Optical contacting of low-expansion materials

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ABSTRACT

We report on optical contacting or "direct bonding" of glass to glass for optical and precision engineering applications. Fused silica (SiO2) and ultra-low-expansion (ULE) glass materials with low and extremely low coefficients of thermal expansion, respectively, were investigated. Large glass wafers of up to 150 mm diameter and about 1.5 mm thickness were bonded to each other and to plane glass substrates of up to 20 mm thickness. Successful bonding was achieved after extensive chemical cleaning and low pressure plasma surface activation, using a commercial wafer bonding equipment. High quality (optically transparent) bonds with a very low fraction of aerial defects were obtained at low bonding temperatures of about 250 °C, by applying compressive forces of several tens of kN in a high vacuum environment. Typically, only small unbound locations occurred at the rim, where insufficient pressure had been applied in the bonding process. Bonding strengths were estimated from destructive "razor-blade" testing of bonded wafer pairs, resulting in bond energies up to about 2 J/m². For surface activation, Nitrogen-plasma was tested in comparison to Oxygen-Plasma without significant differences. However, ULE wafers were found to bond much stronger than fused silica wafers under nominally identical bonding conditions. An exemplary "sandwich" structure was generated from ULE materials by bonding wafers from both sides to a core structure, obtained by perforating a massive plane plate with bore holes. This illustrates possible use in light-weight and stiff construction for high precision opto-mechanical applications.

Keywords: optical contacting, direct bonding, glass bonding, ultra-low expansion glass, fused silica

1. INTRODUCTION

Optical contacting is known in optics since more than 100 years for bonding of extremely flat, smooth and clean glass surfaces without auxiliary materials (like glues or solders), just by breathing and wiping (traditionally with a leather cloth) on it and finally pressing the surfaces together. The procedure, also known as "Ansprengen" in German optics literature, was long regarded as "black magic", strongly dependent on individual skills of the optician and barely reliable, i. e. not useful for broad applications. Systematic investigations on similar "hydrophilic" bonding of oxidized Si-wafers during the last two decades, however, have elucidated basic adhesion mechanisms involved in this type of bonding.
Under the term of "Direct Wafer Bonding", a refined technology has emerged for Si-wafer bonding in micro-electronics and MEMS applications, in which a low surface roughness (less than 1 nm root-mean-square (RMS)) and extremely flat (or otherwise conform) surfaces of the bonding partners play a decisive role[1]. Reliable bonding is achieved with extended cleaning and activation procedures before the final contacting step, which is often executed in a vacuum environment under compressive pressure at moderate temperature. Although the oxidized surfaces of Si-Wafers are chemically not very different from fused silica (SiO2 glass) surfaces, application of the technology to glass substrates for optical or precision mechanical applications are still rare[2-4]. This ignores the great potential benefits with respect to established glass assembly technologies, like adhesive bonding, soldering or –in part– even laser welding:

a) Full transparency (bonding layer is "invisible")
b) No uncontrolled creep/drift under mechanical load (bonding layer is "stiff")
c) No outgassing at elevated temperatures
d) No stress from melting/re-solidification processes or thermal mismatch (for identical materials)
e) Assembly of individual parts „accurate to gage blocks" (bonding layer is "infinitely thin")

Such bonding properties are clearly interesting for high precision set-ups and most useful for laser resonators, optical platforms, lithography tools, or imaging optics under harsh conditions. Furthermore, high dimensional stability can be

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achieved when low-thermal expansion materials are utilized for this bonding technology, like fused silica with a coefficient of thermal expansion (CTE) of about $50*10^{-8}$/K, or dedicated ultra-low ("zero") expansion materials like ULE glass (from Corning) or ZERODUR glass-ceramics (from Schott) with CTEs of about $2*10^{-8}$/K or even less in a narrow range around room temperature.
In the following, we report on "Direct Bonding" of fused silica and ULE (thin) glass wafers to corresponding massive (thick) substrates of identical material. As the sophisticated surface preparation and temper procedures originally developed for "Direct Wafer Bonding" of Si-wafers were used, the term "Direct Bonding" may in fact describe the process more exactly than "Optical Contacting".

2. SAMPLE SURFACE CHARACTERIZATION

Before bonding, sample surfaces were characterized with respect to flatness by Fizeau or Grazing-Incidence Interferometry and with respect to roughness by White-Light Interferometry, respectively. For the glass wafers, flatness measurements were done by the manufacturers with the wafers laid down on a plane support and flattened by gravity.

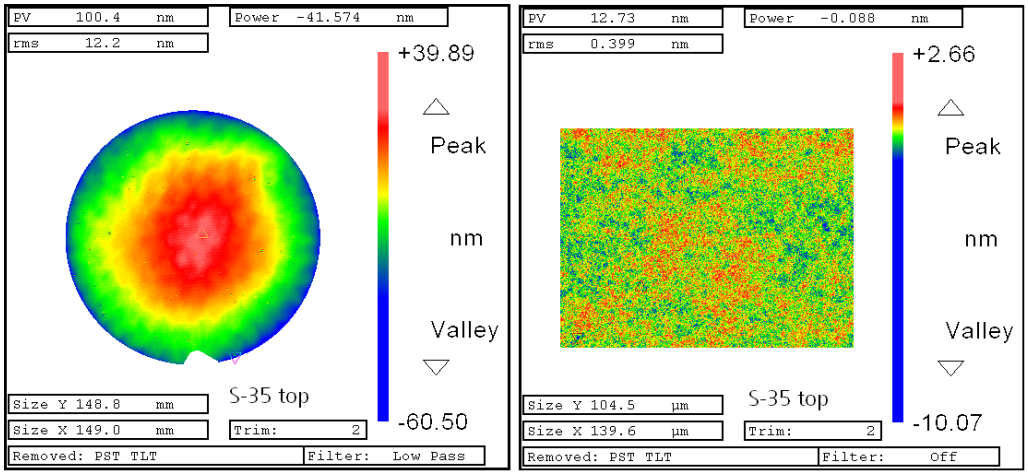

Figure 1: Surface flatness (left, ≈100 nm PV) and roughness (right, ≈0.4 nm RMS) of a massive SiO2 substrate

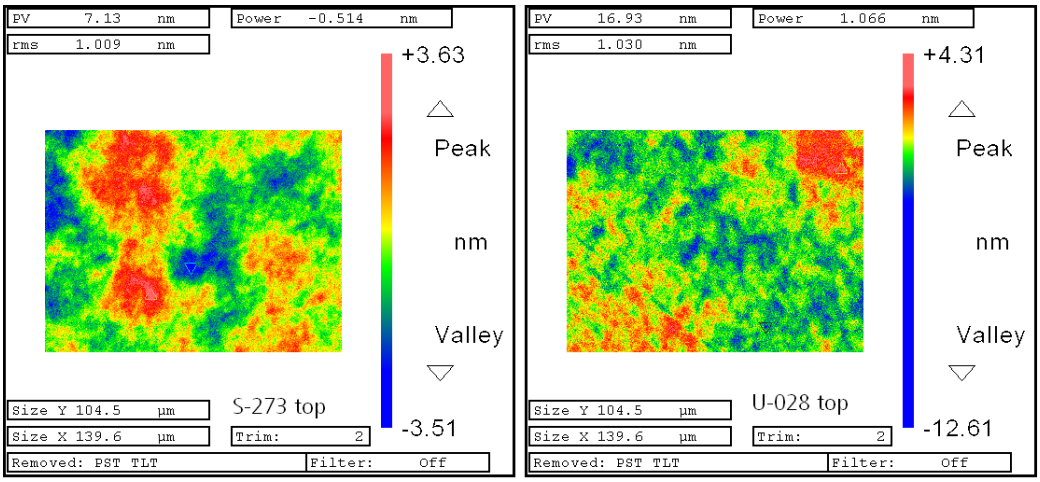

Figure 2: Surface roughnesses of a SiO2 wafer (left, ≈1.01 nm RMS) and an ULE wafer (right, ≈1.03 nm RMS)

For all glass wafers, flatness values (warp) of <15 µm peak-to-valley (PV) were reported by the manufacturers. These specifications were not verifiable, albeit not considered critical in view of the materials residual flexibility at 1.5 mm thickness, at Young's moduli of about 73 GPa and 68 GPa for fused silica and ULE, respectively.

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The flatnesses of the thick substrates were measured in our lab with a 300 mm ZYGO Veryfire interferometer, equipped with a reference flat of λ/30 at λ= 632.8 nm. The roughnesses of wafers and thick substrates were determined with a ZYGO New View 600S equipped with a Mirau objective at 50x magnification.
Exemplary results for a massive SiO2 substrate of diameter 150 mm and thickness ≈17 mm are displayed in Fig. 1. Surface flatnesses for the thick substrates were found to be <150 nm PV for SiO2 and <300 nm PV for ULE, respectively. Corresponding surface roughnesses were <0.6 nm RMS for SiO2 and <0.8 nm RMS for ULE, respectively. Surface roughnesses of the glass wafers were considerably poorer and found to be around 1.1 nm RMS for both materials. Exemplary results for wafers of diameter 150 mm and thickness 1.5 mm are displayed in Fig. 2. Since roughness values of <1 nm RMS are generally considered necessary from experiments with oxidized Si-wafers[1], the surface quality of the glass wafers is quite critical for direct bonding and may result in limited bonding strengths.

3. SAMPLE CLEANING & ACTIVATION

Upon receipt, all glass samples were passed through a proprietary, multistage ultra-sonic assisted bath cleaning process (CL-3002), containing organic and alkali surfactant stages and thorough rinsing in de-ionized (DI) water, to remove any residuals from the preceding lapping and polishing processes.
Immediately before bonding, another –wet chemicals as well as mega-sonic assisted– cleaning process was executed on a spinning stage in a commercial cleaner tool from EVG (Austria). Subsequently, the bonding sides of the glass parts were subjected to a short (low-pressure) plasma treatment in Oxygen or Nitrogen, rinsed again with DI water, dried on a spinning stage and finally contacted to each other. Alignment was done under normal atmosphere with thin separating spacers inserted at the rim, which were later removed in the bonding chamber after attaining vacuum.
This processing sequence reproducibly results in highly clean and hydrophilic glass surfaces, as demonstrated previously by measuring the wetting angles (with DI water) and the residual particle counts on fused silica wafers[4]. This was now also verified for ULE wafers. Fig. 3 shows the corresponding DI water wetting angles of two ULE wafers during various surface treatments, as determined with a commercial measurement tool, capable of resolving static and dynamic wetting angles down to about 3 deg.

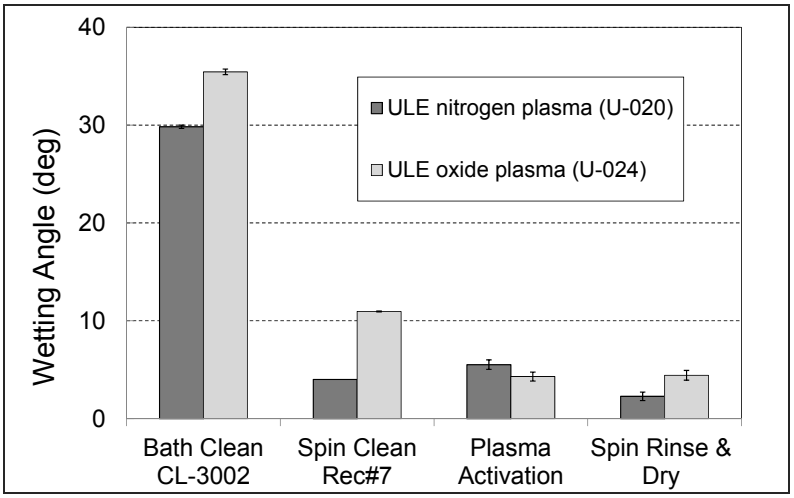

Figure 3: DI water wetting angles versus surface treatments for two ULE wafers

The initial bath cleaning process (CL-3002) –although found to be helpful for particle removal– is insufficient to create reasonably hydrophilic surfaces for a longer period. However, the chemical assisted spin cleaning, plasma activation and rinsing processes before bonding greatly improve the initially poor situation and result in highly hydrophilic surfaces with similar wetting angles of about 5 deg for ULE as for fused silica previously. Oxygen and Nitrogen low pressure plasma activation were tested alternatively, without discerning significant differences with respect to hydrophilic behavior. All handling and processing was performed in a class 10 to 100 clean-room environment.

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4. GLASS WAFER BONDING

Fused silica and ULE glass wafers were bonded at a vacuum of better than 10^{-4} mbar using the "wafer bow" feature of the bonding equipment, i.e. by applying some spring pressure in the center of the wafer stack before releasing the spacers, to ensure a strictly outward moving front of contacting. Subsequently, the wafer pair was compressed with forces of several tens of kN and heated up to temperatures around 250 °C, to extract water from the bonding area and create stable, permanent Si-O-Si bonds between the two surfaces. Typical bonding results for fused silica and ULE glass wafers are displayed in Figs. 4. Although particle cleanliness is a permanent issue, visual appearance in the interior is mostly excellent and free of defects. This does not hold for the outer rim, where –amongst others– the laser numbering of the wafers (if facing the bonding area, as seen at 6 o'clock to the left in Fig. 4) or the retraction of the spacers under the load of the "wafer bow", induces fringes or fine scratches (barely visible at 6 and 10 o'clock to the right in Fig. 4).

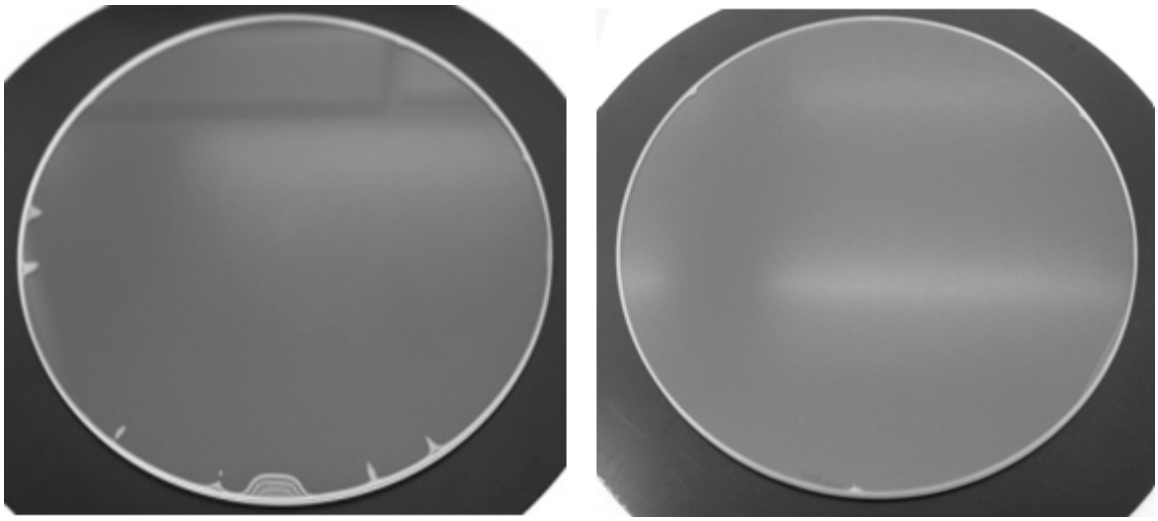

Figure 4: Bonded pair of SiO2 wafers (left) and bonded pair of ULE wafers (right)

To identify possible influences on bonding strength, low pressure (<1 mbar) plasma activation was performed alternatively with Oxygen or Nitrogen gas. Besides, the ultimate compressive pressure (after attaining bonding temperature) was varied between ≈1 and ≈2 MPa, and duration of pressure was changed between 2 and 4 hours. The crack opening method was applied to evaluate influences on the bonding strengths.

Table 1: Bonding strengths as determined from razor blade (Maszara) tests

Wafer ID	**Wafer Material**	**Plasma Activation**	**Bonding Pressure (MPa) and time**	**Bonding strength (J/m²)**
S272/S273	SiO2	N2	≈2 for 2 h	0.8 ± 0.2
S270/S271	SiO2	O2	≈2 for 2 h	0.5 ± 0.2
S268/S269	SiO2	N2	≈2 for 4 h	1.4 ± 0.2
U027/U040	ULE	N2	≈2 for 2 h	2.3 ± 0.8
U036/U039	ULE	O2	≈2 for 2 h	2.0 ± 0.7
U046/U045	ULE	N2	≈1 for 2 h	1.1 ± 0.1
U037/U041	ULE	N2	≈1 for 4 h	1.7 ± 0.1
U073/U074	ULE	N2	≈2 for 4 h	2.2 ± 0.8

The method involves insertion of a razor blade of known thickness (≈100 µm in our case) between the bonded wafers by force and measuring the length of the opening gap[5]. It is considered less exact than the alternative Chevron method[6], but much simpler and reasonably credible, if two nominally "identical" wafers are involved. Major error sources come from

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an extreme sensitivity to the gap length, the deviation of the gap shape from simple one-dimensional beam geometry, and "corrosion enhancement" from moisture entering the gap. Exemplary results (mean values and standard deviations from at least 3 tests per sample) are given in Table 1 and related to the corresponding processing conditions. The test procedure is illustrated in Fig. 5.

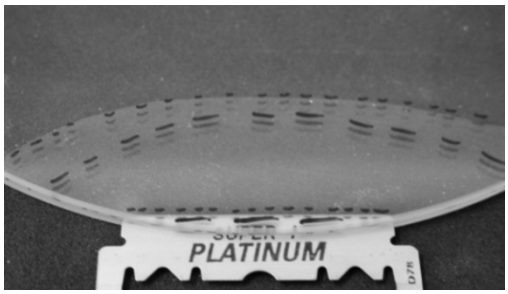

Figure 5: Applying the razor blade (Maszara) test to a bonded pair of ULE wafers

A significantly higher bonding strength (roughly a factor of 2) for the ULE wafers as compared to the SiO2 wafers shows up with similar bonding conditions. Since surface roughnesses and Young's moduli are nearly identical for both materials, this might be related to chemical influences, presumably the TiO2 content of ULE. Further, bonding strength seems to improve with pressure at the selected temperature, which is probably related to the poor roughness of the surfaces. Finally, a trend to slightly higher bonding strengths with Nitrogen activation relative to Oxygen may be suspected, but statistics and resolution are too poor for a definite conclusion.
Bond strength is in reasonable agreement with previous results of ≈0.6 J/m² for bonding of SiO2 wafers of 200 mm diameter and ≈0.8 mm thickness under comparable conditions[4]. Yet, these values appear rather poor when compared to about 2 J/m² as obtained for essentially equivalent bonding in case of Si-to-SiO2 wafers[7] (at probably reduced surface roughness). Note, that the corresponding values for ULE wafers match well with the latter result, which approaches the "limiting" value of about 2.5 J/m^2, estimated for "low temperature" (<600 °C) bonding of oxidized Si-wafers[1].

5. BONDING A WAFER TO A MASSIVE SUBSTRATE

To take advantage of a suspected higher bonding strength with Nitrogen, only this gas was used for low pressure plasma activation in the following bonding processes involving "thick" (17 to 20 mm) glass substrates of diameter 150 mm and thin (1.5 mm) glass wafers.

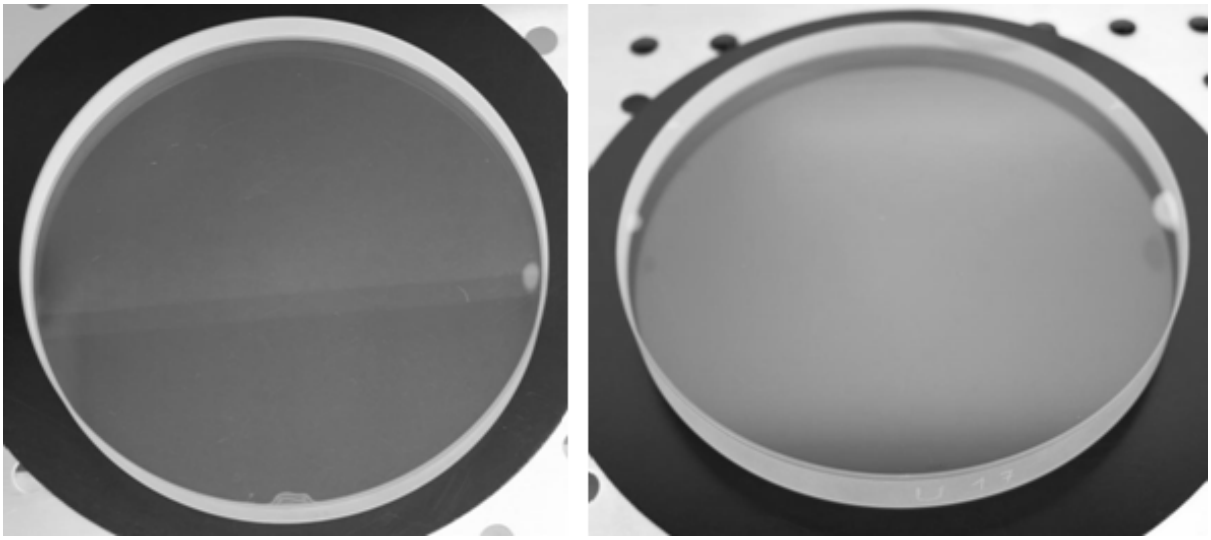

Figure 6: Glass wafer bonded to massive glass substrate of identical material (left: fused silica, right: ULE)

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Compressive pressure at temperature was set to ≈2 MPa and time at temperature to ≈2 h. Exemplary results of bonding a thin glass wafer to a massive glass substrate of identical material are shown in Fig. 6 for fused silica (SiO2, at the left) and ULE (at the right), respectively.
There are several bonding defects visible at the sample rim in both cases. In the left of Fig. 6, the laser numbered face of the wafer was bonded accidentally, creating a defect area with interference fringes at 6 o' clock. Further defect areas are visible as cloudy regions close to the rim at 3 and 9 o'clock. They mark domains, where no compressive pressure can be applied during the bonding period, due to constructive constraints. Surprisingly, this phenomenon was barely observable when wafer pairs were bonded. An influence of the increased stiffness of the massive substrate appears to be effective, although the exact mechanism is not clear at this time.

6. LIGHT-WEIGHT "SANDWICH" BONDING

Weight is an issue in all dynamic imaging applications with optical elements (for example when used as a scanning mirror in pointing tasks etc.) and is also critical in large quasi-static applications, like in astronomic observation. This holds for earth bound telescopes and all the more for usage in space. To avoid distortions from uncontrolled thermal drift of the functional surfaces, ultra-low expansion materials are utilized preferentially in specific light-weight designs for these applications.
Direct bonding of thin glass wafers (or even shaped components) from both sides to a light-weight core structure may provide a simple alternative to the much more sophisticated manufacturing processes of such elements used today. An extensive incorporation of recesses into a bulk material may be replaced by a simple "straight through machining" of the core material in combination with a strong and stiff bonding technology of negligible thermal impact. Apparently, using ultra-low expansion materials and the "Direct Bonding" technology as described above, combines the great benefits mentioned in the introduction with the material advantages of thermal invariance.
The full potential of this approach was not explored, but rather "symbolically" –in a first step to such an application– a light-weight core was manufactured by simply boring several holes through a plane, massive ULE substrate of about 20 mm thickness and 150 mm diameter. This reduced weight moderately by <30 % (a higher weight-reduced prototype with hexagonal openings is in preparation). Then, to obtain a reasonably stiff "sandwich" structure, two ULE wafers of 1.5 mm thickness were bonded from both sides to the core plate by essentially the same process steps as described above (only the time at temperature was increased).
For the sandwich structure, a bonding strength comparable to the respective ULE wafer pairs above, i.e. about 2 J/m² (or slightly better in view of the lower roughness of the core substrate), is expected.

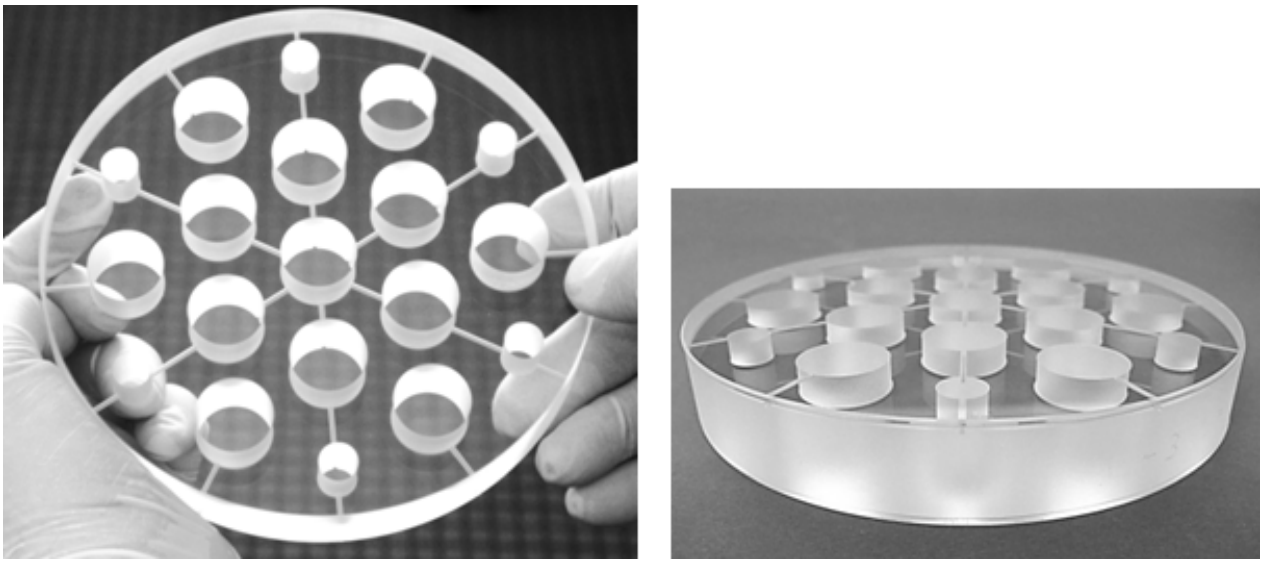

Figure 7: ULE "sandwich" structure (diameter 150 mm, thickness ≈23 mm)

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The results of "sandwich bonding" are displayed in Fig. 7 from two perspectives. Note, that the top and bottom wafers are completely "closed", i.e. free of holes or other openings, to obtain a maximum stiffness and exclude any micro-cracks as possible sources of fracture at the loci of maximum stress under bending. As can be inferred from Fig. 7, a high bonding quality with respect to visual transparency (without the edge defects of Fig. 6) was obtained.
For use in a vacuum environment as well as under ambient conditions, small ventilation grooves were dug radially into one side of the core plate, as can be seen in the figure. Although not expected (since previous investigations revealed residual stress from bonding only around particle contaminated areas[4]), an interferometric investigation for possible distortions of shape from internal stresses is planned to take place under temperature cycling conditions in the near future.

7. CONCLUSIONS

Optical contacting is known in optics since more than 100 years, but risk of failure was rather unpredictable in the past. Research into "direct bonding" of oxidized Si wafers has elucidated the basic bonding mechanisms and eventually paved the way for a renewed application in optics with more calculable, reproducible bonding results.
We have demonstrated successful application of the technology of "direct bonding" to low and ultra-low thermal expansion glass materials (fused silica and ULE, respectively) of diameter 150 mm and thickness up to about 20mm. Sample characterization, surface cleaning and activation processes, bonding conditions and bonding results were described in detail. Although surface roughness of the glass wafers was critical with respect to common experience in Si-wafer bonding –about 1 nm RMS– essentially defect free, fully transparent bonding was obtained, except possibly for the edge regions. Bonding strength was evaluated with the destructive gap opening method on bonded wafer pairs. This revealed a significantly higher bonding strength for the ULE materials as compared to fused silica (SiO2).
Finally, application of the bonding technology to the assembly of a light-weight "sandwich" body, made from a perforated plane core plate and two wafers (all made out of ULE materials) was demonstrated. This indicates, that the technology of "direct bonding" may be used for manufacturing of light-weight and stiff optical elements, like platforms, scan mirrors, optical resonators or other compounds for use in a broad range of opto-mechanical applications.

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