LASER INTERFEROMETER GRAVITATIONAL WAVE OBSERVATORY

LIGO Laboratory / LIGO Scientific Collaboration

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Ear de-bonding ideas

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The change of ribbon to round fibers requires the replacement of the fused silica ears already mounted (silicate bond) on the LASTI pathfinder test mass.

Possible mis-assemblies or damage to ears may require further de-bonding.

Present experience with de-bonding

This is a collection of past experiences, more information may come from the experience in LISA.

- The first unintentional de-bonding experiment, or better accident, at LIGO was at the beginning of the LIGO bonding experience. Riccardo lifted a Glasgow test mass from the four ears and two of them (on one side) came off.

The lifting shear effort from the fingers was sufficient so that one of the ears came off clear (no trace of bonding left) and the transient stress broke the second ear on the same side. The break was on the test mass side and left a crater on the test mass.

This incident is not a controlled de-bonding, and is reported mainly to illustrate the dangers that may be involved in de-bonding.

Helena successfully de-bonded several flawed bonds. There were bonded for less than an hour. She separated some of them by submerging them in DI water, on others ultrasound was used. After de-bonding there is a bonding residue in the glass, like a heavy water mark that can not be cleaned with just water, it needs to be scrubbed with a mild polishing compound. Cerium oxide or calcium carbonate were used in those instances.

- The silicate bonding process was used extensively for the construction of GP-B at Stanford. Roger Route at Stanford sent the document in appendix 2, and referred us to Sheila in Glasgow, there has been no activity on that front for decades at Stanford and the know-how is practically dead.

The salient points of the report are:

“Although curing to full strength takes few weeks depending also on the interface area and geometry, it takes only one or two days before we may safely handle the bonded part”

The bond is “water resistant, No concern about humid environments, reliable for under-water applications, survives wide pH range.”

Which is not comforting from the de-bonding point of view.

- Sheila’s response is as follows.

We removed an ear shortly after bonding it (a few hours after bonding) - I don't remember doing that with the GEO ears after a longer period.

However we have removed ears from other systems a long time (months) after bonding, and actually have done quite a lot of studies of de-bonding for another project.

In all cases we used an ultrasonic bath with hot DI water and some detergent.

n.b.: if de-bonding samples that have been bonded for long time it can take a -long-time- for the de-bonding.

Samples which had been bonded for 14 days took ~9 hours to de-bond.

Clearly if one is going to expose polished surfaces to the action of detergent and ultrasonic for extended periods one needs to keep an eye on what the ultrasonic action is doing to the surface of the silica.

- Helios Vocca of Virgo reported as follows.

His experience is that if we used KOH bonding it is hopeless, the bonding would be hopeless to break, mainly because that type of bonding requires lambda/10 flatness to make a solid bond. Although the bonds are slightly attacked by water, there is no significant water penetration.

If used NaOH plus silicates were used, that requires less flatness and there is hope that sufficient water may seep under.

They had success with hot water, time and some shear pressure.

But if we used polished surfaces to lambda/10, then it is next to impossible again.

Also if the bond is older than a few weeks, water does not seem to work anymore.

They found that when they wanted to break a bond they could do it with thermal shocks obtained with alternating flame heating and cold water while maintaining a shear force on the ears (The heat apparently does not transmit as well across the bond and allow some thermal stress despite the low FS TEC).

It is quite brutal but effective in most cases (sometimes it broke the glass though).

Of course he does not recommend it for a real optics.

They are very happy that they abandoned the idea of ears for their test masses.

Katie Green at CSIRO reports that they have done quite a bit of work on bonding and de-bonding with hydroxide solutions (NaOH, KOH, etc) and have also had some experience with bonding using sodium silicate solutions.
There are two silicate bonding techniques:
In the first method the KOH generates the silicates and is generically known as hydroxy-catalysis bonding. The KOH just supplies the hydroxide ions, which catalyse the reaction.

Other hydroxide solutions (eg. NaOH, which was preferred for our particular application) can also be used to catalyse the reaction and form bonds.

The NaOH with the cocktail of silicates that you have used is the second method.

Successful de-bonding of hydroxide bonded samples (first method), without damaging the bonding surfaces of the two components, has two main factors that limit success: the amount of time the two samples have been bonded for and if any subsequent treatments (e.g. baking) have been carried out. We have successfully de-bonded samples bonded with this method up to 4 hours after bonding. De-bonding was carried out via mechanical means - essentially pushing on the edge or corner of one sample and allowing the surfaces to "peel apart" in a controlled way. We varied the design of the de-bonding fixture depending on the physical dimensions of the samples.
If the samples are left bonded for significantly more time, it is unlikely that you will be able to separate them without causing damage to the bonded surfaces.

We have not been able to de-bonded sodium silicate bonded samples without significant deterioration to the bonding surfaces.
We have not pursued the second method since it does not allow the flexibility and control of sample positioning compared to the hydroxy-catalysis method. From the limited experiments we have carried out, you can separate the samples if you do it quickly (< 1 day) but there is likely to be significant damage to the two surfaces.
Sorry I can't offer more details on de-bonding of the second method but we did not pursue it very far, since other properties of the bond were unsuitable for our application.

Situation with the LASTI test mass ears

The good thing is that LIGO uses NaOH and silicates bonding, not K-OH. The bad is that both the flats and the ears of LIGO are polished to lambda/10, which according to Helios, will make things difficult. for the LASTI test mass.

Additionally, the LASTI bondings were performed almost a year ago, which may make the de-bonding operation more difficult.

In theory we could grind off the ears and flip the mass, there is risk involved in this as well. We consider that it is a good idea to try a de-bonding technique for now and later, especially considering that de-bonding with ultra-sound, water and tensioactives is a relatively low risk operation.

We would try Sheila and Helena's recipes in sequence, using a tensioactive detergent first to increase the water penetration and, if that fails, trying Liquinox, which is more aggressive on the bond, but also more dangerous on the coatings

We should leave the idea of shear effort (in addition to Liquinox and ultrasound) as a last resource, as it often result in chipping either side of the bond (as illustrated by the first example).

Of course we can forget the idea to use a torch against the optics, a desperate technique that was

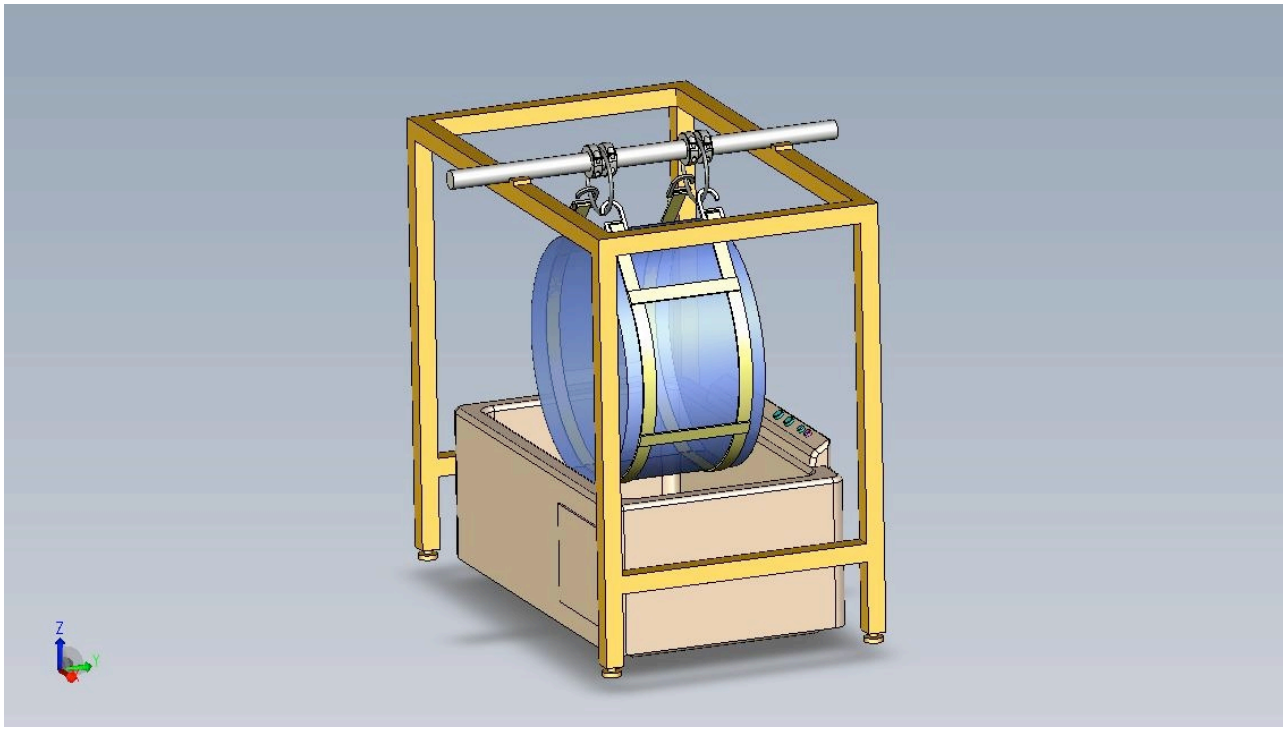
used only on cheap R&D parts and that would wreck the mirror surface.

Precautions have to be taken to protect the mirror surfaces, even with the ultrasound technique.

Naturally we are not thinking of sinking the test mass in a large bath and leave it there for hours or days, the solvent and extensive UltraSound action may damage the mirror surfaces (Liquinox is now to do it). Additionally dipping just the ears and the flats in ultrasound may localize the effects of Ultrasound and make it even more effective

The first question is how to suspend the mirror over the UltraSound bath with the ears dipping in the liquid.

Part of the setup shown below (except the straps themselves) was built, is already at LASTI, and could be used to suspend the test mass.



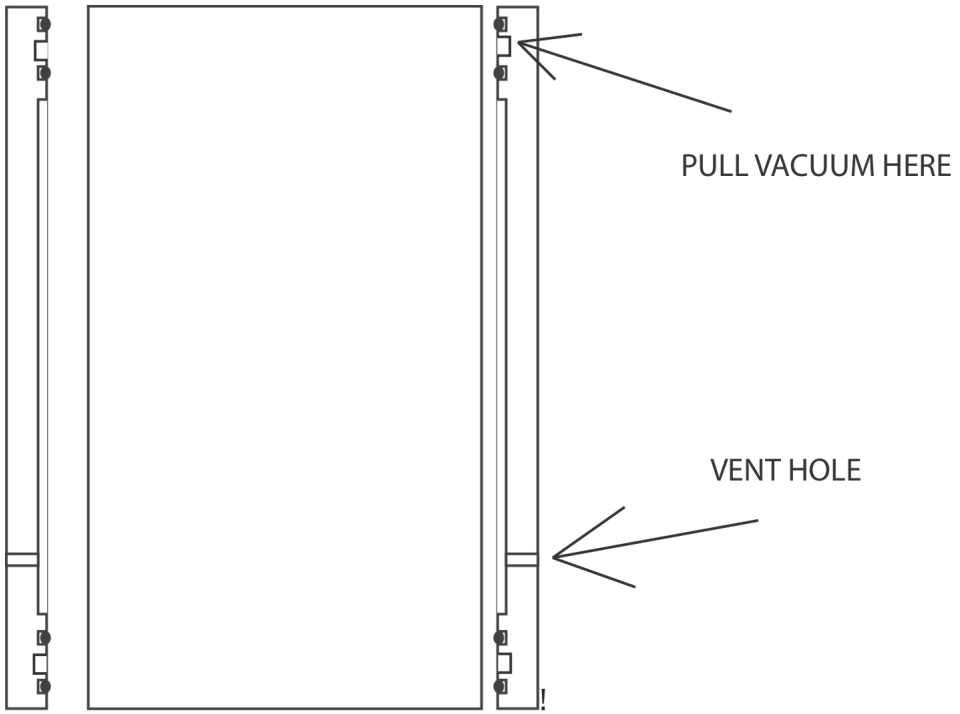
There is the problem on how to protect the mirror surfaces.

Making two simple O-ring sealed cups and pulling vacuum is considered risky, because it could suck water and detergent in, just the opposite of what desired.

Vacuum as a holding mechanism is attractive though, and was used for the GEO optics.

A compromise solution, useful for the LASTI test mass only, could be two Teflon lids with the same OD of the test mass (allowing for the flats of course).

Each lid would have two concentric O-rings, one near the periphery, and one at a radius 2-3 cm smaller. Vacuum would be pulled continuously between the two rings, while a small venting hole in the central part would keep that from being de-pressurized.



This solution allows the use of light disks because, although the holding force would still be of the order of 1000 N, the force is localized to the periphery, the lids are not subject to pressure over the entire surface and there is no need to make them very stiff.

Any leak that would allow water to be sucked in the evacuated volume would result in liquid confined in the outer ring, far from the used central region (not the case for a real test mass though).

Some elastic straps connecting the two lids are recommended to hold them in place in case of lock of vacuum.

We recommend that we go ahead with this technique to de-bond the ears from the LASTI test mass, starting as soon as possible, as there are no counter-indications. Note that it may takes from many hours to days to de-bond, if successful at all. If everything fails we can always grind off the existing ears and flip the mass.

If we successfully de-bond these old bonds we will have learned something useful.

The probability of damage to the TNI mirror to such an extent as to impede its use is very low

(mainly connected with violent accidents like dropping the mass), but even in the case of such an accident it is useful to incur into it early on, to have the maximum possible reaction time.

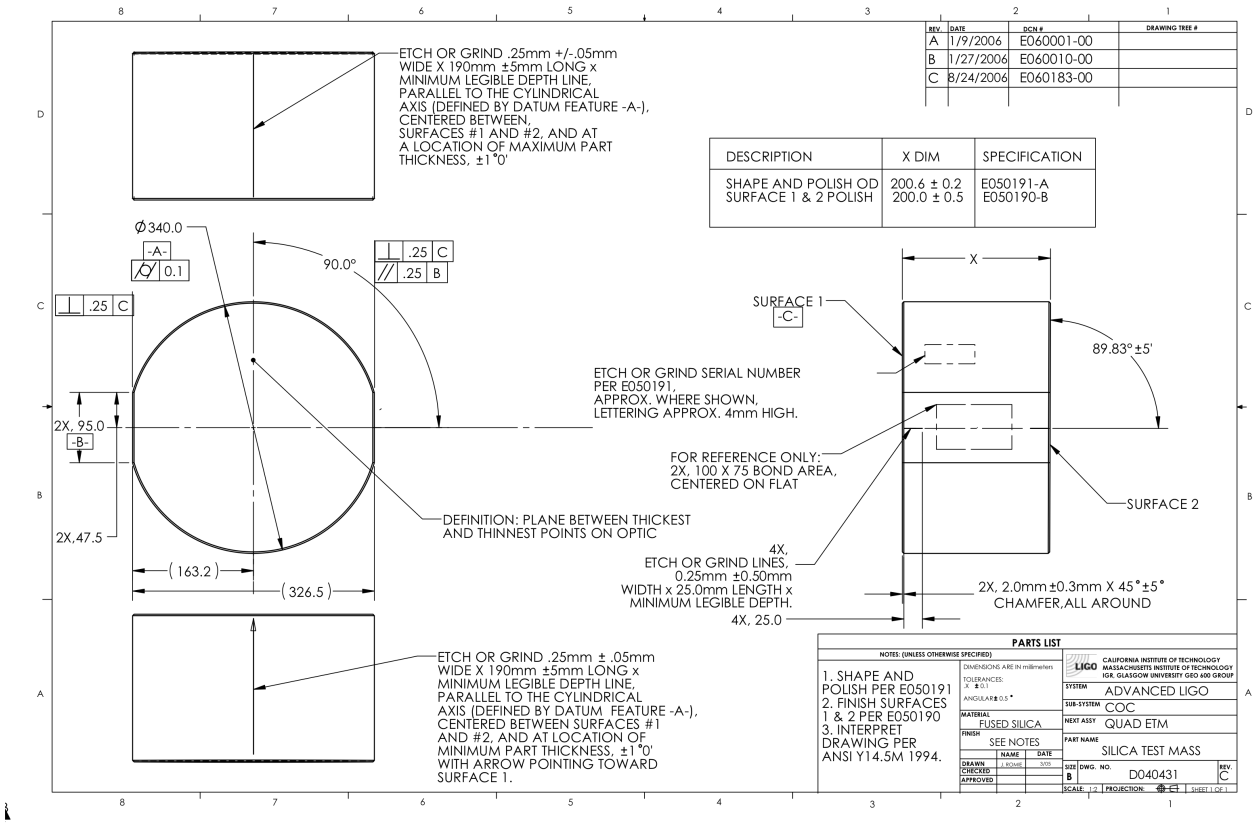
Additional tests

Helena has scavenged four centimeter-size samples, a few years old. Two of the samples are FS to FS and two are FS to Sapphire. We could use them to play with.

We also recommend that several ears are manufactured and bonded to flats, and that the de-bonding technique is tested on these test ears samples, at different times from bonding, to test and establish the technique for future use. It is useful, in case of further mistakes, to determine the length of the grace period in which we can expect to be able to successfully de-bond from a real test mass without causing it damage.

Appendix 1

LASTI test mass drawings



Appendix 2

Stanford patent application

An Ultra-Precision and Reliable Bonding Technique Using Hydroxide-Catalyzed Surface Hydration/Dehydration

(One attachment to the patent application)

Dz-Hung (Jason) Gwo

Feb. 26, 1997 (Rev.3)

A. General Purpose of Invention

The technique was invented to bond solid materials whose surfaces can be hydrated and dehydrated through hydroxide catalysis, in a simple, inexpensive, yet extremely reliable and optically precise manner.

B. Technical Description of Invention

To establish strong chemical bonds, the new bonding technique employs aqueous solutions of hydroxide(s) with or without some kind(s) of silicate, such as aqueous solutions of

* NaOH with or without silicate, or
* KOH with or without silicate (best for silica-based materials)

where silicate refers either to highly hydrated silicon dioxide dissolved in aqueous solution or to other forms of water soluble silicate salts. Here hydroxides serve as catalysts not only (a) for the hydration reaction of the bonding surfaces, and, in the presence of silicate, of silicate as well, but also (b) for the dehydration reaction among the hydrated bonding surfaces and the interface siloxane network resulting, for example, from the hydroxide-catalyzed dehydration of silicate.

Therefore, it is preferable for the bonding surfaces to have surface hydroxyl groups, which are signs of surface hydration. (In this case, water molecules trapped in the interface may also contribute some bonding strength through hydrogen bonds.) Typical examples are surfaces of silicon, silica, silica-based materials, aluminum, alumina, alumina-based materials, iron, etc..

As a result, the bonding is dominated by strong chemical bonds rather than weak van der Waals or hydrogen bonds, as evidenced by the extensive bulk fractureing observed in attemps to shear along the bonding interface.

In the case of *silica-based* materials, the interface siloxane network can be generated *in situ* from the hydration/dehydration reaction of the bulk

materials near the bonding surfaces. In other words, for silica-based materials with reasonably fast surface hydration rate, such as fused quartz and fused silica, it is generally unnecessary to use silicate, which however usually helps shorten the settling time in the bonding process. (Footnote: Settling time refers to the time needed for the bonding becomes impossible to be sheared or split manually without leaving permanent surface damage on the bonding surfaces.) For non-silica-based materials or silica-based materials with slow surface hydration rate, it is generally suggested that silicate be part of the aquaeous solution to increase the effective fill factor for the interface figure mismatch, and thus to maximize the bonding coverage and strength.

The concentrations of hydroxide and/or silicate in the bonding aqueous solution may vary widely according to the application envisioned. For example, when applied to a 1-inch diameter circular interface between fued-silica and fused-silica, a silicate-free KOH aqueous solution with a KOH : H_2O molecular ratio of 1:500 may allow a settling time of ~40 minutes, facilitating precision optical alignment when needed. However, for another specific example, when the equivalent molecular ratio of NaOH : SiO_2: H_2O is 1 : 1.3 : 57, the settling time becomes as short as few tens seconds. (However, solutions saturated with hydroxide, particularly NaOH, occasionally do not work properly.) In other words, the settling time is adjustable through concentrations of hydroxide and silicate.

The differences between the options of, for example, NaOH and KOH as applied to fused silica or fused quartz, are as follows:
(1) Bonding resulting from KOH is expected to have a higher temperature rating. (Being verified)
(2) KOH is less aggressive in terms of hydration than NaOH. Therefore, KOH provides a higher recoverability of bonding surfaces in case debonding is necessary, for example, when misalignment happens before the bonding settles.
(3) KOH generally results in higher mechanical strength.
(4) When cured, excess KOH solution, as free of silicate, in the chamfer around the interface generally causes no particulate residue around the interface edge. However, excess NaOH solution and solutions containing silicate sometimes do.

So far extensive testing has been done successfully in a Class-100 clean environment with fused-quartz and fused-silica samples having sub-micro-meter bonding surface figures over dimensions ranging from few mm's to ~20 cm. The results indicate the bonding material serves as a filling material up to at least a sizable fraction of a micro-meter, which is so far limited by the surface figures and the filter element used in the solution preparation. (The maximum filling dimension in the direction normal to the interface remains to be tested.) The upper bound for the minimum interface width is currently measured to be ~10 nano-meter, which is the resolution of the scanning

electron microscope employed to verify the bonding profile. These facts imply the bonding method can be used in situations when precision alignment is stringently required. It is certainly preferable to conduct the bonding in a clean environment to maximize the success rate. (In fact, bonding from optical-quality surface to fine-ground surface as well as bonding between two fine-ground surfaces have both been demonstrated.)

It is helpful to have intrinsic surface hydrophilicity maximized, and surface hydrophobicity, such as caused by organic or silicone contamination, minimized. However, surface chemical cleanliness is less critical than in the case of optical contacting.

Minimum volume required is approximately 50 nano-liters per square centimeter when the volume loss to the chamfer area due to the capillary effect is negligible. If the amount applied is too little, there could be a risk of premature bonding caused by inadvertent optical contacting before proper alignment is achieved. Although excess volume does not degrade the bonding performance in general, it might leave residues around the edge of the interface. There are many ways of applying the bonding material to the bonding surfaces, including direct pipetting, atomization (aerosol), etc.. When pipetting is chosen, the actual number of drops depends on the interface area as well as geometry, which determines the volume loss at the chamfer, and the capillary effect between the two bonding surfaces as gently compressed can help to spread out the bonding solution. (In most cases, as a safety practice to avoid premature bonding, no force other than the gravity due to the upper part of the interface is needed at all.)

For fused silica or fused quartz, precision alignment is allowed during the initial settling time typically from few tens seconds to ~30 minutes depending on the bonding solution chosen. Although curing to full strength takes few weeks depending also on the interface area and geometry, it takes only one or two days of waiting before we may safely handle the bonded part in a regular environment. (Many of the bonded samples, with no exception, safely went through further wet polishing and violent machining processes only ten days later.) Three to four days after the bonding, we may optionally bake it for several hours with microwave (for example, in a 700 W 2.45 GHz household system), which roughly doubles the curing speed. Other conventional baking methods such as using vacuum oven may also be used instead if the advantages of using microwave, such as zero-temperature gradient across the interface, are unimportant. (As a footnote, any metal or semiconductor part of the sample needs to be electrically grounded during the microwave baking.)

C. Advantages and Improvements over Existing Methods

This new bonding technique is superior in many aspects to index-matching optical epoxies, optical contacting, and Corning's proprietary high-temperature frit bonding. It essentially combines all advantages of these common approaches, and avoids almost any of their concerns and problems in a wide temperature range.

Primary advantages (Primarily test results from fused silica and fused quartz samples):

• Extremely high mechanical strength
- Close to fused quartz/silica (Nominal shear strength for H_2O : KOH = 500 : 1 higher than 4000 psi.)
- Mechanical strength tests always result in tearing of bulk material.
- Percentage uncertainty bar in strength smaller than conventional optical contacting
- Can survive any conceivable spacecraft launch load

• High reliability
- More reliable than optical contacting in any regard

• Tolerate wide temperature range, thermal shocks and cyclings.
- Tested range: [4, 423] K, i.e. from liquid Helium temperature up to 150°C at least
- Survives ~-20 K/minute cooldown rate to 77 K and ~-100 K/ hour cooldown rate to near liquid Helium temperatures

• A room temperature process
- Easier than Corning's proprietary frit bonding at ~900°C
- No side effects due to high-temperature treatments

• Excellent success rate
- Less demanding in surface requirements but more repeatable than optical contacting

• Settling time & bonding reversibility are adjustable through preparation of bonding solutions
- Settling time adjustable from few tens seconds to 40 minutes
- In case of alignment mistakes, debonding is possible within 40 minutes with bonding surfaces recoverable after proper treatments
- Allows enough time for precision optical alignment

• Nearly-zero interface width
- Under optimal condition, less than 10 nano-meter (resolution of SEM)
- Generally ranges up to 0.2 μm limited by filter element used in solution preparation
- Essential for ultra-precision optical alignment

• Almost no detectable optical distortion from room temperature to near liquid-helium temperatures
- Verified with Fizeau interferometry

• Transparent in the visible and infrared at least
- Corning's frit bond is opaque.

• No degradation observed in accelerated life tests corresponding to more than five years of aging
- No effects on strength and optical qualities

• Outgassing problem for vacuum applications negligibly small

• No magnetic contamination concerns
- Even in perhaps the most magnetic-sensitive applications such as Gravity Probe-B

• Water resistant
- No concern about humid environments
- Reliable for under-water applications

• Survives wide pH range

• Applicable to a wide range of silica-based optical materials and to other materials such as sapphire.

D. Possible Variations and Modifications

According to the chemistry involved in the bonding process, we can easily generalize this method to other silica-based optical materials such as Zerodur, ULE, Borosilicate, etc., even to aluminum-oxide based material such as sapphire. So far, experimental evidence indicates it is potentially applicable to stainless steel. In fact, instead of using sodium and potassium ions in the bonding material, we may use other alkali or alkaline earth metal ions as well for different temperature ratings.

This technique may also be used to bond semiconductor wafers, for example, to create nano-meter scale insulation layer between semiconductors.

E. New Features

(See Section C.)