Mechanical characterisation of direct bonding

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Résumé :

Le collage par adhérence moléculaire est un collage qui ne nécessite pas l'utilisation de matériau adhésif. Ce procédé est déjà utilisé dans le domaine de l’optique terrestre et spatiale. Cependant, même si un prototype a passé avec succès les conditions du spatial, il reste à quantifier les limites de la tenue mécanique tout en trouvant des solutions pour les améliorer. L'utilisation de traitement thermique semble être la solution la plus adaptée, ainsi, afin d'étudier l'influence de la température et du temps de recuit et de trouver les paramètres optimaux du process, des essais de double cisaillement ont été réalisés. Des essais de clivage ont ensuite été mis en œuvre afin de valider les paramètres optimaux obtenus précédemment. Enfin, des essais de clivages au coin ont été effectués afin de déterminer l'énergie de collage des interfaces adhérentes. Les résultats montrent une évolution de la résistance mécanique avec le temps et la température. De plus une d'équivalence temps-température semble apparaître, ouvrant la voie d’une solution afin d'améliorer les propriétés mécaniques des interfaces adhérentes sans dégager les propriétés optiques des verres de silice utilisés.

Abstract :

Direct bonding does not need any adhesive or additional material. This process is increasingly used in terrestrial and spatial optical manufacturing. However, even if a prototype already passed with success spatial environment, mechanical resistance of assemblies needs to be quantified and improved. The use of thermal treatment seems to be the best solution, thus, in order to study the influence of annealing time and temperature and to find optimum parameters of the process, double shear tests were performed. Cleavage test were made to validate optimum parameters found. Then, bonding energy of the bonded interfaces were measured using wedge tests. Results show an evolution of the mechanical resistance with temperature and time. Moreover, a kind of time-temperature is noticed giving a way to improve resistance of assemblies without losing optical properties of fused silica glasses used.

Mots clefs : Direct bonding, Double shear tests, Wedge tests, silica glass

1 Introduction

Direct bonding consists in joining two surfaces without the use of any adhesives or additional material \cite{1}, \cite{2}. When two flats, well-polished and clean silica glasses surfaces are contacted at room temperature, hydrogen bonds\textsuperscript{1} appears and the two surfaces bonds or adhere \cite{3},\cite{4}, \cite{5}. In case of no thermal treatment applied, the bonding is reversible \cite{5}.

Direct bonding process is used in the manufacturing of terrestrial optical system such as interferometers or slicers \cite{6} and increasingly in spatial instrument applications. The use of no mechanical part, no adhesive material combined with no risks of contamination associated with degassing are advantages in spatial context where mechanical and thermal constraints involved (thermal fatigue, vibrations, etc.)

\textsuperscript{1}. bond between a hydrogen atom and a negative polarized atom such as oxygen atom
are very different from those encountered on Earth. This kind of process have already passed with success the mechanical and thermal environment of space [7] however, this is necessary to quantify the bonding strength and to improve the mechanical performance of adhesive bonds without degrading optical performances of the silica glasses used.

In this context, double shear tests were performed on silica samples to study the influence of some process parameters such as annealing temperature and time, roughness, etc. and to find optimal parameters to strengthen the bond across the interface without losing the optical properties of glass. Then, cleavage tests were made to validate optimal parameters found. Finally, wedge test were performed to measure the bonding energy of the bonded interfaces.

The direct bonding process is presented in section 1, doubles shear test results are shown in section 3. The validation of optimum parameters are presented in section 4 and wedge test results in section 5.

2 Evolution of bonded interfaces with temperature

On way to improve the mechanical resistance of bonded interface is to change the nature of bonds by applying thermal treatment (Figure 1).

Between 25 °C and 200 °C, surfaces are contacted via clusters of waters [4], [3] :

- 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. The bonding energy is governed by the number of silanols group bonded with water [8], [3].

- Between 110 °C and 150 °C the bonding energy increases due to the beginning of the releasing of water molecules. When temperature increases, there is formation of tetramers of water molecules, 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 increase with the increasing of the contacted zone (figure 1).

When temperature is between 200 °C and 700 °C, there is formation of cyclic tetramers of water; surfaces are directly contacted via double hydrogen bond [9]. But until the temperature is under 450 °C the bonding energy is still limited by the area of contacted zone. Up to 450 °C, the bonding energy starts to increase. Up to 700 °C, the polymerization reaction is triggered, resulting in the formation of covalent siloxane bonds. Bonding energy greatly increases [4], [3].

![Figure 1 - Evolution of the nature of bonds at the interface during thermal treatment](image)

**Figure 1** – Evolution of the nature of bonds at the interface during thermal treatment
3 Double shear tests

Double shear test were performed to study the influence of the annealing temperature and time on the mechanical resistance in order to improve mechanical resistance of bonded interfaces by finding optimal parameters of the process. Table 1 resumes the experiments performed.

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.

<table>
<thead>
<tr>
<th>Material</th>
<th>Annealing temperature</th>
<th>Annealing time (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silica</td>
<td>100, 200, 400, 700, 800, 900</td>
<td>15</td>
</tr>
<tr>
<td>Silica</td>
<td>200</td>
<td>1, 15, 35, 120</td>
</tr>
<tr>
<td>Silica</td>
<td>200</td>
<td>120</td>
</tr>
</tbody>
</table>

Table 1 – Resume of tests performed

In Figure 2, results show that the mechanical resistance increase non-linearly with annealing temperature and time. This can be explained by the chemical mechanism of direct bonding as explained in section 2.

A thermal treatment at high temperature seems to be the best solution to improve mechanical resistance of bonded interfaces. However, samples cannot be annealed at high temperature to not degrade optical properties of the material.

Results also show a kind of equivalence between temperature and time treatment. Thus, optimal parameters of the process seems to be a thermal treatment at 200°C during 120h.

Results show a lot of dispersion which can be explained by the double shear test configuration. Indeed, the two small cylinders have chamfers and during the mechanical test an "infinite" strain appears at the bonded interface. If this interface has the slightest defect, the crack propagates instantaneously and the interface broke. This is an unstable test from the mechanical point of view. 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 comprehension of the interface behaviour.

![Figure 2 - Evolution of the nature of bonds at the interface during thermal treatment](image-url)
4  Cleavage tests : validation of optimum parameters

Cleavage tests were made to validate optimum parameters found by comparing samples with no thermal treatment and samples annealed at 200 °C during 120h. All the tests were performed on the same sample. Between each tests, surfaces have been cleaned and re-adhered.

Cleavage test sample is constituted with two blades of 10 mm of thickness, 40 mm of width and 40 mm of length bonded together.

Results presented on Figure 3 show an increase of the mechanical resistance with optimal parameters, thus an improvement with the new parameters. Moreover, mechanical resistance slightly linearly decrease with successive adhesion/cleavage test/re-adhesion highlighting a damaging of the bonded interfaces with the four successes re-adhesion made in our experiments.

\[ G_{1c} = W_{\text{adhesion}} = \gamma_1 + \gamma_2 - \gamma_{12} \]  

Figure 3 – Cleavage test: comparison between initial parameters (no thermal treatment) and optimal parameters (200 °C during 120h) for four successive re-adhesion

5  Wedge tests

One of the main parameters generally used to characterize adhesion is the bonding energy [10], which can be connected to the critical strain energy release rate [11]. The most popular method to measure the bonding energy is the crack propagation method or wedge test [12].

A razor blade is inserted at the interface, a crack will appear along the bonded interface until the establishment of an equilibrium between the elasticity of blades and the bonds responsible for the adhesion - hydrogen bonds in our case.

The length of the crack is measured using interference fringe due to the small thickness of air trapped at the open interface and an optical system.

At the equilibrium, the critical strain energy release rate is equal to the work of adhesion [1] :

\[ G_{1c} = W_{\text{adhesion}} = \gamma_1 + \gamma_2 - \gamma_{12} \]  

With \( \gamma_1 \) and \( \gamma_2 \) the surface energies of the two surfaces and \( \gamma_{12} \) the bonding energy.

The critical strain energy release rate can be approximately related to the length L using the following equation [13] :

4
With $E$ the Young modulus, $t$ the blade thickness, $y$ the razor blade thickness and $L$ the length of the crack.

Samples are constituted with two blades of 500 $\mu$m of thickness, 10 mm of width and 80 mm of length. One tip of the blades is built-in the device and a razor blade (20 $\mu$m of thickness) is inserted in the chamfer of the other tip.

Results presented in figure 4 show an increase in the critical strain energy release rate and a diminution of the crack length with annealing time and temperature. Then, when the time - during which the razor blade is inserted - is twice the re-adhesion is lower due to the high amount of pollution at the interface. For example, for samples with no thermal treatment, when time of insertion is twice, the area of rebonded zone is divided by 8. Thus, the re-adhesion is possible in case of presence of hydrogen bonds and depends of the pollution at the interface. The process is quasi reversible in case of hydrogen bonds.

![Figure 4 - Evolution of the critical strain energy release rate and crack length with annealing time and temperature](image)

6 Conclusions

Results of double shear test and wedge test shown an increase of the mechanical resistance and the bonding energy with annealing temperature and time. Moreover, the process is quasi reversible when no covalent bonds are present at the interface.

Despite the dispersion, optimal parameters - annealing treatment during 120h at 200$^\circ$C - have been found thanks to double shear tests and confirmed using cleavage tests.

A kind of time-temperature equivalence is also observed as a phenomenon of sample damaging with successive re-adhesion.

Other experiments need to be made to study the influence of other parameters like roughness or humidity level during adhesion at room temperature. Then a law between the critical strain energy release rate and chemical properties (annealing parameters, roughness, etc.) of the bonded interface has been developed similar to that proposed in [10]. A finite element model has been implemented using that law and applied on real assemblies.
Références


[12] Cognard, J.Y. 1986 The mechanics of the wedge test The journal of Adhesion 20, 1 1-13