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Hydroxide-catalysis bonding for stable optical systems for space

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Abstract

Space-based optical systems must be made from lightweight materials which can withstand significant acceleration and temperature changes. Materials such as ZERODUR[®], ULE[®] (Ultra Low Expansion material) and silica are all potentially suitable. Depending on the specific requirements of the optical system and the transmissive or reflective nature of the optical layout these materials can be used by themselves or together to fabricate optical benches. The geometrical layouts of these optical systems are often very complicated and the requirements for mechanical stability very stringent, thus jointing components presents a challenge. In this paper we present developments of a novel chemical bonding process, originally invented at Stanford University for bonding silica components for the optical telescope for the Gravity Probe B mission. Colloquially called silicate bonding, this process utilizes hydroxide catalysis to join optical components to optical mounts to obtain high stability whilst accommodating the requirement for precise alignment procedures.

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1. Introduction

Jointing techniques with high mechanical strength and stability are required for use in the fabrication of optical systems used in space-based applications such as telescope assemblies and optical benches [1]. The technique of hydroxy-catalysis, or ‘silicate’ bonding was invented, and patented [2, 3], at Stanford University by Gwo for the purpose of jointing the fused silica pieces forming the star-tracking telescope assembly used in the Gravity Probe B space experiment [4], successfully launched in April 2004 [5]. We have used a variant

of this technique to fabricate the quasi-monolithic fused silica suspensions now installed in the GEO 600 gravitational wave detector [6] and have carried out experiments to investigate the suitability of extensions of this jointing technique for application in the construction of high stability optical benches such as those intended for use in the proposed LISA space-based gravitational wave detector mission planned by ESA and NASA [1, 7, 8] and in the technical demonstrator for this mission, LISA Pathfinder. The proposed optical bench design incorporates fused silica optics mounted on a ZERODUR[®] slab to form part of an interferometric sensing system contained within a satellite flying in a drag-free mode around a proof mass [8]. Typically the relative positional stability of the components must be high; in the case of LISA the stability in the operating frequency band of 10^{-1} Hz to 10^{-4} Hz must be within a tolerance of \sim a few pm Hz^{-1/2}. In the long term there is a possibility of relative drift of jointed components which may lead to a misalignment of the interference pattern or a misalignment in position of where the beam hits the test mass. To maintain a sufficient overlap of the interfering beams the angular tolerance of each component in tilt or rotation should remain within ± 10 μ rad.

Here we give an overview of the silicate bonding technique and present the results of our studies of the chemical and mechanical properties of the bonds. In particular we have studied methods of extending the time available for positioning of the bonds through modifications of the surfaces to be jointed, controlling the bonding environment and varying the chemical make-up of the bonding solution.

The choice of material to be used in space-based optical systems is usually assessed by its high strength to weight ratio and its low thermal distortion ratio α/κ , where α is the coefficient of thermal expansion and κ is the thermal conductivity of the material. When the optical layout requires transmissive and reflective components the optics are generally made from glassy materials such as silica, ZERODUR[®] and ULE[®].

While many existing bonding technologies are available for the above materials [3], hydroxide-catalysis bonding is particularly suitable for building an assembly that will withstand launch conditions and thermal cycling.

We have studied the relative stability of silicate-bonded components mounted on a ZERODUR[®] slab and report on the results in companion papers [9, 10].

Any technique used to mount the optics to an optical bench must have sufficient mechanical strength to withstand launch forces, thus we have studied the mechanical strength of silicate-bonded components. The optics must be positioned with an accuracy equivalent to ± 10 μ m to achieve required beam alignments, necessitating a jointing technique which allows sufficient time to accurately position optics with interferometric precision.

2. Chemistry of hydroxide-catalysis bonding

As discussed by Gwo [2, 3], bonding may occur between flat surfaces of a number of materials if a silicate-like network can be created between the surfaces. The most obvious of these are silica glass or glass ceramic composites such as ZERODUR[®] or ULE[®]. The chemistry of hydroxide-catalysis bonding can be considered to take place in three steps, namely, hydration and etching, polymerization and dehydration [11].

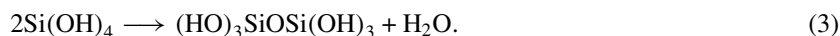
To allow bonding to take place, an alkaline bonding solution, typically sodium or potassium hydroxide or sodium silicate, is placed on one of the surfaces to be bonded and the other silica surface is then placed gently in contact. The OH⁻ ions in the bonding solution act as a catalyst and etch the silica surfaces in contact with the solution: as a result the surfaces liberate silicate ions. This hydration and etching process can be described by equation (1):



As silicate ions are released into the bonding solution the overall number of active OH^- ions in solution is reduced, and so the pH of the bonding solution is decreased. Once the pH of the bonding solution decreases to a value below 11 the silicate ion disassociates to form $\text{Si}(\text{OH})_4$ [11] as described by equation (2):



These $\text{Si}(\text{OH})_4$ molecules can then combine and polymerize to form siloxane chains and water as described by equation (3):



The siloxane chains provide an overall rigidity at the interface and start to form the bond as the water evaporates or migrates into the bulk of the material [12]. As the dehydration continues, a 3D network of tangled siloxane chains rigidly joins the surfaces [11]. Increasing the initial pH of the bonding solution increases the amount of time it takes for the silicate ions to disassociate and thereby increases the overall time it takes for the bond to set. This is discussed in more detail in section 6. Tests have shown that maximum bond strength is achieved after curing for approximately four weeks [14] at room temperature. This time can be reduced by heating the bond or lengthened by cooling.

Other oxide materials, which cannot form a silicate-like network on the surface at a reasonably fast rate, can also take part in the bonding process by chemically attaching to an existing silicate-like network. In the case of sapphire (aluminium oxide) an aluminosilicate network is formed. If such alumina surfaces are to be bonded, a sodium silicate bonding solution (for example 10–14% NaOH and 25–27% SiO_2 with a pH ranging between 11 and 12) is necessary for the formation of the silicate-like network. Further aspects of the chemistry of sodium silicate bonding are given in [13].

3. Method of bonding

For bonding of silica with a hydroxide solution, samples must have an overall global flatness of $\leq \lambda/10$ (where $\lambda \approx 600$ nm). If, however, a silicate solution is used as discussed above in section 2, the tolerance of interface figure mismatch may be significantly relaxed to $\leq \lambda/4$.

To allow the creation of a successful bond it is essential that the surfaces are rigorously cleaned to be free of chemical and particulate contamination. Cleaning and bonding should be carried out in a clean environment. The hydroxide bonding solution (with or without silicate) is then filtered and dispensed on the surface of one of the bond areas, in a scale of $\geq 0.4 \mu\text{l cm}^{-2}$. The second piece is then gently placed on top and may be slightly compressed (depending on the initial viscosity of the solution) to ensure a uniform bond. The pieces are carefully aligned and held in position until dehydration commences. This is the initial setting of the bond. Finally the bonds are left to cure for approximately four weeks, to reach full strength and may be heated. It should be noted however that the samples reach significant strength after the first day.

4. Physical properties of bonds

4.1. Bond thickness measurements

Bond thickness measurements were made to estimate how the relative alignment of optical components may be affected by the joining of two surfaces.

Twelve silica to silica bonds were made, eight using hydroxide bonding solutions with silicate (sodium silicate solution: 14% NaOH, 27% SiO_2 in aqueous solution) and the

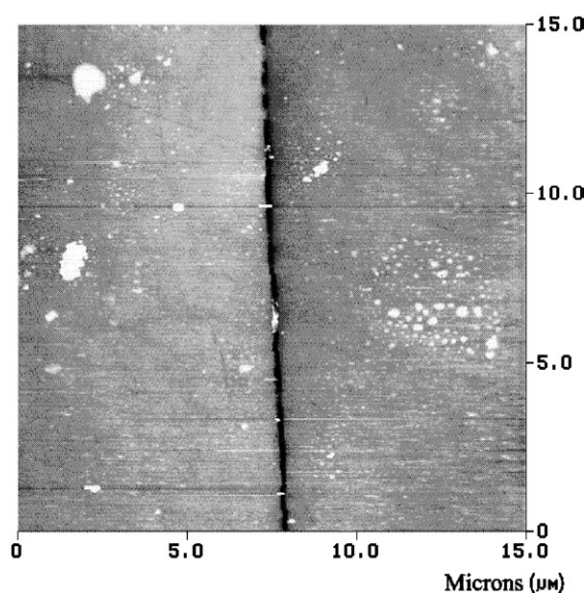


Figure 1. Image of a cross-section of a sodium silicate bond using an atomic force microscope.

Table 1. Bond thickness measurements using an atomic force microscope for bonding solution with and without silicate.

Bonding solution	Concentration	Average bond thickness (nm)	No of bonds measured
KOH:H ₂ O	(1:500) (molecular ratio)	26 ± 6	4
Sodium silicate solution:H ₂ O	(1:6) (volume ratio)	81 ± 3	4
Sodium silicate solution:H ₂ O	(1:4) (volume ratio)	100 ± 7	4

remainder without. These samples were then cut and polished at right angles to the bond layer, so that one could image a cross-section of the bond using an atomic force microscope (AFM) in tapping mode [15] as shown in figure 1. Using standard imaging techniques each measurement was averaged for the bond thickness over a bond length of 15 μm . The values given in table 1 are the average values for four measurements made on four separate samples for a given bonding solution.

It is clear that with a bond of nominal thickness d and length l any wedge angle introduced between the two components being bonded will be much less than d/l . Thus for components of 100 mm² footprint and the thickest bonds in table 1, angular tolerances significantly better than $\pm 10 \mu\text{rad}$ can be guaranteed.

5. Temperature cycling

Mismatched values of the coefficient of thermal expansion can lead to failure when bonds made between dissimilar materials undergo thermal shock. With samples of mass of a few grams, simple estimates suggest that temperature equilibrium is reached in timescales of a few minutes. Hydroxide-catalysis bonds made between materials of mismatched α were dropped in liquid nitrogen ($\sim 77 \text{ K}$) for a period of 5 min and taken out and immediately heated

Table 2. Details of bonds studied in temperature cycling experiments.

Bond type	Bonding solution (volume ratio)	CTE mismatch (at room temp.) (ppm K ⁻¹)	Temp. range (K)	Bond area (mm ²)
Silica to ZERODUR [®]	Sodium silicate solution:H ₂ O 1:6	0.33	77–350	500
Silica to ULE	Sodium silicate solution:H ₂ O 1:6	0.75	77–350	500
Silica to sapphire	Sodium silicate solution:H ₂ O 1:6	3.55	77–350	300

Table 3. Mechanical strength values of silica to silica bonds made with different surface roughnesses (the standard deviation is given as the error in each breaking stress measurement).

Bonding solution (volume ratio)	Bond area (mm ²)	Average breaking stress (MPa)	Bond type
Sodium silicate solution:H ₂ O 1:4	245	0.80 ± 0.33	Rough to rough
Sodium silicate solution:H ₂ O 1:4	50	1.30 ± 0.11	Rough to polished
Sodium silicate solution:H ₂ O 1:4	50	2.14 ± 0.54	Polished to polished
Sodium silicate solution:H ₂ O 1:6	50	1.45 ± 0.08	Polished to polished

under a heat lamp to ~ 350 K. Upon inspection there was no visible sign of degradation in any of the samples after this temperature cycling. Table 2 details the specific materials tested [16]. However, work done at Caltech has shown that baking sapphire to silica bonds (bond area ~ 123 mm²) at 393 K for 24 h may increase the risk of mechanical failure due to a stress mismatch at the bonded interface [17].

6. Alignment of silicate bonds

In order for bonding to occur surfaces must have a minimal surface figure mismatch. When building optical systems, alignment of optical components is paramount. This requires the use of micro-mechanical adjusters *in situ* to manipulate the components into position. Surfaces may optically contact if they are placed in dry contact with one another when testing for alignment prior to bonding.

Experiments have shown that this problem can be overcome in a variety of ways. For example, using a colloidal lapping compound, surfaces can be lightly ground to obtain a local surface roughness whilst maintaining the overall global flatness. This effectively stops unwanted contacting. To test the effect of such a procedure on the overall bond strength three bonds between silica substrates were made, with each bonding surface having a global flatness of $\sim \lambda/10$ and an rms surface roughness on average of ~ 500 nm rms. Subsequently using the same bonding solution three silicate bonds were made with one of the bonding surfaces having a polished surface of $\sim \lambda/10$ with a subnanometre surface roughness and the other surface having a global flatness of $\sim \lambda/10$ and an average surface roughness of ~ 500 nm rms.

The breaking stresses measured for these bonds were then compared to the conventional method by bonding three silica to silica bonds where all the bonding surfaces had a subnanometre surface roughness rms and were polished to a flatness of $\sim \lambda/10$. The mechanical strength was tested in the same manner as described in section 6.3. Results are given in table 3, from which it can be seen that there is some, but relatively small, loss of strength in using bonded ground surfaces.

Table 4. Mechanical strength values and settling times of silica to silica bonds made from various molarities of sodium hydroxide (the standard deviation is given as the error in each breaking stress measurement).

NaOH bonding solution (molecular ratio NaOH:H ₂ O)	Average breaking stress (MPa)	Average settling time (s)
1:8	1.46 ± 0.71	564 ± 15
1:16	2.13 ± 1.27	552 ± 30
1:32	2.70 ± 1.40	355 ± 30
1:64	3.07 ± 0.42	214 ± 17
1:128	4.09 ± 1.83	170 ± 22
1:256	2.51 ± 1.41	116 ± 2
1:512	1.39 ± 0.42	83 ± 11
1:1024	1.06 ± 0.98	50 ± 5

6.1. Variation of bond settling time with molarity of bonding solution

One major advantage of silicate bonding is that as mentioned earlier it is possible to increase or decrease the settling time by varying the pH of the bonding solution. Further, the settling time can also be adjusted by altering the temperature at which the bonding process takes place (see section 6.2). The settling time refers to the time elapsed before the materials become attached and impossible to move without mechanical destruction of the bond.

A room temperature experiment ($T = 291$ K) was conducted whereby small fused silica blocks were placed on silica substrates (contact area ≈ 50 mm²) with a bonding solution of 0.2 μ l dispensed between the contacting surfaces. As a bond formed (as described in section 2) the assembled pieces were slightly perturbed at small intervals compared to the time it takes for a rigid bond to occur. Results suggested that sodium hydroxide bonding solutions provide the strongest bonds between silica substrates for a given concentration (see section 6.3). Three bonds were made for each concentration of sodium hydroxide bonding solution used. A total of 24 bonds were made using bonding solutions of molecular ratio of one part NaOH and 2^{*n*} parts H₂O where ($n = 3, 4, 5, 6, 7, 8, 9, 10$). Initial placement of the silica block allowed for movement in all directions until the bonding solution began to form siloxane chains and the bond region became rigid. The time taken to reach this settling time was measured.

An attempt to repeat the experiment with a caesium hydroxide bonding solution led to the fabrication of only 15 successful bonds because above a molarity of 1.0 the bonds failed to show any signs of adhesion within a 10 min period. A possible reason for this is that caesium hydroxide has a higher mean activity coefficient as a function of concentration [20] which may suggest that the bonding solution failed to polymerize within the time frame allowed as the pH of the bonding solution remained above 11.

The bonds were cured immediately after bonding under a heat lamp for 1 h. They were then left to cure at room temperature for one month before each bond was tested for breaking strength. The results of the mechanical strength tests may be affected by the slight mechanical movement of the pieces throughout the bonding procedure and should only be interpreted relative to other bonds which have undergone a similar process. Results are given in tables 4 and 5. It can be seen that the settling time increases with concentration as predicted in section 2.

6.2. Variation of bond settling time with temperature

An experiment was carried out to investigate the dependence of bond settling time with temperature. A temperature controlled environment (dimensions 1.0 m \times 0.7 m \times 0.5 m) was

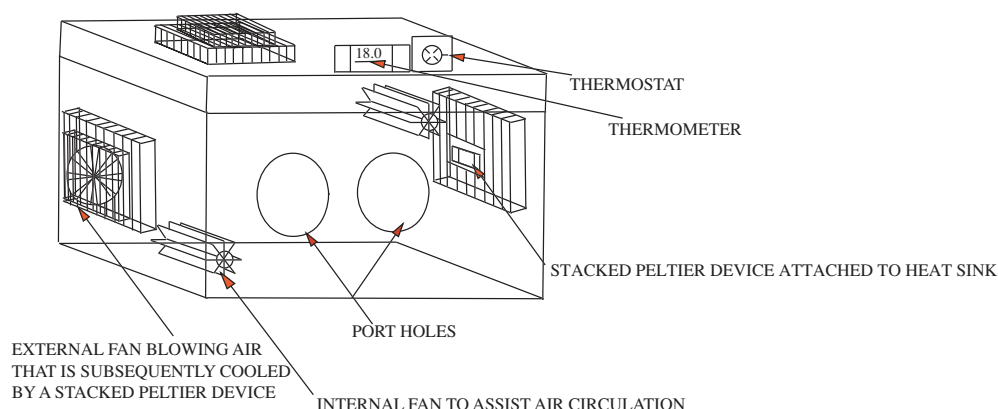


Figure 2. Temperature controlled environment used to fabricate silicate bonds at various temperatures below room temperature.

Table 5. Mechanical strength values and settling times of silica to silica bonds made from various molarities of caesium hydroxide (the standard deviation is given as the error in each breaking stress measurement).

CsOH bonding solution (molecular ratio CsOH:H ₂ O)	Average breaking stress (MPa)	Average settling time (s)
1:64	1.64 ± 0.69	820 ± 10
1:128	2.87 ± 0.25	269 ± 7
1:256	3.01 ± 0.39	185 ± 5
1:512	2.40 ± 0.41	214 ± 19
1:1024	2.18 ± 0.54	136 ± 33

built using multiple Peltier devices to allow the fabrication of bonds at stable temperatures below room temperature, see figure 2. Two internal cross-blower fans were used to circulate the air in a box of approximately 0.35 m³. A capillary thermostat was used to sense the internal temperature and allow control by means of regulating the power to the Peltier devices.

The inside of the box was made from Lexan[®] polycarbonate and was insulated with polystyrene and covered with aluminium foil to reflect radiated heat. The front of the box was double glazed with polycarbonate with a 16 mm air gap for optimal thermal insulation [21]. Two glove holes on the front window allowed access for bonding to be carried out within the environment: these were blocked off by aluminium foil covered polystyrene stoppers when the environment was not in use.

Using a sodium hydroxide bonding solution of molecular ratio 1 part NaOH to 256 parts H₂O, two bonds at each of four set temperatures were made. The settling time was measured in the same manner as previously described in section 6.1. Figure 3 indicates how the setting time varied with temperature.

6.3. Mechanical strength

The mechanical strength of bonds made from various bonding solutions was tested to investigate their capability to withstand launch conditions. The specification for launch for the current LISA Pathfinder mission is that all systems should withstand ~35 × g. The proposed

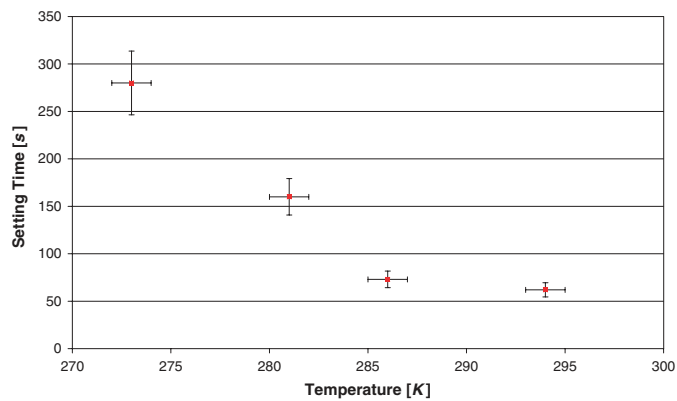


Figure 3. The dependance of settling time with temperature using a sodium hydroxide bonding solution (molecular ratio 1:256 NaOH:H₂O) for silica to silica bonds of bond area $\approx 50 \text{ mm}^2$.

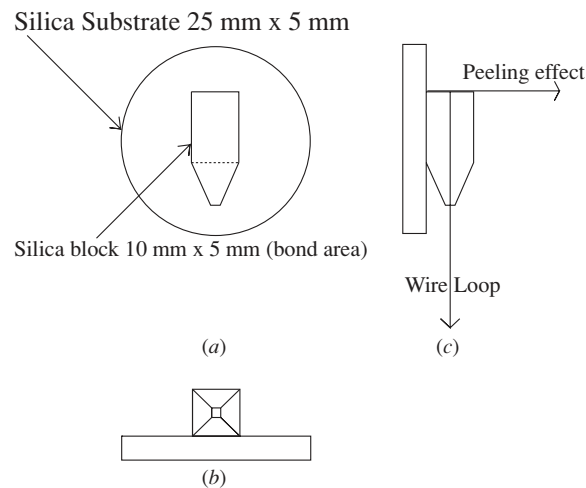


Figure 4. Example of a silica to silica bond used for strength testing: a wire loop is placed around the silica block and attached to a linear drive to test the mechanical strength of the bond. The line of action of the wire is offset from the bond line thus creating a peeling effect at the top edge of the silica block as shown in (c).

bond area for optical components for this mission is $\sim 100 \text{ mm}^2$ with an approximate mass of $5 \times 10^{-3} \text{ kg}$ per piece, which translates to a requirement of bonds between components to withstand a stress of the order of tens of kPa.

Bond strengths were measured using bonds made between small fused silica blocks joined to silica flats (as in figure 4). Five bonds were made for each of four different hydroxide solutions of the same molecular ratio from group 1A of the periodic table. The bonding was carried out in a laminar flow cabinet. All bonds were left to cure for four weeks.

Strength tests were carried out by looping a wire around the small fused silica block as shown in figure 4. The loop was then pulled using a motorized linear drive to the point of bond failure. A load cell was used to measure the maximum force and hence the stress required to break the bonded assembly. Tests showed that the fracture rarely occurred at the bonded interface suggesting that the bonds possessed a strength equivalent to that of bulk fused silica

Table 6. Mechanical strength values of silica to silica bonds made using different hydroxides (the standard deviation is given as the error in each breaking stress measurement).

Bonding solution	Concentration molecular ratio	Bond area (mm ²)	Average breaking stress (MPa)
NaOH:H ₂ O	1:500	50	2.70 ± 0.23
KOH :H ₂ O	1:500	50	1.19 ± 0.14
RbOH:H ₂ O	1:500	50	0.87 ± 0.11
CsOH:H ₂ O	1:500	50	1.31 ± 0.16

(~50 MPa). However, it should be noted that the line of action of the wire pulling on the assembled bond, acted parallel to the bond line but at a distance ≈ 2.5 mm away. This method induced a torque/peeling effect on the bonded region and therefore the results presented here are best described as Von Mises breaking stress values. There is of course some possibility that the water in the bonding solution weakens the breaking stress of the bulk silica. Such an effect was not observed by Gwo [18] but the possibility will be investigated in future experiments.

Five bonds for each hydroxide were tested. It was found that the sodium hydroxide bonds at a molecular ratio of 1:500 with de-ionized water had the highest mechanical strength at an average of 2.7 MPa (see table 6). Results show that the overall mechanical strength of the bond is determined by the hydroxide used. The hydroxides used in this experiment were provided by Sigma-Aldrich [22], the product numbers of which are as follows: caesium hydroxide-23, 204—1, rubidium hydroxide-24, 369—8, potassium hydroxide-30, 656—8 and sodium hydroxide-30, 657—6.

Each hydroxide is a strong base and will ionize/disassociate fully in water. It is known that different cations lead to different forms of silicate structure and this may influence the mechanical strength observed [23]. The results presented here could provide an insight into future work.

7. Construction of a prototype optical bench as a precursor to the engineering model for LISA Pathfinder

Using hydroxide-catalysis bonding we built a prototype optical bench as a precursor to the engineering model for LISA Pathfinder. The aims of this were twofold: to test the practicality of precision aligning and bonding a complicated optical system, and to then use interferometry to test the dimensional stability of the system. The optical bench consisted of a total of 15 silica components bonded to a ZERODUR[®] baseplate (figure 5). The baseplate had dimensions $400 \times 400 \times 25$ mm and the components were all $20 \times 20 \times 5$ mm. For each output beam the optimum alignment was achieved by aligning and bonding the last two components in the beam path. These components had to be aligned to an accuracy of the order of ± 70 μ rad. Each output beam was aligned, the bonds were allowed to set before the next output beam was aligned. The three output beams thus required a total of six components to be precision aligned and bonded.

The interferometric testing of the stability of the completed bench is described in detail in a companion paper [9]. The result was that the optical bench met the LPF stability requirement of 10 pm Hz^{-1/2} over almost the complete frequency range from 3 to 30 mHz. The only exception was a slight excess noise around 3 mHz driven by temperature variations in the test lab. The temperature stability on LPF will be much greater than available in our test lab and this noise source should be reduced to insignificant levels.



Figure 5. Prototype optical bench fabricated using hydroxide-catalysis bonding.

Many of the techniques developed in the construction of this test optical bench were incorporated in the design and construction of an engineering model for the LPF optical bench built at the Rutherford Appleton Laboratory. This model was then subjected to thermal cycling, vibration testing, vacuum testing and stability performance testing and has passed all tests [10, 24].

8. Conclusion

Hydroxide-catalysis bonding has proved to be a simple and effective method for the fabrication of space-based optical systems. Temperature cycling and mechanical strength results indicate that bonds should meet the stringent requirements of both LISA and the LISA Pathfinder missions.

Acknowledgments

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