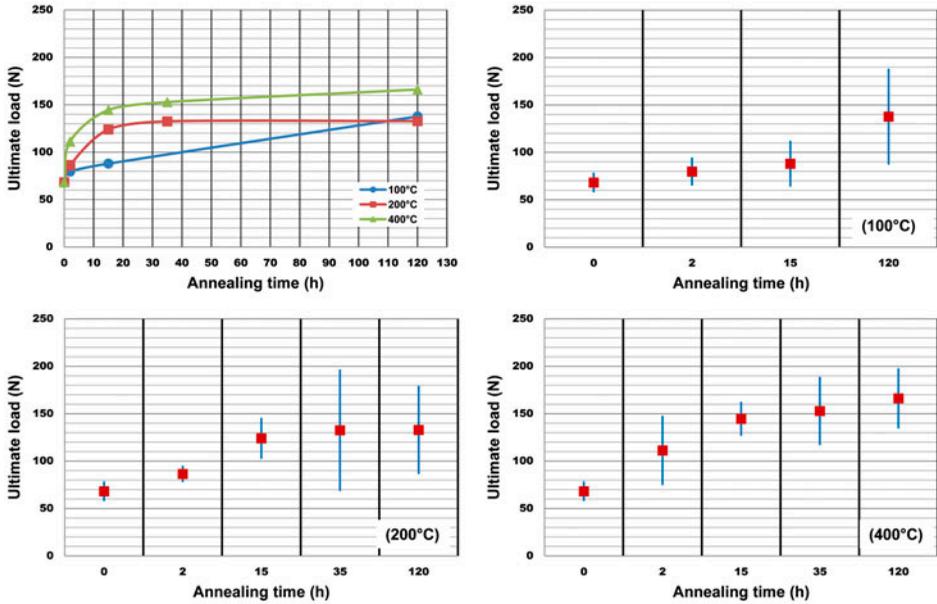


Figure 12. Annealing time influence at low temperatures on fused silica glass tested in double shear configuration: average values and standard deviations of measured ultimate loads.

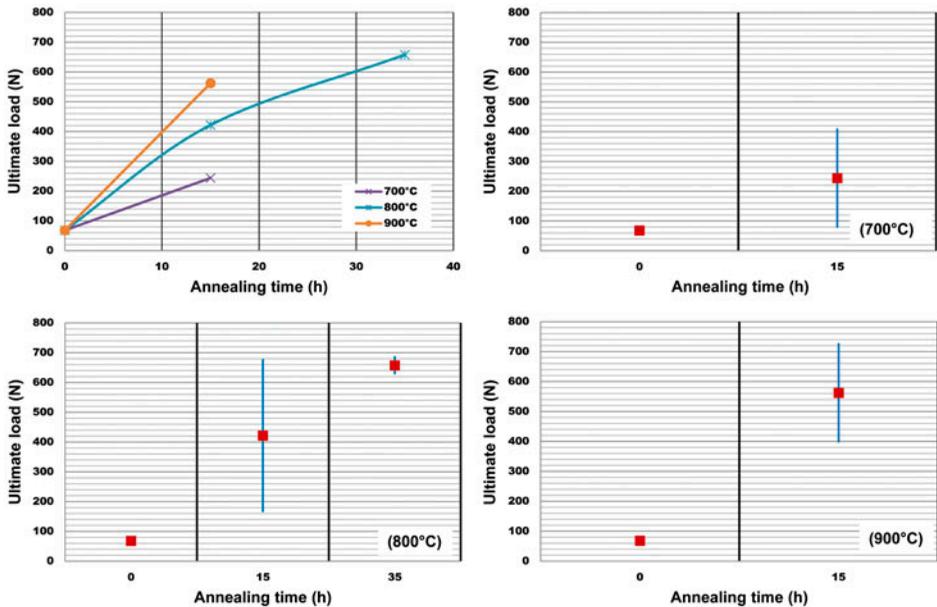


Figure 13. Annealing time influence at high temperatures on fused silica glass tested in double shear configuration: average values and standard deviations of measured ultimate loads.

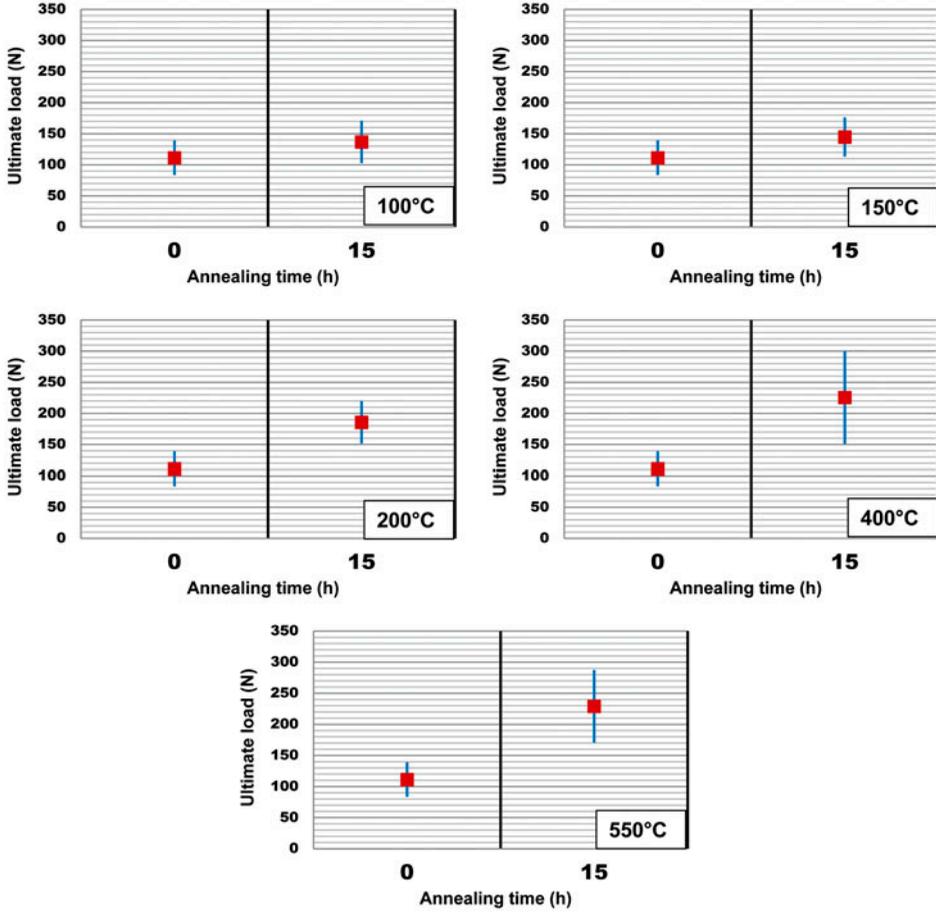
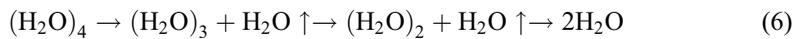


Figure 14. Annealing time influence at different temperatures on Zerodur® glass tested in double shear configuration: average values and standard deviations of measured ultimate loads.

can be modelled using a kinetic model.[13,14] When temperature increases, there are changes in the nature of bonds at the interface, at the passage between phase I and phase II, hydrogen bonds between clusters of water break to form tetramers of water, and a double hydrogen bond appears between surfaces. Thus, between 200 and 700 °C, the following reaction is triggered:



Up to 700 °C:



where k_1 and k_2 are the rate constants of reaction.

By supposing reaction (5), total we have:

$$\frac{d[\text{SiOH} : \text{SiOH}]}{dt} = k_1[\text{SiOH} : (\text{OH}_2)_2 : (\text{OH}_2)_2 : \text{SiOH}] = k_1(n_0 - [\text{SiOH} : \text{SiOH}]) \quad (9)$$

with:

$$k_1 = \frac{1}{\tau} e^{\left(-\frac{E_{a1}}{kT}\right)} \quad (10)$$

where $[\text{SiOH} : \text{SiOH}]$ represent the number of double hydrogen bonds between Si–OH of the both surfaces, $\frac{d[\text{SiOH} : \text{SiOH}]}{dt}$ represent the reaction rate of the double hydrogen formation. K is the Boltzmann constant, E_{a1} is the activation energy of the reaction, n_0 the number of adhesion site, τ the time characteristic constant and T absolute temperature. At room temperature, the activation energy is around 50 meV, the energy of a hydrogen bond.[14] The solution to Equation (9) is:

$$[\text{SiOH} : \text{SiOH}](t) = n_0(1 - e^{(-k_1 t)}) \quad (11)$$

where t is the time. We make the assumption that the gain of surfaces energy is proportional to the number of double hydrogen bonds Si–OH: Si–OH and we obtain the following expression for the bonding energy W (Figure 15):

$$W = (W_{\text{II}} - W_{\text{I}})(1 - e^{(-k_1 t)}) + W_{\text{I}} \quad (12)$$

with W_{I} and W_{II} maximum specific surface energy on phase I and phase II, respectively.

Figure 15 presents the evolution of the kinetic model for different annealing time. Thus, in this kinetic model, the evolution of the bonding energy with annealing parameters is exponential. The evolution of the ultimate force with thermal treatment parameters on Figure 16 also shows the same kind of exponential evolution. Moreover, we have made the assumption that the bonding energy and the ultimate force are proportional, the use of the kinetic model to explain the evolution of the mechanical

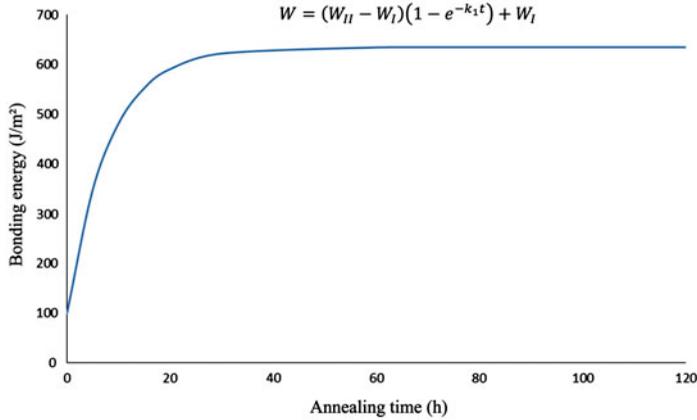


Figure 15. Evolution of the bonding energy modelled using Equation (12) with annealing temperature and time.

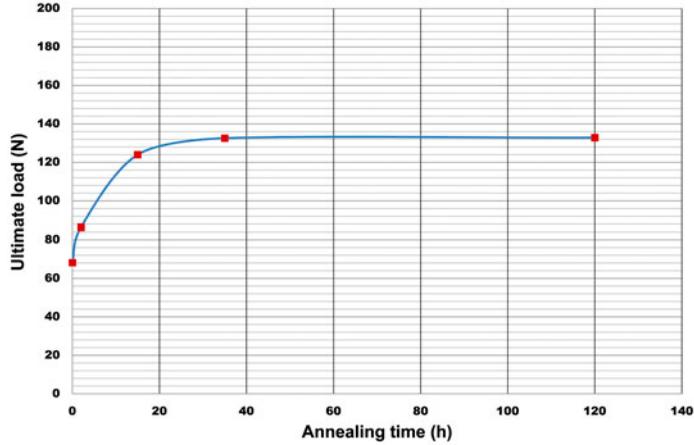


Figure 16. Annealing time influence at 200 °C on fused silica glass tested in double shear configuration: confrontation with a kinetic model of bonding energy.

resistance with annealing time is validated. Furthermore, results also show a kind of equivalence between long-time annealing at low temperature and short-time annealing at high temperature as shown in Figures 12–14.

4.3. Influence of roughness

The results, in Figures 17 and 18, show the influence of roughness combined with thermal treatment – 120 h at 200 °C and 120 h at 130 °C, respectively, for fused silica glass and Zerodur[®] samples – on the ultimate load measured. The mechanical strength increases until a roughness of 0.6 nm RMS then decreases. Although those results do not agree with literature in other context [1,8,9,12] and suggest that – for the Winlight Optics Company surface preparation – a higher roughness is better. A first explanation of those results could be the friction of surfaces during double shear tests experiments; moreover, low roughness leads to the appearance of residual stresses on surfaces during polishing process. Furthermore, between 150 and 450 °C, the bonding energy – thus

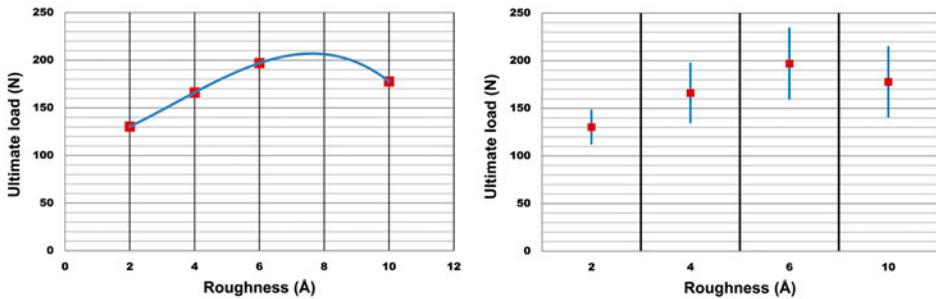


Figure 17. Roughness influence combined with thermal treatment (200 °C, 120 h) on fused silica glass tested in double shear configuration: average values and standard deviations of measured ultimate loads.

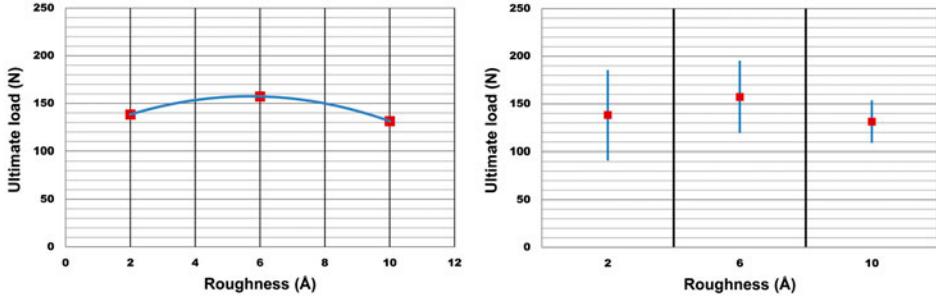


Figure 18. Roughness influence combined with thermal treatment (130 °C, 120 h) on Zerodur[®] glass tested in double shear configuration: average values and standard deviations of measured ultimate loads.

the mechanical strength – is limited by the contacted area (Figure 10) and, in case of high roughness, contacted zones are higher in explaining the results. Adhesion also depends on the height of asperities, the radius of curvature, the wavelength, the real contact area and the local stiffness of contact surfaces.[31–33]

Thus, the existence of a maximum roughness of 0,6 nm RMS suggests that a medium roughness, compromised between all the previous parameters, is better for the Winlight Optics Company process. Moreover, results of Zerodur[®] samples are worse than fused silica samples. This could be explained by the temperature of thermal treatment. Indeed, the curve on Figure 4 shows a inflection point at 130 °C, characterizing surely a change in the microstructure leading to a sharp decrease of mechanical properties.

4.4. Influence of humidity level

We have also studied the influence of relative air humidity during the room temperature contacting step. Results related in Figures 19 and 20 show that the mechanical strength presents a maximum of 32% for fused silica glass samples and 55% for Zerodur[®] samples. Those results can be explained by the size of water clusters. Indeed, we have shown that there is formation of clusters of water at the interface. Thus, if there is more molecular water at the interface, clusters would be higher. And then, distance between

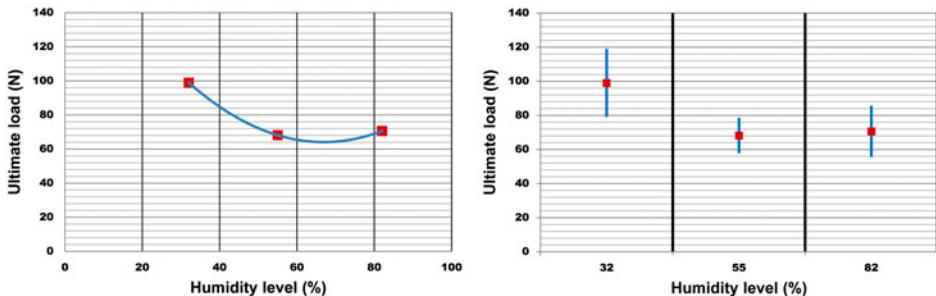


Figure 19. Humidity level influence on fused silica glass tested in double shear configuration: average values and standard deviations of measured ultimate loads.

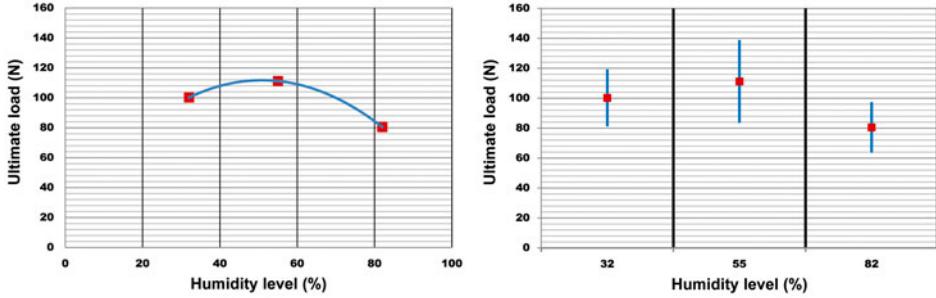


Figure 20. Humidity level influence on Zerodur[®] glass tested in double shear configuration: average values and standard deviations of measured ultimate loads.

the two surfaces would be higher resulting by a decrease of the bonding energy. Moreover, when there is too much water, we could lead to think that there is a phenomenon of glass damaging.

4.5. Dispersion of the results

All the results show lots of dispersion, which can be explained by the brittle behaviour of the direct bonding and heavy sensitivity of the mechanical tests to the presence of defects along the interface. Results strongly depend of the infinitesimal defects present in the bonded interface and depend of their location. However, due to the high number of samples, the tests coupled with a statistical study allow us to identify highly interesting trends in the point of view of the understanding of the interface behaviour.

4.6. Optimal parameters

Despite dispersion of the double shear test, results give a good idea of optimal parameters to apply in order to increase the mechanical strength of bonded interfaces. In order to not degrade optical properties of materials, annealing temperature cannot be higher than 200 °C for both types of samples. As shown in Section 2, there is a kind of equivalence between annealing time and temperature, thus in order to have stronger bonds at 200 °C, thermal treatment has to be long. Based on all the double shear tests results, the chosen parameters are 200 °C during 120 h for both fused silica and Zerodur[®] samples. We also have chosen 200 °C for Zerodur[®] samples due to sharp decrease of mechanical properties at 130 °C. Then, roughness chosen is 0.4 nm RMS for all the samples – compromise between literature and experimental results, 32% of humidity for Zerodur[®] samples and 55% of humidity for fused silica samples.

5. Validation of optimal parameters

In the last section, optimal parameters have been found. In this section, to validate this choice, those parameters are compared to the initial conditions – no thermal treatment applied on samples – using cleavage test which is the Winlight Optics Company validation test. Results presented on Figures 21 and 22 show an increase of the mechanical resistance with optimal parameters, thus an improvement with the new parameters.

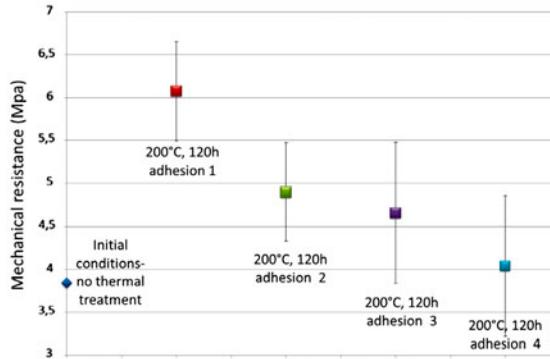


Figure 21. Comparison between initial parameters (no thermal treatment) and optimal parameters (200 °C during 120 h) in cleavage test configuration: for successive re-adhesions of fused silica samples.

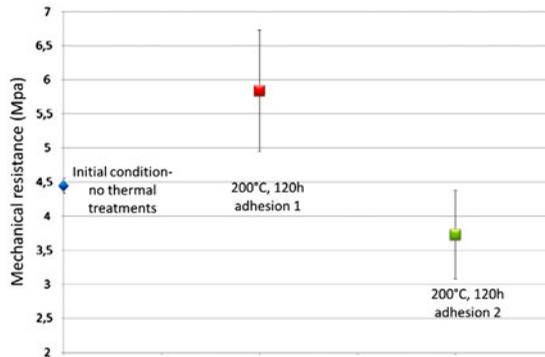


Figure 22. Comparison between initial parameters (no thermal treatment) and optimal parameters (200 °C during 120 h) in cleavage test configuration: for successive re-adhesions of Zerodur® samples.

Moreover, mechanical resistance slightly linearly decreased with successive adhesion/cleavage test/re-adhesion highlighting a damaging phenomenon of the bonded interfaces with successive re-adhesion made in the experiments. This phenomenon was also observed on the wetting tests results. Results for Zerodur® samples are a little worse than results for fused silica sample, because, samples have already undergone other treatment before those experiments. The results confirm the use of optimal parameters found to increase the mechanical resistance of samples.

6. Conclusion

The analysis of fused silica glass and Zerodur® surfaces confirm the reversibility (with damaging) of the process when no thermal treatments are applied i.e. when no covalent bonds are responsible for adhesion. Moreover, Zerodur® samples seem to be a better candidate for direct bonding. This result is confirmed by double shear tests results. Despite the dispersion of the experiments, optimal parameters of the process have been

found, annealing treatment at 200 °C during 120 h for fused silica glass samples with a 0.4 nm RMS roughness and 55% of humidity, and 200 °C during 120 h with a 0.4 nm RMS roughness and 32% of humidity for Zerodur® samples. Moreover, a kind of equivalence between a long thermal treatment at low temperature and a short thermal treatment at high temperature appears. Cleavage tests performed confirm the choice of the optimal parameters and highlight a damaging phenomenon of bonded interfaces with successive re-adhesion. With these optimal parameters, the mechanical strength is twice as much as the strength obtained with the initial parameters. We found a way to increase the mechanical strength of direct bonded interfaces for the Winlight Optics procedure.

The final aim of this work consists in the development of an interface mechanical model of the direct bonding. The implemented law relates the bonding energy, the mechanical critical strain energy, the process parameters and the chemical kinetic in a multi-physic and multi-scale formalism. Soon, a wedge test will be set up to measure the bonding energy vs. process parameters in order to identify the law of the direct bonding model and to implement it in a finite elements model. For the future, new investigations will be lead on to find new original process in order to improve the mechanical strength and to develop simulation of complex spatial optical assemblies.

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