

# Hydroxide-catalysis bonding for space-based optical systems

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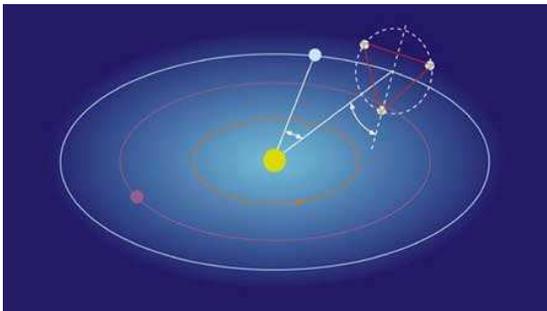
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**Abstract:** The bonding materials for space-based optical systems have stringent requirements for strength, stability, and longevity. The LISA and LISA pathfinder missions will require bonds with femtometer stability that are able to withstand launch stresses of approximately 35g. Previous research has shown hydroxide-catalysis bonding to be a strong candidate for the construction of space-based optical systems. A series of tests were performed to analyze the optimal amount of bonding solution per unit area for different concentrations of hydroxide bonding solutions forming bonds between different types of materials. While the conclusiveness of the results are marred by questionable experimental accuracy, the optimal amounts of sodium silicate bonding solution were determined for several concentrations of solution and as well as for two different types of bonds: fused silica glass to glass, and fused silica glass to polished silicon carbide tile.

## I. Introduction

The Laser Interferometer Space Antenna (LISA) is a joint mission between NASA and the European Space Agency. Scheduled for launch in 2015, LISA is part of the Beyond Einstein program and aims to detect gravitational waves. Although several

ground-based gravitational wave observatories are already in operation, earth-based interference prohibits these detectors from having accuracy in low-frequency gravitational wave detection. The mission's goal is to observe gravitational radiation from binary neutron star systems and the merging super-massive black holes believed to play a critical role in the formation of galaxies. In order to achieve this, the LISA mission is designed to detect gravitational radiation in the 30  $\mu\text{Hz}$  to 1 Hz frequency range. LISA consists of three spacecraft in a triangular formation, separated by five million kilometers, in a heliocentric orbit trailing the earth by twenty degrees (Figure 1). Each spacecraft will contain freely floating test masses which will be shielded from outside forces such as solar radiation or solar wind. As a gravitational wave passes through the LISA constellation, the relative distances between the test masses will change. These changes will be detected through the process of laser interferometry. In a simplified view of the experiment, the three spacecraft can be considered as a large Michelson Interferometer. The interferometry will be accomplished using 1064 nm Nd:YAG lasers. The challenge of the detection process comes from the precision required in detecting length changes. The strongest gravitational waves to reach the spacecraft are predicted to produce typical length contractions of approximately  $10^{-17}$  m/m [1]. Thus, the sensitivity goal for LISA is  $10^{-21}$  m/m. This creates a problem since the laser phase noise will be



**Figure 1: A representation of the LISA spacecraft with respect to Earth's orbital plane**



**Figure 2: A reference optical cavity [1]**

several orders of magnitude greater than the precision required to measure length changes on this scale. As part of the process to overcome this hurdle, each spacecraft will employ a technique known as laser pre-stabilization. Laser light will be sent through an optical cavity made of an ultra-low expansion glass (Figure 2). A mechanical servo will continuously adjust the laser frequency so that it remains at the cavity's resonant mode. Within the context of the LISA mission, the required precision translates to femtometer stability for the materials within the optical cavity used to stabilize the laser's frequency [2].

While all of the materials in consideration for optical cavities have documented coefficients of thermal expansion (CTE) of less than  $2 \times 10^{-8}/\text{K}$ , the bonding methods used to construct the optical cavity have been tested less extensively. Optical contacting is used in ground-based gravitational wave detectors because the bonds are essentially free of noise. However, the launch forces of the spacecraft will require bonds capable of withstanding stresses in the range of megapascals. Developed for the Gravity Probe B mission, hydroxide-catalysis bonding presents a unique solution. In this method, the formation of a silicate network between bonding surfaces produces a bond which can be very strong. While this method has seen much recent evaluation for both strength and low optical interference [3], few data exist relating the optimal amounts of solution per unit area at different solution concentrations to provide the strongest bonds.

## **II. Materials and Preparation**

For the bonding shear strength tests performed, the two materials used were fused-silica glass pieces and polished silicon carbide (SiC) tile. Each glass piece measured approximately 1.27 cm in diameter, providing a bonding area of approximately

1.266 cm<sup>2</sup>. Additionally, each glass piece was polished to  $\lambda/8$  with a 20/10 scratch/dig on one side, and a ground polish on the other giving the option of bonding with rougher surfaces. The tests were conducted with glass to glass bonds and glass to silicon carbide bonds using hydroxide and silicate bonding. SiC is an attractive choice as a material for constructing the telescope support structure. Although SiC has a relatively large CTE at room temperature (compared to materials such as low-expansion glasses and some carbon fiber reinforced plastics), within the realm of the temperature fluctuations at which LISA will operate its CTE is sufficiently low. CTE aside, SiC poses an excellent material choice: it has outstanding thermal conductivity (to keep the temperature gradient across any given portion of the surface to a minimum), a high Young's modulus, and is relatively lightweight [2].

Although hydroxide-catalysis bonding has been shown to be effective on surfaces with an average roughness greater than what would be acceptable for optical contacting, smoother surfaces will still provide better bonds [3]. In order to determine and evaluate

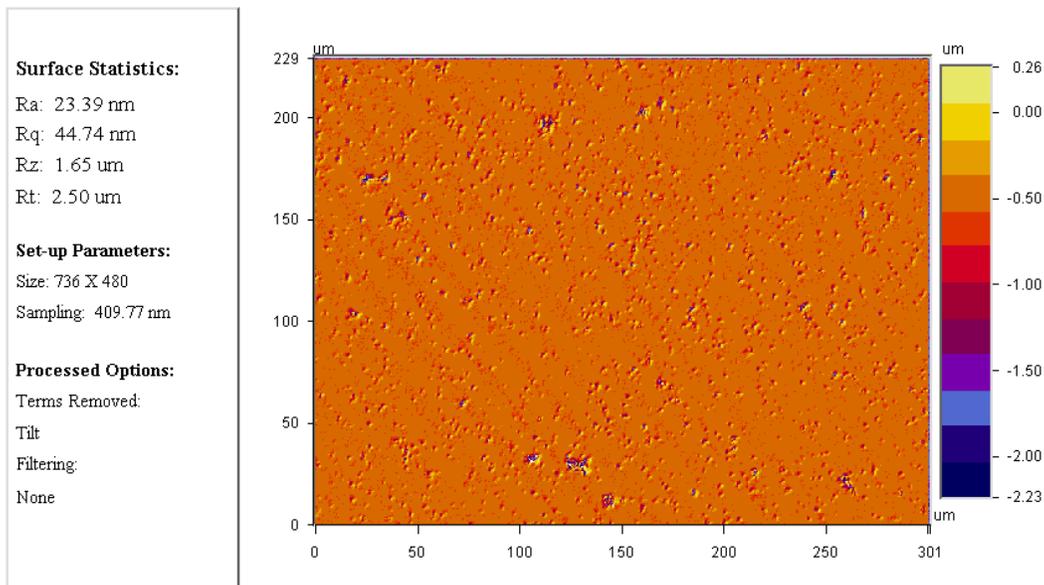
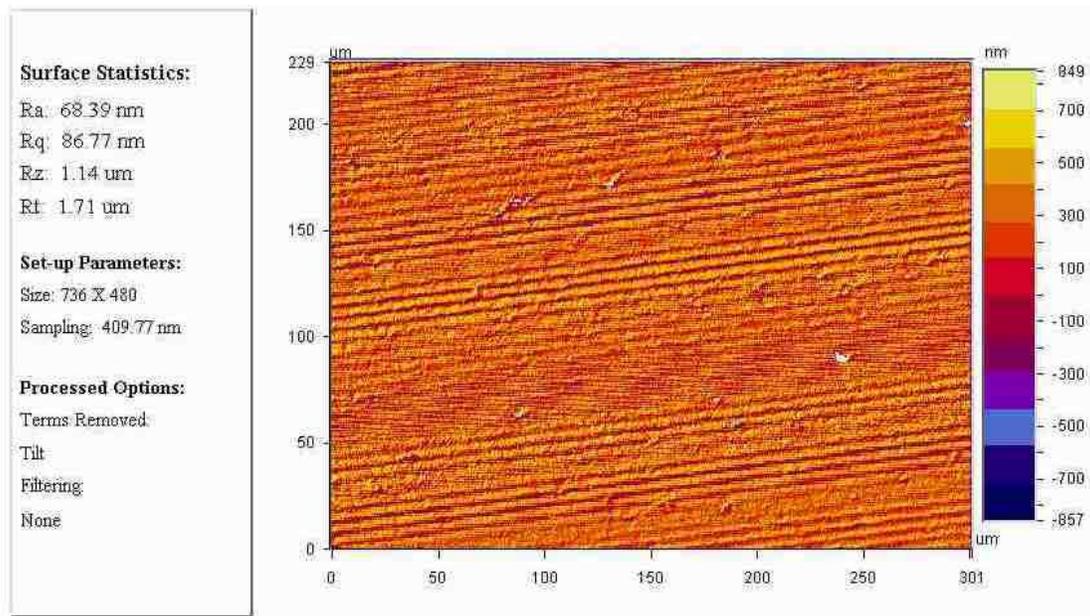


Figure 3: Surface profile of a polished silicon carbide tile



**Figure 4: Surface profile of an unpolished silicon carbide tile**

surface roughness, pieces of SiC tile were examined with a WYCO NT1000 surface profiler. Several pieces of tile were first polished with a solid cerium oxide compound and washed with distilled water and baking soda. Of the six locations profiled on both polished and unpolished tiles, those tiles which had undergone the polishing procedure showed an average surface roughness of approximately 23.5 nm (with a standard deviation of 3.25 nm) while those which did not showed an average surface roughness of approximately 53.8 nm (with a standard deviation of 12.77 nm). Figures 3 and 4 provide a typical comparison between a polished tile (Fig. 3) and an unpolished tile (Fig. 4). As a result of the lower surface roughness, the cerium oxide polishing became part of the materials preparation procedure.

For hydroxide-catalysis bonding to be successful, the bonding surfaces must be free of debris. In order to accomplish this, all bonding and preparation took place in a clean-room environment. All surfaces to be bonded were first rinsed with deionized water and sprayed with compressed nitrogen gas. The test pieces were then placed on a micro fiber cloth in a sonicator. After being immersed in deionized water, the pieces were bathed in ultrasonic waves for approximately 30 minutes. Before bonding, all surfaces

were wiped clean with a sterile optical cloth wetted with methanol. All bonding solutions were administered with a micro-pipette.

### **III. Bonding**

Optical contacting is the adhesion of polished surfaces without the use of any external bonding materials. Highly polished surfaces with very little surface figure mismatch, when brought into contact, form a bond through molecular adhesion. One of the main disadvantages to optical contacting is that once bonds are formed it is very difficult to adjust the position of optical components. This can be overcome by adding a drop of an alcohol-based cleaning solution at the interface between the two bonding surfaces. A weak bond forms, and gradually strengthens as the solution evaporates. During this period of time, adjustment should be possible [4]. Although optical contacting is widely used throughout science and industry, we experienced difficulty in reproducibly forming optical contacts. Although strong bonds were formed, the vast majority withstood so little shear stress that they could not be accurately measured with the breaking procedures used. This was likely due to the lack of a standardized optical contacting procedure rather than a problem with the bonding itself. Many research endeavors have enjoyed great success with optical contacting [5].

In contrast to optical contacting, hydroxide-catalyzed bonding is a chemical bonding procedure. The bonding is achieved by applying a solution containing hydroxide ions to bonding surfaces and allowing a chemical bond to form [6]. To help compensate for surface figure mismatch, a silicate material was also used. The undiluted chemical solution used was approximately 14% NaOH and 27% SiO<sub>2</sub> by molecular weight. The solution was then diluted to different concentrations by volume. As described in [3], the

sodium silicate solution bonding process is best viewed as three separate reactions. In the first stage, the Silicon molecules are hydrated and etched into the surfaces as described by Equation (1):



As time passes, the Silicate ions bond with the Hydroxide ions, reducing the pH of the solution. After the pH of the solution reaches a critical value (about 11), The  $\text{Si(OH)}_5^-$  molecules begin to disassociate as described by Equation (2):



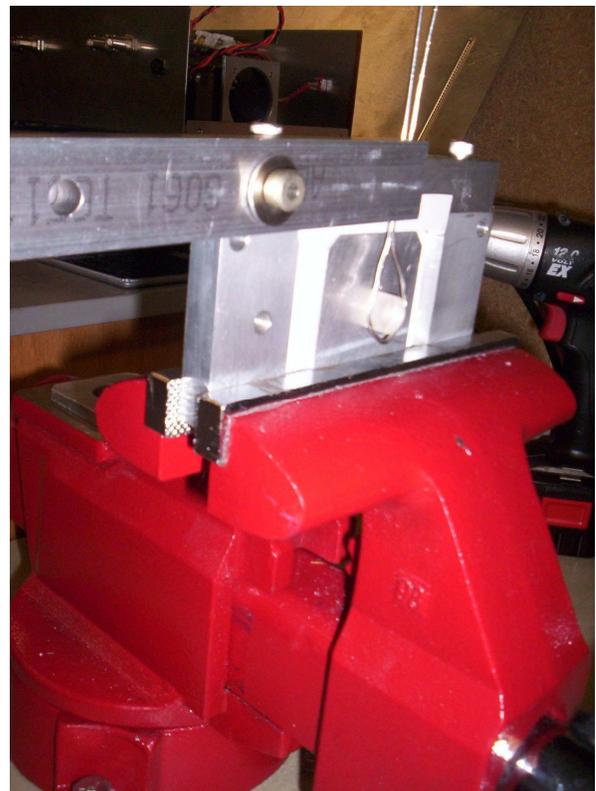
The  $\text{Si(OH)}_4$  molecules then form siloxane chains as described by Equation (3):



These chains provide a rigid structure as the water evaporates and the bond strengthens.



**Figure 5: The lever-arm setup with force meter**



**Figure 6: A closer view of the lever-arm apparatus**



**Figure 7: Polished glass pieces bonded to a SiC tile**

#### **IV. Experimental Setup**

All bonds were tested for shear strength with a specially constructed lever arm shown in Figures 5 and 6. The lever-arm apparatus was equipped with a 3" by 2" slot capable of accepting either a lens holder for breaking glass-glass bonds, or the SiC tiles for breaking the glass-SiC bonds. The samples were held in place with a table-mounted clamp, and the shear stress was administered to the bonded surface via an 1/8" piece of stainless steel wire. The lever arm had an approximately five-to-one advantage, and the force applied was measured with the spring-based hanging force meter seen in Figure 5. It should be noted that, similar to the experiments conducted in [3], the stainless steel wire used to administer the shear stress was several millimeters from the actual bond, which created a slight torque or peeling effect on the bonding area perpendicular to the direction of the shear stress.

#### **V. Results**

A varying series of tests yielded some useful data. Figures 8-11 represent some of the key results graphically. All of the bonds were broken after being allowed to set for 24 hours in a class-100 clean room. With the strength tests, each data point represents a minimum of three samples (and as many as six). In all graphs, the error bars represent the range of the data, and the blue dots represent the average. All glass pieces used for bonding had a surface area of  $1.266 \text{ cm}^2$ . Figures 8-10 compare breaking strengths versus the amount of solution used. A critical point to consider when viewing these results is the wide range of shear stresses the fused silica glass is able to withstand. Above about 3.75 MPa, the glass was sometimes observed to fail before the bond.

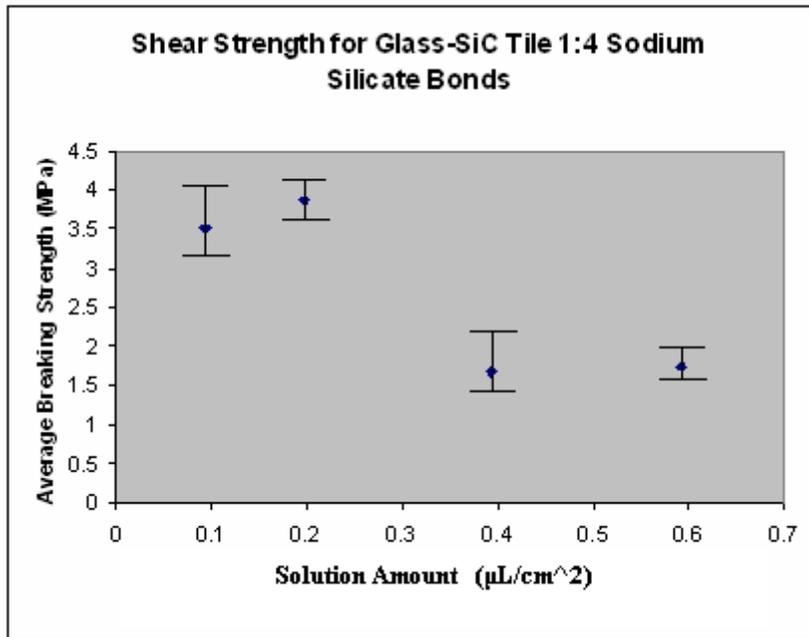


Figure 8

Figure 8 represents the shear strengths for polished glass to SiC tile bonds using a (volumetric) 1:4 dilution of the sodium silicate solution discussed previously. Average breaking strengths of a set of samples are showed versus the amount of solution used per square centimeter of bonding surface area.

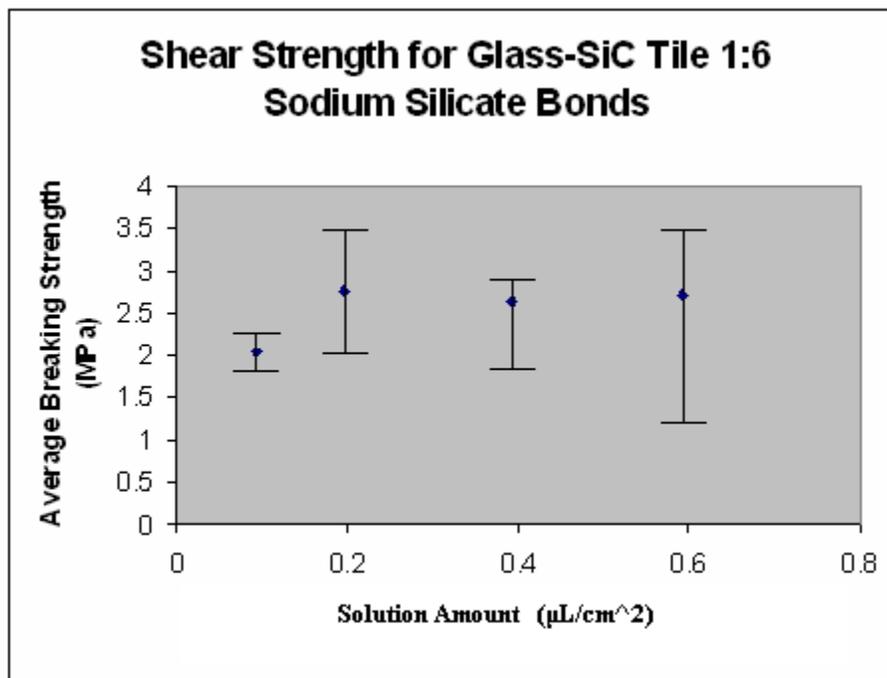


Figure 9

Similarly, Figure 9 represents the same setup, but with a 1:6 volumetric dilution of the sodium silicate solution.

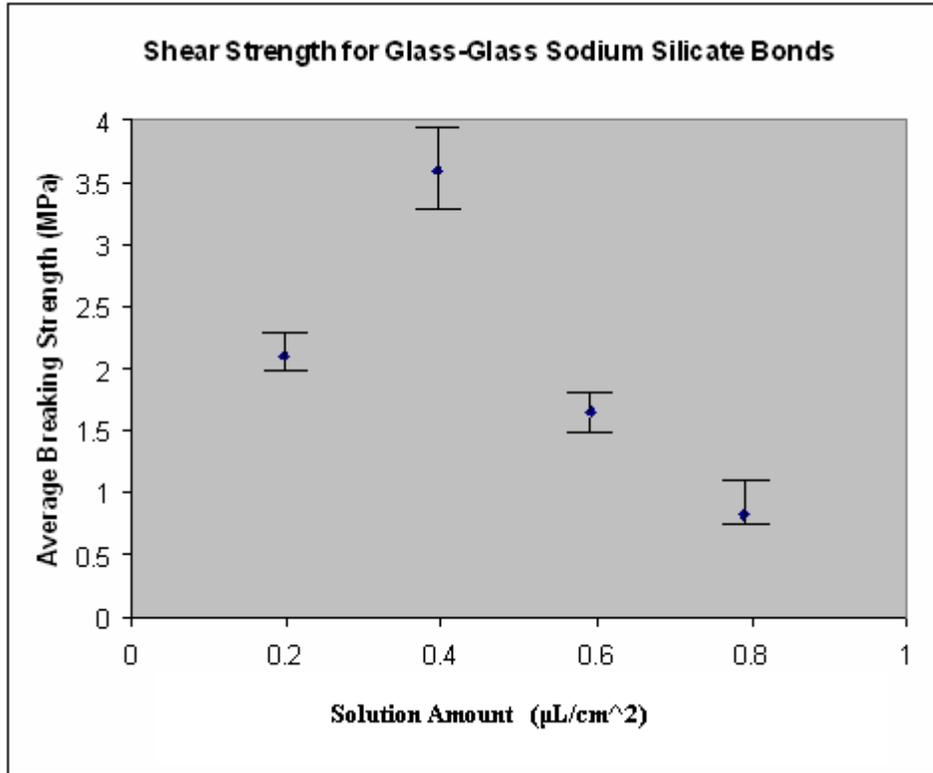


Figure 10

Figure 10 represents shear strengths for polished glass to glass bonds using a 1:4 dilution of the same solution. The results suggest that for different types of surfaces, there exists an optimal amount of solution per unit area. For unknown reasons, the 1:6 sodium silicate bonds showed a much greater variation than the others, and no trends are clearly visible.

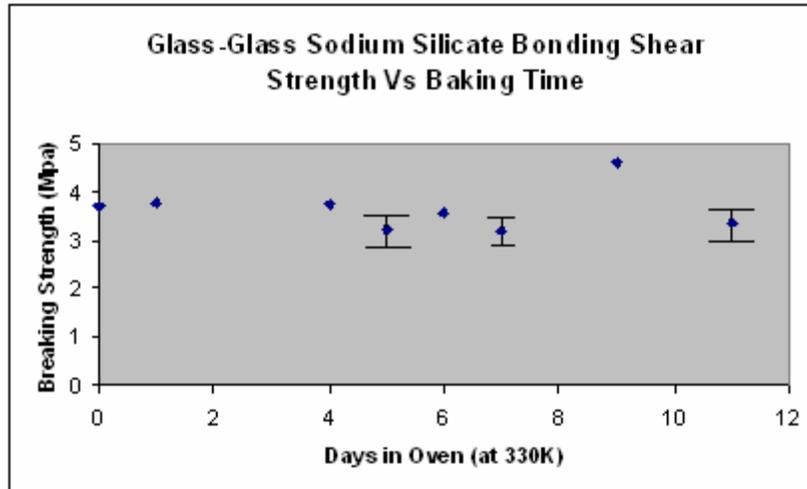


Figure 11

Figure 11 represents the results of a drying time test. Glass to glass bonds were made with approximately 0.4 microliters/cm<sup>2</sup> of 1:4 diluted sodium silicate solution. The bonded surfaces were first allowed to sit for 24 hours. One set of bonds was broken as an initial control point, as shown in Figure 11. Another set of bonds remained in the clean room as an additional control. The remainder were placed in an oven at 330K. After a varying number of days, the shear strength of the bonded surfaces were tested. The unheated control pieces were broken after 11 days in the clean room and yielded similar breaking stresses: both were about 3.2 MPa. Although the bond strengths overall varied by as much as 25%, the test did not yield a conclusive link between bond shear strength and drying time.

## VI. Uncertainty and Questions Raised

Many observable factors beyond our control contributed a great deal of uncertainty to the previous tests. One of the greatest problems was the variability of the internal breaking stress of the glass pieces used. We conducted a follow-up test of several unbonded glass pieces. Four samples were loaded into the lever arm and broken in the

same manner as the bonded pieces described in the previous section. The results were rather unsettling: the breaking stresses on the four unbonded glass pieces showed a wide range of internal breaking stresses. We observed a range of breaking stresses of 3.76 MPa- 7.87 MPa, with an average of 5.67 MPa. As mentioned briefly in Section V, the fused silica glass occasionally failed before the bond. During the glass to glass 1:4 sodium silicate bond shear tests, for example, one glass piece failed at 3.3 MPa with the bond still intact, while in another case the glass remained intact until 4.725 MPa where the bond failed. In the first case, the strength of the bond was not accurately measured since it was not the bond that failed, but rather the glass itself. There is a strong possibility that the bonding solution weakens the internal structure of the glass to varying degrees, since it may seep through the glass pieces as the bond settles. Additional material property tests and extended shear strength tests with a wider range of samples and additional variables including settling time may help to clear some of this uncertainty.

## **VII. Conclusion**

Although further study is obviously needed, hydroxide-catalyzed bonding has continuously shown itself to be very strong. As discussed in the previous section, the bonds are often stronger than the bonding materials themselves. Similar results were also seen with hydroxide-catalyzed bonds between fused silica glass in the experiments done by [3]. Although many of the bonding technique requirements for LISA (temperature stability, bond thickness, contraction and expansion, etc.) are outside the scope of this paper, from the perspective of shear strength, hydroxide-catalyzed bonding has so far proven itself to meet the LISA requirements.

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