



Shear strength evaluation of hydroxide catalysis bonds for glass–glass and glass–aluminum assemblies

Hyo Soo Kim^a, Tony L. Schmitz^{b,*}

^a Department of Mechanical and Aerospace Engineering, University of Florida, Gainesville, FL, United States

^b Department of Mechanical Engineering and Engineering Science, University of North Carolina at Charlotte, Charlotte, NC, United States

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ABSTRACT

Precision bonding is an important manufacturing step for many glass–glass and glass–metal assemblies. Various bonding methods are available, such as optical contacting, epoxy bonding, and hydroxide catalysis bonding. In this paper, the shear strength of hydroxide catalysis bonds is explored. The influences of: (1) the amount of aqueous bonding solution; (2) concentration of the bonding solution; (3) curing time; and (4) curing temperature on bond strength are investigated. Experimental results are presented for glass–glass and glass–aluminum assemblies. Comparison is also made to bond strength and thickness for a commercially available optical cement.

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1. Introduction

The ability to generate high quality bonds at glass–glass and glass–metal interfaces is critical for complex optical assemblies used in interferometry, microscopy, vacuum, defense, and space applications. Specific examples include displacement measuring interferometers for lithographic steppers used in the semiconductor industry, laser targeting systems for precision delivery of laser-guided munitions, and laser range finders. Both glass–glass and glass–metal bonds are necessary in these cases. The interfaces require high alignment accuracy, mechanical stability, the ability to be located in a vacuum environment, and low transmitted wavefront distortion.

Well-known bonding methods include: optical contacting, diffusion bonding, frit bonding, adhesive bonding, and mechanical fastening [1]. An alternative to these traditional bonding methods is hydroxide catalysis bonding (HCB). HCB was first described by Gwo [2–4] to join the fused silica components which formed the star-tracking space telescope used in the Gravity Probe B space experiment [4]. HCB achieves bonding at room temperature between various materials if a

silicate-like network can be formed between the surfaces. Example materials include silica, Zerodur, fused silica, ultra-low expansion glass, granite, and other materials that can be oxidized on their surfaces (including metals such as aluminum and titanium).

In this study, the mechanical strength of hydroxide catalysis bonds is examined. The influences of: (1) amount of aqueous bonding solution; (2) concentration of the bonding solution; (3) curing time; and (4) curing temperature are investigated. The paper is organized as follows. Section 2 provides background information on the HCB process. Section 3 describes the experimental setup, surface flatness measurements for glass and aluminum samples, and the bonding procedure. Section 4 provides experimental results and conclusions are presented in Section 5.

2. HCB background

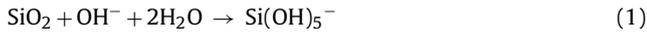
In HCB, the materials are bonded using an aqueous alkaline bonding solution, such as sodium hydroxide (NaOH), potassium hydroxide (KOH), or sodium silicate (Na_2SiO_3) dissolved in de-ionized (DI) water. The chemical process of hydroxide catalysis bonding consists of three steps: (a) hydration and etching; (b) polymerization; and (c) dehydration. A description of these three steps follows.

* Corresponding author. Tel.: +1 704 687 8421.

E-mail address: tony.schmitz@unc.edu (T.L. Schmitz).

(a) Hydration and etching

The hydroxide (OH⁻) ions in the bonding solution act as a catalyst and etch the silica surfaces in contact. This causes the liberation of silicate ions.

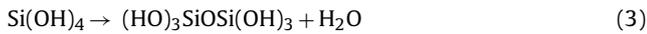


(b) Polymerization

As the active number of OH⁻ ions reduces during the hydration process, the pH of the solution decreases. If the pH is less than 11, the silicate ions dissociate:



and siloxane chains and water are formed. The bond is achieved via the siloxane chains.



(c) Dehydration

Water generated from the polymerization process migrates or evaporates during curing.

Several follow-on efforts to Gwo's initial work have further explored the HCB process. For example, Preston et al. [5] studied the mechanical strength of BK7–BK7 and silicon carbide (SiC)–BK7 bonds, while van Veggel et al. [6,7] tested SiC–SiC and silicon (Si)–Si bonds. Elliffe et al. [8] reported mechanical strength variations based on different types of bonding solution and concentrations of hydroxide ions for various materials. Reid et al. [9] explored the influence of temperature and hydroxide concentration on the settling time (i.e., the time required after alkaline solution application before the assembly can be safely removed from the fixture for curing).

3. Experimental setup

The materials used in this study were: (1) water white, low iron glass microscope slides; (2) BK7; and (3) aluminum. The microscope slides were selected for their low cost in studying a large variety of bonding conditions with a sufficient number of tests to establish statistical variation. BK7 was selected because it is a common optical material and aluminum was chosen because it is often used as a mounting base for optical assemblies. The sample preparation is described in the following paragraphs.

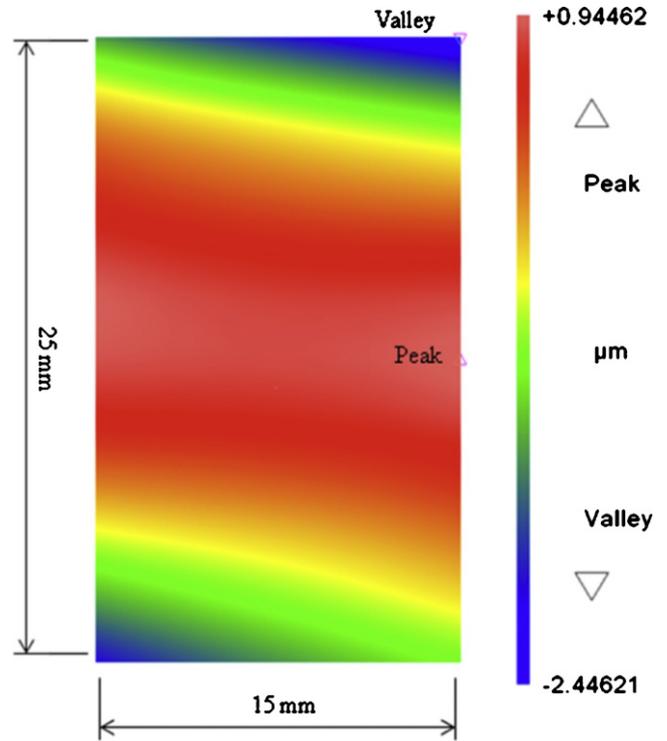


Fig. 1. Example height map for glass slide sample. The '∇' symbol indicates the valley, while the 'Δ' symbol shows the peak. The PV flatness is 3.39 μm over the sample size of 15 mm × 25 mm.

The 25 mm × 75 mm × 1 mm thick glass slides were cut into 15 mm × 25 mm sections using a dicing saw. The peak-to-valley (PV) flatness for several samples was measured using a scanning white light interferometer (SWLI); typical values were on the order of 3 μm. An example height map is provided in Fig. 1. The map peak (Δ) is located at the center of the right edge, while the valley (∇) is in the right upper corner. Fig. 2 shows a surface profile along the sample diagonal.

The manufacturer-specified PV flatness for the BK7 samples (12.5 mm × 12.5 mm × 3 mm thick) was λ/4 (158 nm at a wavelength of 633 nm). The SWLI measurement result shown in Fig. 3

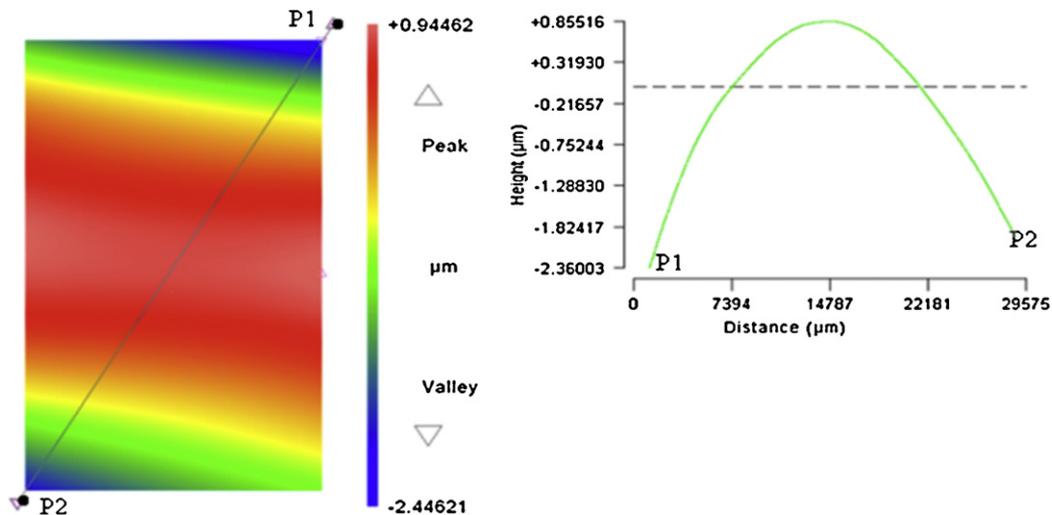


Fig. 2. Surface profile between points P1 and P2 for a glass slide sample.

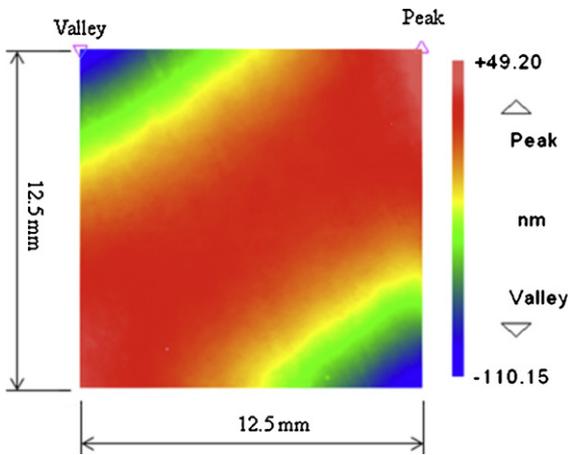


Fig. 3. Example height map for BK7 sample. The PV flatness is 159 nm over the sample size of 12.5 mm × 12.5 mm.

(PV = 159 nm) agrees well with the manufacturer's specification. A surface profile is provided in Fig. 4.

A single-side polished 5052 aluminum sheet (300 mm × 300 mm × 1 mm thick) was cut into 15 mm × 25 mm sections using a picosecond micro-machining laser system (Oxford Lasers, Inc., J-355 PS System). Fig. 5 displays a typical height map. The PV flatness is 6.9 μm. A surface profile is provided in Fig. 6.

The samples were cleaned in an ethyl alcohol ultrasonic bath for 10 min. The remaining alcohol on the surface was removed using an optical wipe and the surface was observed using a magnifier with a high intensity light source. Any remaining particles were removed using a wet alcohol wipe. Room temperature bonding was performed in a class 100 laminar flow clean cube to avoid including any airborne particles in the bond interface, which could degrade the bonding strength; see Fig. 7.

Fig. 8 depicts the bonding process. As seen, a jig was used to align the top and bottom samples with a bonding area of 307.5 mm² (15 mm × 20.5 mm). The bonding solution used in this experiment was sodium silicate solution, containing 14% sodium hydroxide (NaOH) and 27% silicon dioxide (SiO₂), dissolved in DI water. The bonding solution was dispensed on the top surface of the bottom sample using a micropipette as shown in Fig. 8. The second sample was then placed on top of the first sample. Light pressure was applied to the top sample to uniformly spread the solution. The bonded pieces were left to settle in the jig for 5 min [9] and then moved to another location in the clean cube for curing.

Fig. 9 shows the axial load frame test setup used to measure the shear strength of the bond interface. A small vise was used to

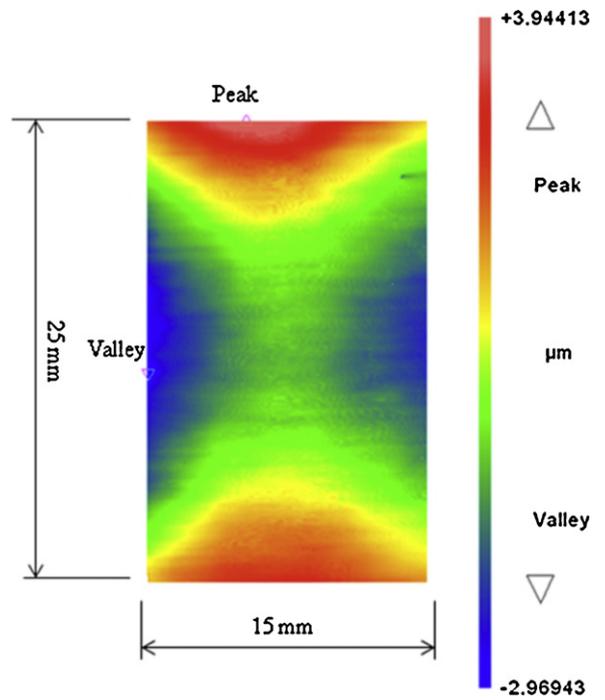


Fig. 5. Example height map for aluminum sample (polished side). The PV flatness is 6.9 μm over the sample size of 15 mm × 25 mm.

vertically support the bonded sample so that the force axis was parallel to the bonding interface. Note that the vise did not restrict vertical motion of the assembly, but was simply used as an alignment tool. After an assembly was loaded on the lower crosshead, a downward force was applied by the upper crosshead with a speed of 10 mm/min until the sample bond was broken.

4. Experimental results

This section presents experimental shear strength results for glass–glass, BK7–BK7, and aluminum–glass bonds under various bonding conditions. For the glass–glass bond, the amount and concentration of the bonding solution and curing time/temperature were varied to determine the maximum achievable strength. In addition, a commercially available optical cement was used to bond glass samples for comparison to the HCB results. Each glass–glass test set included 10 bonded samples to determine the mean strength and standard deviation; the aluminum–glass tests used five samples for each set and seven samples were used for the BK7–BK7 tests.

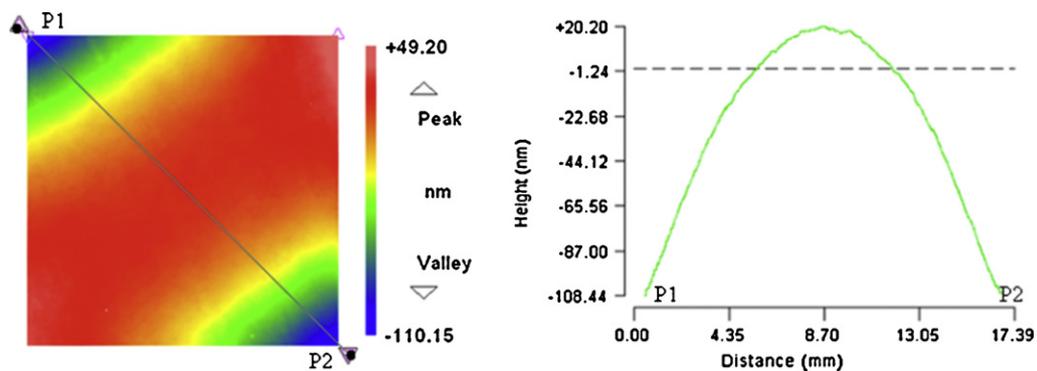


Fig. 4. Surface profile between points P1 and P2 for a BK7 sample.

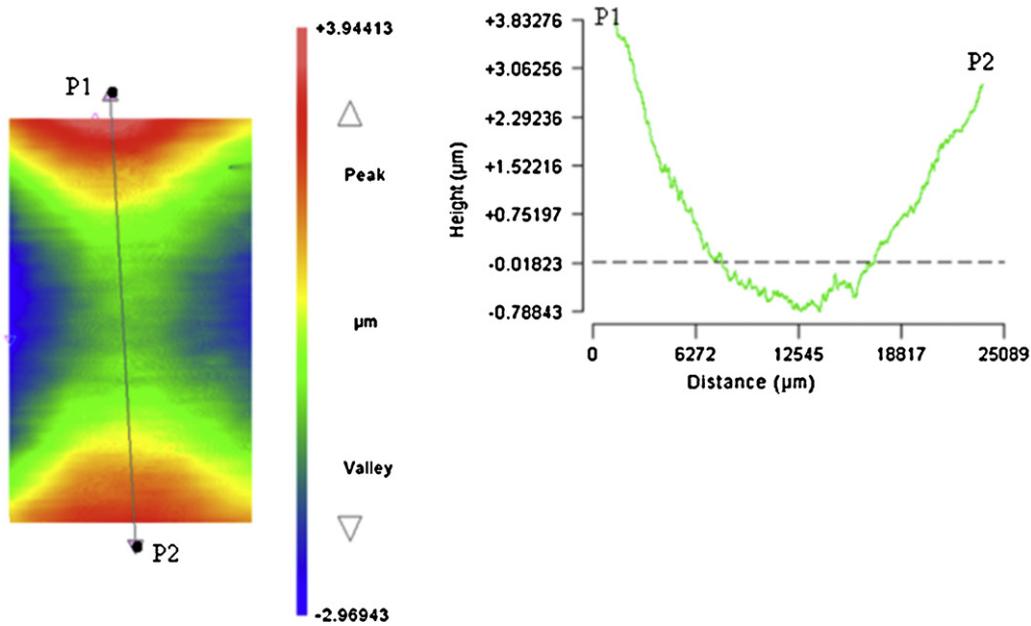


Fig. 6. Surface profile between points P1 and P2 for an aluminum sample (polished side).



Fig. 7. All samples were bonded in a class 100 laminar flow clean cube to avoid including airborne particles in the bonded interface.

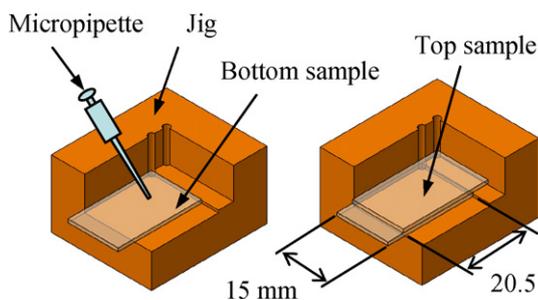


Fig. 8. The samples were bonded using an alignment jig: (left) the bonding solution was dispensed on the bottom sample; and (right) the top sample was placed on top of the bottom sample.

4.1. Glass–glass (HCB)

The shear strength for the glass–glass HCB assemblies was measured using the axial load frame shown in Fig. 9. The shear strength profiles for 10 bonded samples using 2.0 μl of solution (over the 307.5 mm² bonding area) with a volume ratio of 1:4 (sodium silicate solution to DI water) are shown in Fig. 10. The samples were cured for 48 h at room temperature for this test. The mean breaking shear strength was 3.5 MPa with a standard deviation of 0.5 MPa.

To evaluate the effect of the amount of solution applied to the bonding area, the glass samples were bonded with five different solution amounts {0.5, 1, 2, 3, and 5 μl }, over the bonding area of 307.5 mm². The volume ratio was 1:4 and the curing time was 48 h. Fig. 11 shows the mean breaking shear strengths (circles) and one standard deviations error bars for each solution amount. Although the mean breaking strength for 1 μl is slightly lower, the error bars from all tests overlap. Therefore, the test results indicate that the bond shear strength is not strongly dependent on the amount of sodium silicate solution for the range tested here.

In Fig. 12, the mean breaking strengths for a range of sodium silicate solution to DI water concentrations {1:2, 1:4, 1:6, 1:10, and 1:50} were determined. For these tests, 2.0 μl of the bonding

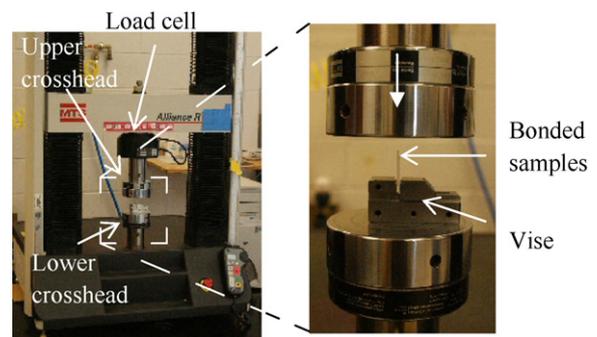


Fig. 9. Shear strength test setup using an axial load frame with a 30 kN load cell. A vise was used to support the bonded samples between the upper and lower crossheads.

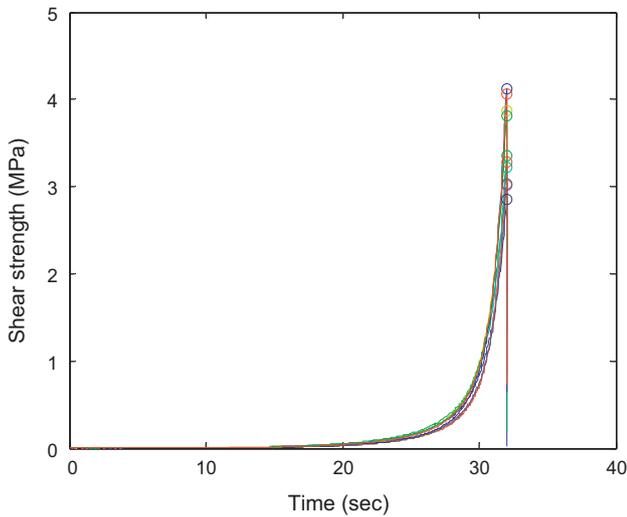


Fig. 10. Shear strength profiles for glass-glass bonding tests with 2.0 μl of 1:4 volume ratio (sodium silicate solution to DI water) solution. The samples were cured for 48 h at room temperature.

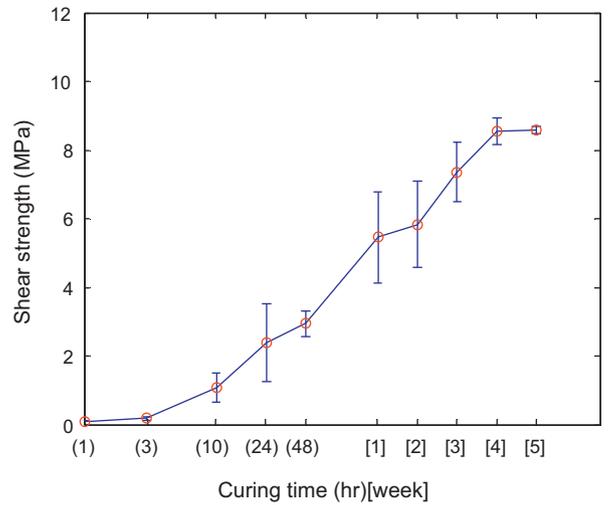


Fig. 13. Breaking shear strength for various curing times. Maximum strength was reached after four weeks of curing at room temperature (2.0 μl of solution with a 1:4 volume ratio).

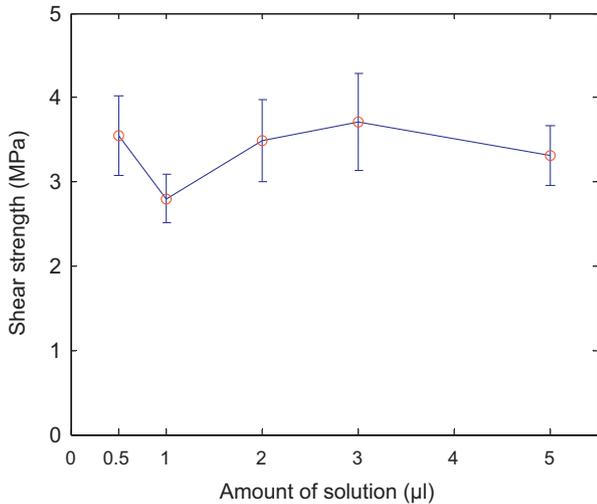


Fig. 11. Breaking shear strength for various bonding solution amounts. The mean values and one standard deviation error bars are provided.

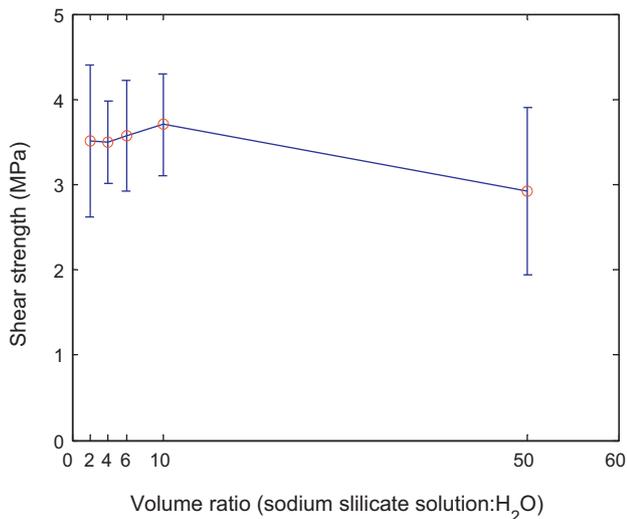


Fig. 12. Breaking shear strengths for various concentrations of the bonding solution (volume ratio of sodium silicate solution to DI water).

solution was applied and the samples were cured for 48 h. No significant strength difference is observed.

The influence of curing time on bond strength was also tested. A solution amount of 2.0 μl and 1:4 volume ratio were used; these values were arbitrarily selected since the shear strength was shown to be insensitive to the amount and concentration of the bonding solution. Fig. 13 shows the breaking shear strengths for curing times ranging from 1 h to 5 weeks at room temperature. The bond strength increased with curing time until four weeks. This contradicts the results presented in Ref. [5] that showed constant shear strength for curing times from 18 h to 11 days. However, it supports Ref. [2], where it was reported that maximum strength was achieved only after four weeks of curing time.

Fig. 14 shows glass pieces that were collected after breaking tests at each curing time. The samples cured for less than 24 h were separated without breaking, while longer curing times produced samples that were broken into several small pieces. The size of broken pieces reduced with curing time, which follows the increase in strength observed for longer curing times. The 4- and 5-week samples are displayed together because few pieces remained after breaking; most were too small to collect.

It is seen in Fig. 13 that the distribution in shear strength for samples cured over 24 h was larger than for the other curing times. This is investigated further in Fig. 15. The mean shear strength (3.5 MPa) for six out of 10 samples (group A in Fig. 15) was about three times higher than the strength (1.1 MPa) of the remaining four samples (group B in Fig. 15). The group A samples were broken during the shear test, while the group B samples were separated without breaking. It was concluded that a curing time of 24 h is the transition time between a permanent and “separable” bond for 2.0 μl of sodium silicate solution with a 1:4 volume ratio.

The thickness of the bonding interface for a 4-week curing time was measured using the SWLI. The bonded sample was sectioned through its center to reveal the bonding interface and then polished; see Fig. 16(A). The interface could not be observed for the 1 μm lateral resolution of the SWLI measurement; see Fig. 16(B). This indicates that the interface thickness was less than 1.0 μm .

It was observed that the full strength for the glass-glass bonding was obtained after a curing time of 4 weeks at room temperature. However, this time is too long for most commercial applications. Therefore, it was desired to reduce the curing period. It is known that water migrates or evaporates out of the bond during curing

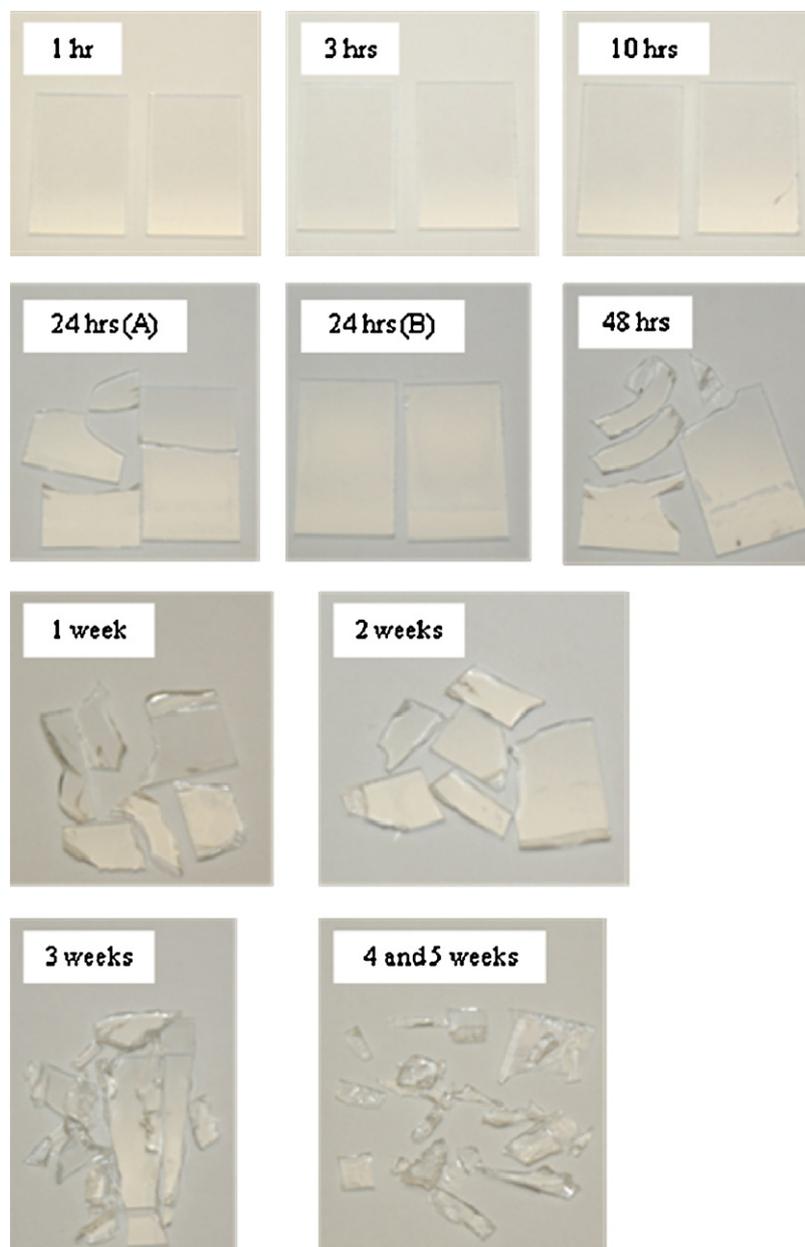


Fig. 14. Photographs of the broken and separated pieces of the bonded samples for various curing times. For the 24-h curing time, two results are shown: (A) broken (higher strength); and (B) separated (lower strength).

(the final step in the HCB process). In this study, two methods were used to aid in this dehydration step: (1) oven; and (2) microwave heating.

Fig. 17 shows strength testing results for oven curing at various temperatures. Again, $2.0 \mu\text{l}$ of 1:4 volume ratio sodium silicate solution was used. The glass pieces were bonded and placed in the clean cube for 24 h at room temperature. The bonded samples were then moved into the oven. At each temperature ($60\text{--}100^\circ\text{C}$), the samples were cured from 1 h to 7 h. Ten assemblies were used for each test set. As shown in the figure, the mean shear strengths of the samples cured at 100°C for all curing times and at 80°C for times of 3 h and higher exceeded the maximum strength (solid horizontal line) obtained from the room temperature cure. These tests provided two important results: (1) the curing time for maximum strength can be significantly reduced from 4 weeks at room temperature to 24 h at room temperature followed by 1 h at 100°C ; and (2) the shear strength of the samples cured at elevated

temperatures exceeded the strength of the samples cured only at room temperature.

The shear strength results for microwave¹ curing tests are provided in Fig. 18. The bonding conditions were the same as for the oven tests. The samples were heated for various time periods {0.5, 1, 3, and 5 min}, after a 24-h cure at room temperature. The maximum shear strength (6.4 MPa) for the microwave tests did not reach the strength (8.6 MPa) obtained from the 4-week cure at room temperature. The strength did approximately double between 1 min and 3 min in the microwave, however.

Since the maximum shear strength for HCB using a sodium silicate solution was determined (4 weeks at room temperature or 24 h at room temperature followed by 1 h at 100°C), it was of interest to compare this result with the compressive breaking force required

¹ 0.92 kW power at 120 VAC/60 Hz.

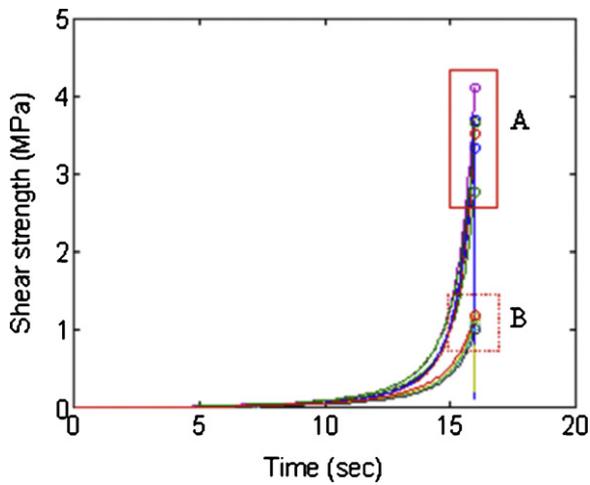


Fig. 15. Shear strength profiles for samples with a 24-h curing time. Two groups (A and B) are observed.

for a single glass sample (no bond). Fig. 19 shows the test setup, where a single glass sample was supported by the vise. The size of the piece was 15 mm × 25 mm with a 1 mm thickness. Fig. 20 shows the force profiles for six samples; the mean breaking force was 5002 N with a standard deviation of 286 N. For comparison purposes, the force profiles for six bonded samples cured for 4 and 5 weeks are provided in Fig. 21 (same vertical scale). The mean breaking force and standard deviation are 2635 N and 80 N, respectively. To enable a convenient comparison, force data is presented in the figure (since there was no bond for the single sample, the shear strength cannot be reported). This data shows that the full force to break the bond using HCB is about half that of the bulk material.

4.2. Glass–glass (optical cement)

A common optical bonding technique is to use an adhesive to join the assembly components at room temperature. The adhesive bonding strength, however, is often sensitive to temperature changes and the chemical environment. Additionally, the

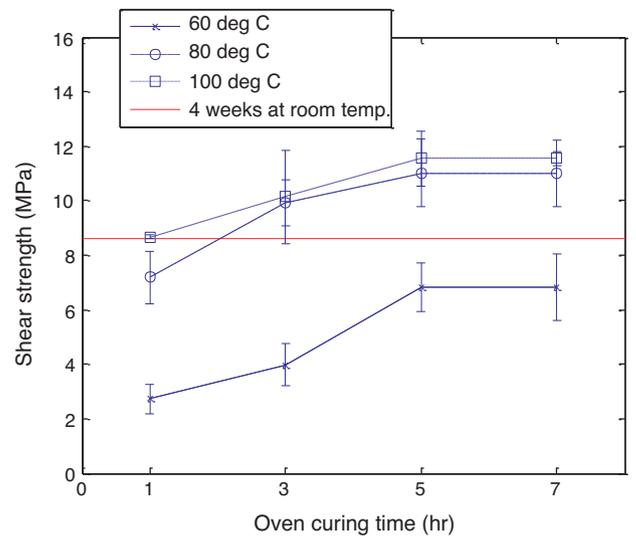


Fig. 17. Variation in shear strengths for oven curing. At a temperature of 100 °C, all bonding strengths exceeded the strength obtained from a 4-week cure at room temperature.

bonding interface is relatively thick, which may lead a refractive index mismatch. In this section, the bond strength and thickness for glass–glass bonds using a commercially available optical cement are presented and compared to HCB results.

A two-component optical cement (Summers Optical, M-62) was used. According to the manufacturer, curing time should be varied with the volume ratio between cement and catalyst. The catalyst was supplied in a controlled-drop bottle. A ratio of three drops of catalyst per 3 ml of cement was used. This ratio required either: (1) an 18 h pre-cure at room temperature followed by a four day (96 h) full-cure; or (2) a 30 min pre-cure at 70 °C followed by a 1.5-h full-cure. In this study, the curing cycle was: (1) 30 min at room temperature; (2) 2 h at 70 °C; and (3) 72 h at room temperature.

The microscope glass slides were again used as materials to be bonded. The bond geometry and breaking process were the same as for the HCB tests. Fig. 22 shows the shear strength profiles for

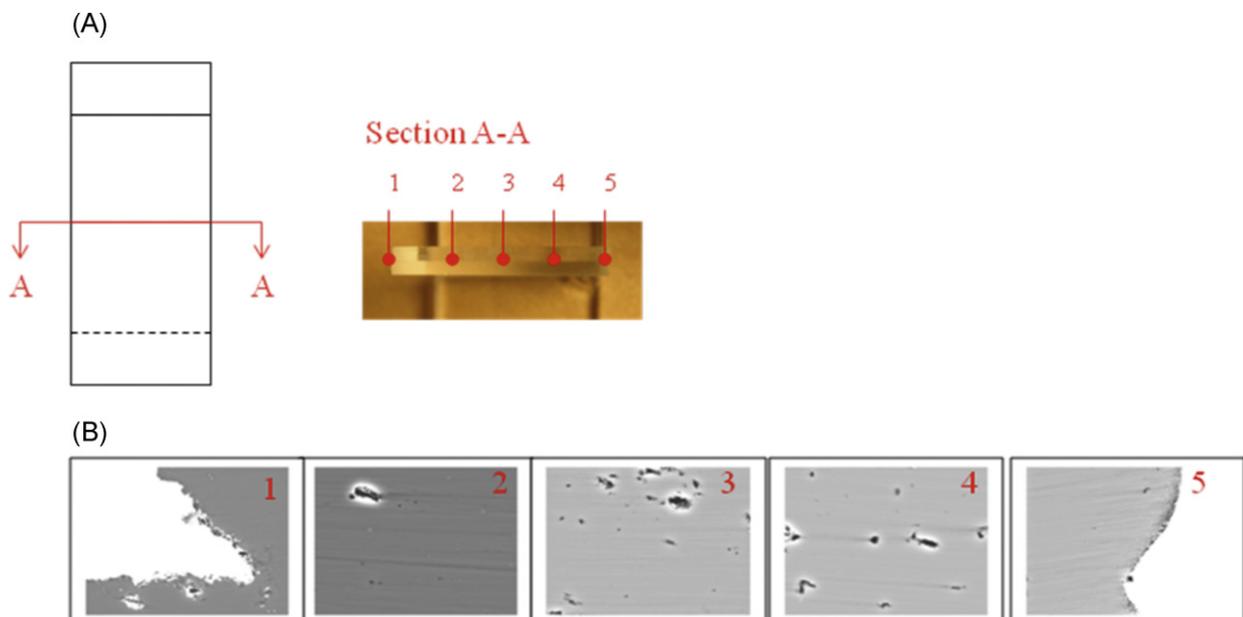


Fig. 16. HCB interface for glass–glass assembly: (A) the bonded sample was cut at the center to reveal the bonding interface; and (B) the sectioned surface was measured at several locations. The bonding thickness was less than the SWLI lateral resolution.

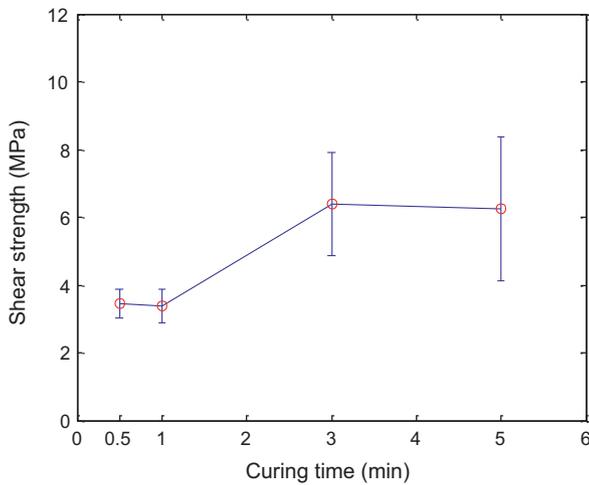


Fig. 18. Shear strengths for various curing times in a microwave. The maximum strength (6.4 MPa) obtained from these tests was less than that (11.5 MPa) for the oven-cured samples (100 °C).

the five samples. The mean strength is 8.5 MPa which is close to the maximum strength (8.6 MPa) obtained for the HCB samples cured at room temperature for 4 weeks.

SWLI images of the bond thickness for the optical cement are presented in Fig. 23. The same 1 μm lateral resolution as for the HCB thickness measurement was available. Fig. 23(B) shows the bonding interface at each location labeled in Fig. 23(A). A uniform bond thickness is observed. The mean bonding thickness of the five locations is approximately 40 μm as shown in Fig. 24(C). This bond thickness ($\sim 40 \mu\text{m}$) is much higher than for HCB ($< 1 \mu\text{m}$), while the bond strengths are comparable.

4.3. BK7–BK7 (HCB)

In this section, shear strength results for BK7–BK7 assemblies using HCB are presented. The bonding and curing conditions matched the glass–glass HCB bonding experiment reported in Section 4.1. The amount of the bonding solution was 0.65 μl over a bonding area of 100 mm^2 ; this provided the same amount per unit area as for the glass–glass bonding (2.0 μl per 307.5 mm^2). The volume ratio (sodium silicate solution to DI water) was 1:4. The bonded samples were cured at room temperature for 24 h in the clean cube. Then, five out of seven samples were placed in an oven for 1 h at 100 °C, while the remaining samples were cured for 7 h at

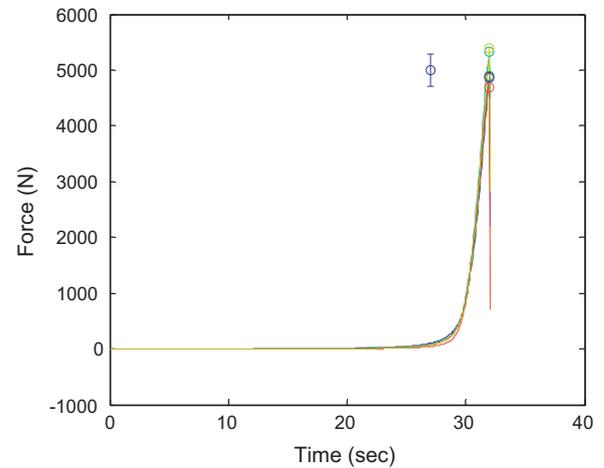


Fig. 20. Force profiles for breaking a single glass sample (25 mm \times 15 mm \times 1 mm thick). The mean breaking force was 5002 N with a standard deviation of 286 N.

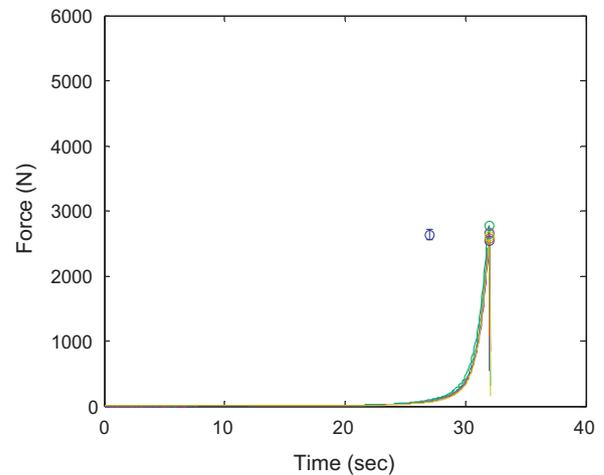


Fig. 21. Force profiles for bonded samples with four and five weeks curing time at room temperature. The mean breaking force is 2635 N with a standard deviation of 80 N.

100 °C. Due to the limited numbers of the samples, only the 1 h and 7 h curing times at 100 °C were tested. The shear strength values for the BK7–BK7 assemblies are shown in Fig. 24. The mean bonding strength (31.8 MPa) for the 7 h cure is slightly higher than the mean

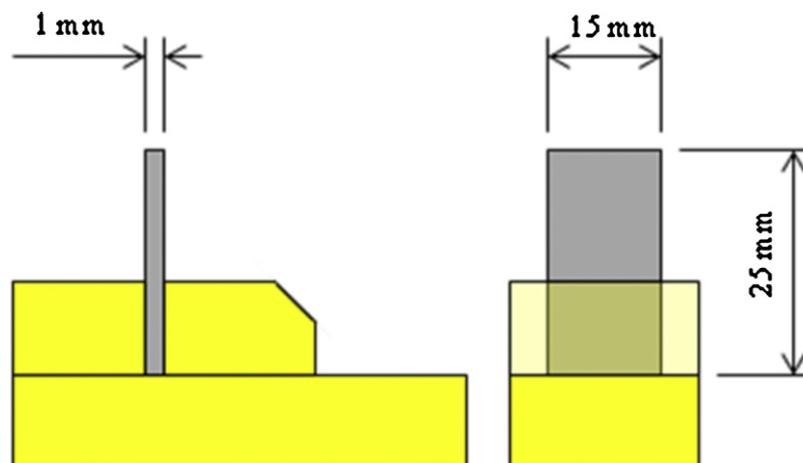


Fig. 19. Setup for breaking a single glass sample supported in a vise. The axial load frame was again used to apply and measure the breaking force.

strength (28.6 MPa) for 1 h cure. The strength result agrees very well with the result for fused silica–fused silica bonding strength (approximately 30 MPa) reported by Gwo [4]. In comparison with the glass–glass bonding, the BK7–BK7 bonding strength is about three times higher. This result was expected because the BK7 surface flatness was much better.

4.4. Aluminum–glass (HCB)

As noted, the native oxide layer on metals enables a hydroxide catalysis bond to be formed with a glass sample. In this case, the glass surface forms the silicate-like network, while the metal surface generates a quasi two-dimensional layer of surface hydroxyl groups [2]. For the aluminum samples tested in this work, the surface was passivated in air by the natural formation of an aluminum oxide, Al_2O_3 , layer. The shear strength of the aluminum–glass bond using this native oxide layer was investigated. From the glass–glass bonding experiment, it was assumed that variation of the bonding solution amount and concentration was not critical. Therefore, only curing time variation tests were conducted. In order to determine the shear strength for aluminum–glass bonds, the samples were cured at room temperature for a range of times from 24 h to

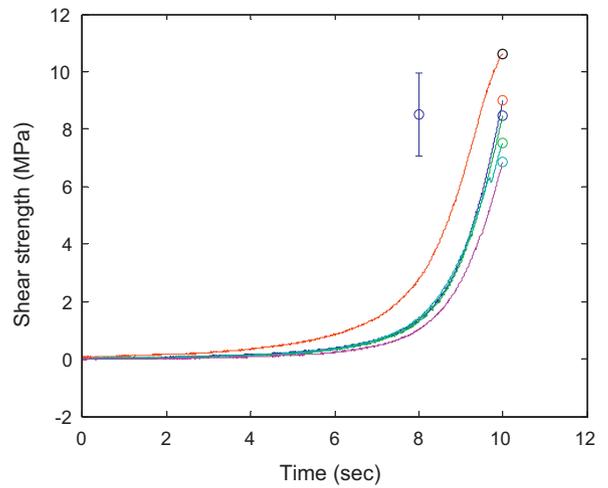


Fig. 22. Shear strength profiles for glass–glass bonding using a commercially available optical cement. The mean shear strength (8.5 MPa) was comparable with the maximum strength (8.6 MPa) obtained by HCB for the 4-week, room temperature cure.

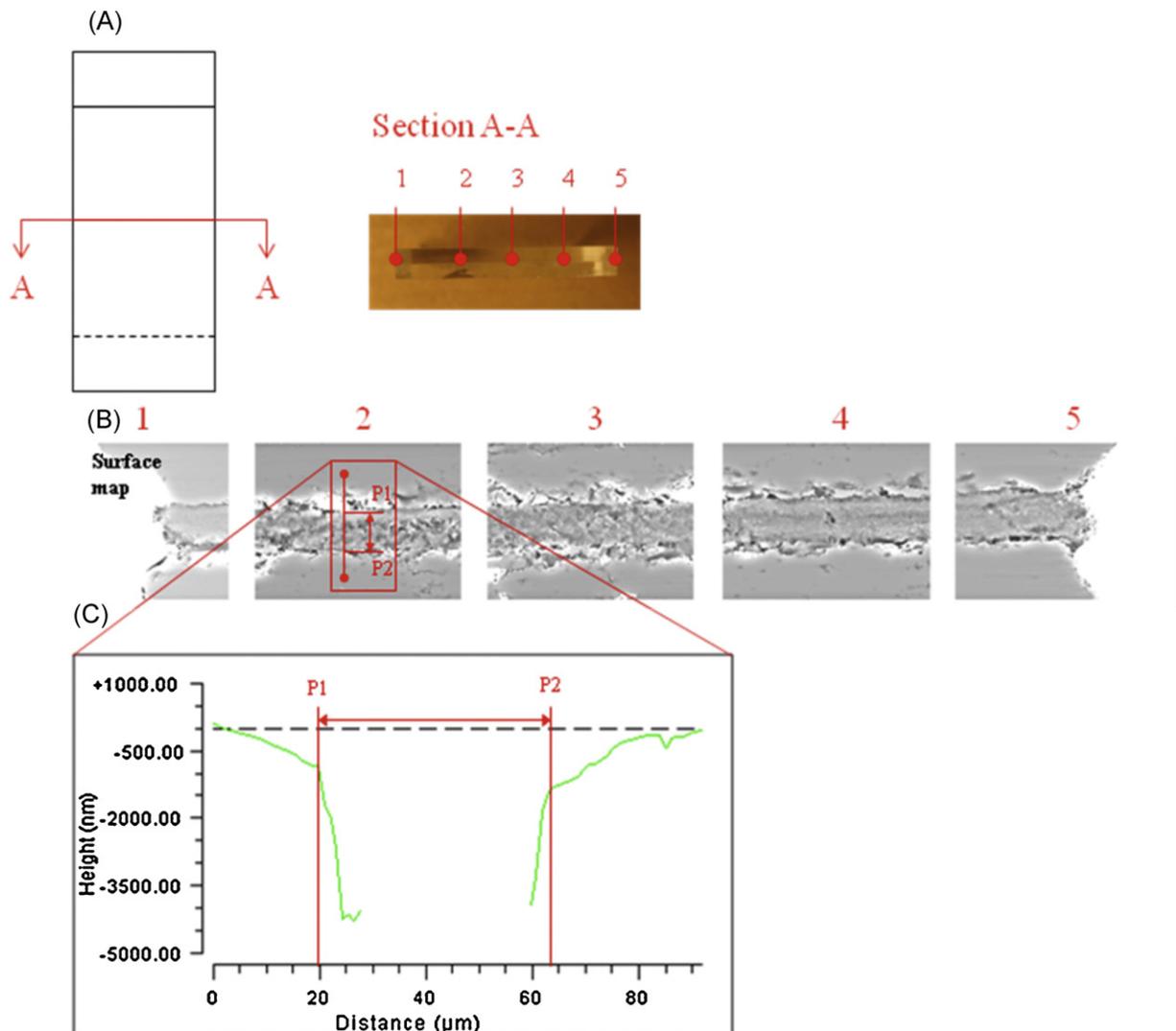


Fig. 23. Optical cement bond thickness measurements: (A) cross-section of the adhesive bond; (B) SWLI images of the bonding interface at several locations; and (C) thickness measurement between points P1 and P2 at location 2. The mean thickness for the five measurements is approximately 40 μm .

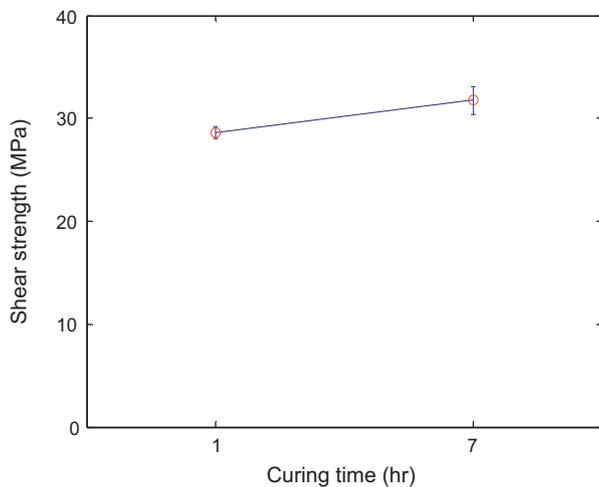


Fig. 24. Shear strengths for BK7–BK7 HCB tests. The samples were cured at room temperature for 24 h and then placed in an oven for 1 h/7 h at 100 °C.

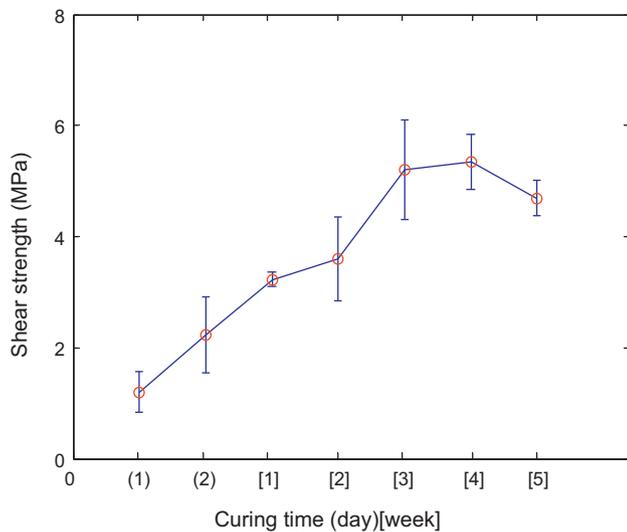


Fig. 25. Shear strength for HCB aluminum–glass assemblies at various curing times. Maximum strength was obtained after a 3-week cure. The strength is 5.3 MPa, which is less than the HCB glass–glass results due to the large figure mismatch between the aluminum and glass samples.

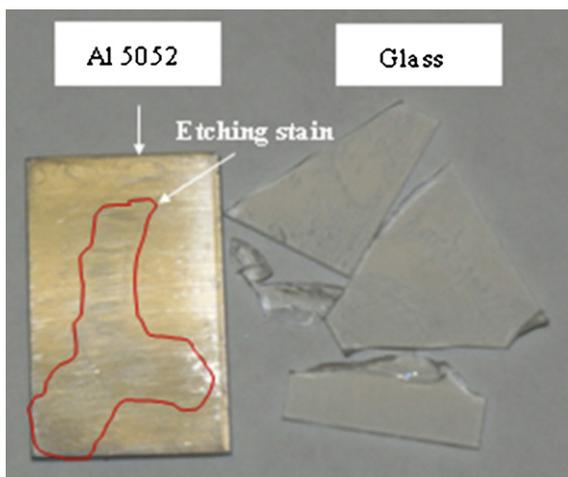


Fig. 26. Photograph of aluminum sample and glass pieces after breaking an aluminum–glass bond. The etching “stain” from the bonding solution indicated that bonding was completed only over a portion of the aluminum–glass interface.

5 weeks. Sodium silicate solution was again used as the bonding solution with an amount of 2.0 μl (307.5 mm^2 bonding area) and a volume ratio of 1:4. Five samples were bonded for each curing time.

Fig. 25 shows the shear strength variation with curing time for the aluminum–glass bonds. Maximum strength was obtained after 3 weeks. Although this strength was about half the strength of the glass–glass bonds (when cured at 100 °C for 7 h), it is reasonable given the large PV flatness value for the aluminum samples. Fig. 26 shows an aluminum–glass bonding assembly after breaking. The glass was broken into several pieces and an etching ‘stain’ from the bonding solution indicated that only a portion of the surface between the aluminum and glass was bonded due to the surface figure mismatch.

5. Conclusions

In this work, the shear strength for glass–glass (water white, low iron glass microscope slides), BK7–BK7, and aluminum–glass bonds produced using hydroxide catalysis bonding was evaluated. It was determined that the solution amount and concentration did not have a significant effect on bond strength for the sodium silicate solution tested here. However, it was found that the bond strength increased with extended curing times and elevated curing temperatures. The maximum bonding strength for the glass–glass bond was achieved after 4 weeks at room temperature. The same strength was obtained for a curing time of 24 h at room temperature followed by 1 h at 100 °C. This shear strength level was comparable to the results for a two-component optical cement, although the bonding interface of the optical cement was much thicker. Results were also presented for BK7–BK7 hydroxide catalysis bonding. Due to the improved surface flatness for the BK7 samples, the bonding strength increased by approximately three times. Aluminum–glass bonding was also completed. The strength levels were lower due to the relative non-flatness (approximately 7 μm peak-to-valley) of the aluminum samples, where it was also observed that bonding occurred only over a portion of the bond area. With comparable flatness, similar strength levels to the glass–glass bonds should be possible.

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