THE INFLUENCE OF CURING TIME ON HYDROXIDE CATALYSIS BONDING STRENGTH

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INTRODUCTION

Glass-glass and glass-metal bonds are required in many high-precision applications, including displacement measuring interferometers, laser targeting systems, and laser range finders. Common methods include optical contacting and epoxy bonding [1]. Optical contacting, a room-temperature process, is sensitive to surface contamination and can yield unreliable strength. In epoxy bonding, the thickness of interface is relatively large, which can be a concern due to refractive index mismatching between the interface and the substrates. In addition, the mechanical strength of epoxy bonding varies with temperature and chemical environment.

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]. In this previous work, it was shown that curing time strongly affects the final bonding strength. The purpose of this paper is to determine the time required to reach the maximum mechanical strength using various approaches.

HYDROXIDE CATALYSIS BONDING

HCB is the process by which a hydroxide, such as sodium or potassium hydroxide, catalyzes the surface to be bonded by hydration and dehydration. The HCB technique consists of three steps: 1) hydration and etching; 2) polymerization; and 3) dehydration. The HCB technique enables bonding between a variety of materials if a silicate-like network can be formed at the surfaces. This is the case for most oxide-containing materials, including silica, Zerodur, fused silica, ULE glass, and granite, as well as metals with an oxidized surface.

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).

EXPERIMENTAL PROCEDURE

The materials used in the experiments were microscope glass slides (water-white, low iron glass) and aluminum samples. 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. 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). Typical PV flatness values for the aluminum samples were approximately 7 μm.

The glass and aluminum pieces were cleaned in an ethyl alcohol ultrasonic bath for 10 minutes and then removed. 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 ethyl alcohol wipe. Bonding was performed in a class 100 laminate flow clean cube at room temperature to avoid any airborne particles which could degrade the bonding strength. Figure 1 depicts the bonding process using a jig, where the pieces are aligned to have a bonding area of 307.5 mm^2 (15 mm × 20.5 mm). A sodium silicate bonding solution was used in this study; the solution contained 14% sodium hydroxide (NaOH) and 27% silicon dioxide (SiO_2) dissolved

in de-ionized (DI) water. The bonding solution was dispensed on the top surface of the bottom piece using a micropipette as shown in Fig. 1(A). The second piece was then gently placed on the first piece. See Fig. 1(B). Light pressure was applied to the top piece to uniformly spread the solution. The bonded pieces were left to settle in the jig for about 5 minutes [9] and then moved to another location in the clean cube for curing.

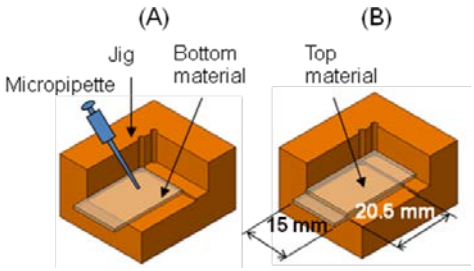

FIGURE 1. The pieces were bonded using a jig for alignment.

Figure 2 shows the test setup for measuring the shear strength of the bonding interface; an axial load frame was used to apply the shear force. A small vise was used to vertically support the bonded sample so that the force axis was parallel to the bonding interface. After a sample 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.

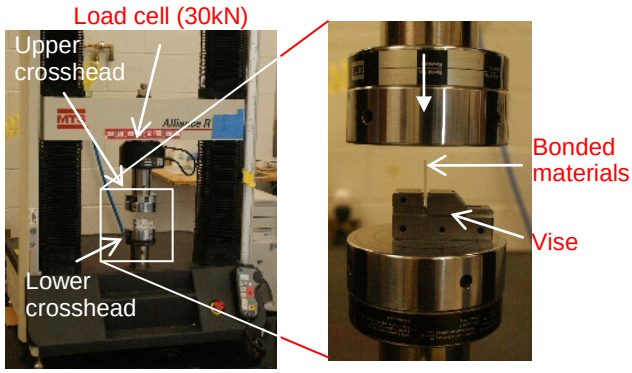

FIGURE 2. Shear strength test setup using an axial load frame with a 30 kN load cell.

RESULTS

As noteed, the mechanical strength of the hydroxide catalysis bond depends on curing time. In this section, shear strength results for glass-glass assemblies under various curing times are presented. In an effort to reduce the curing time, the samples were placed in an oven with elevated temperature to aid the water evaporation. In addition, a commercially-available optical cement was used to bond glass samples for comparison to the HCB results. Since metal components often serve as mounting surfaces and structures for optical bonding applications, the mechanical strength was also tested for aluminum-glass interfaces using the HCB technique.

Glass-glass (HCB)

The influence of curing time on bond strength was tested. A solution amount of 2.0 µl and 1:4 volume ratio were used. Figure 3 shows the breaking shear strengths for curing times ranging from 1 hr to 5 weeks at room temperature. The bond strength increased with curing time until four weeks. This contradicts the results presented in reference [5] that showed constant shear strength for curing times from 18 hrs to 11 days. However, it supports reference [2], where it was reported that maximum strength was achieved only after 4 weeks of curing time.

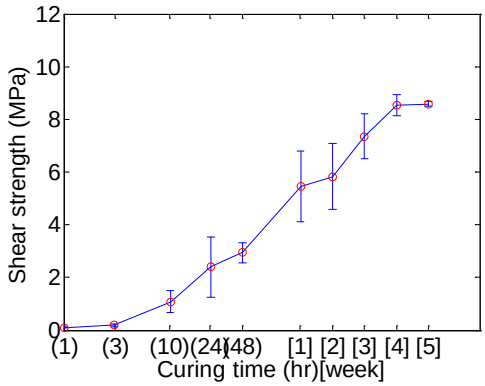

FIGURE 3. Breaking shear strength for various curing times.

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. The interface could not be observed for the 1 µm lateral resolution of the SWLI measurement. This indicates that the interface thickness was less than 1 µm.

It was observed that the maximum 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 (the final step in the HCB process). In this study, an oven was used to aid in this dehydration step.

Figure 4 shows strength testing results for oven curing at various temperatures. Again, 2.0 µl of 1:4 volume ratio sodium silicate solution was used. The glass pieces were bonded and placed in the clean cube for 24 hrs at room temperature. The bonded samples were then moved into the oven. At each temperature (60 deg C to 100 deg C), the samples were cured from 1 hr to 7 hrs. Ten assemblies were used for each test set. As shown in the figure, the mean shear strengths of the samples cured at 100 deg C for all curing times and at 80 deg C for times of 3 hrs 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 hrs at room temperature followed by 1 hr at 100 deg C; and 2) the shear strength of the samples cured at elevated temperatures exceeded the strength of the samples cured only at room temperature.

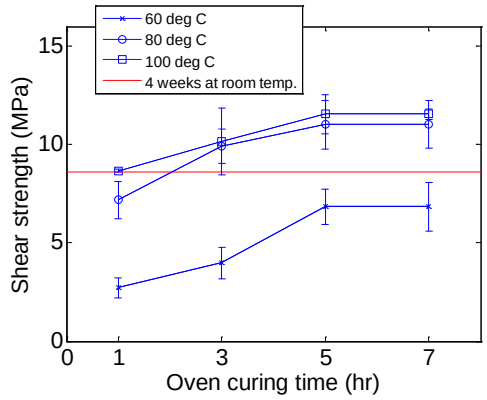

FIGURE 4. Variation in shear strengths for oven curing.

Glass-glass (optical cement)

A commercially-available two-component optical cement (Summers Optical, M-62) was used for glass-glass bond. The manufacturer-specified curing conditions were followed: 1) 30 minutes at room temperature; 2) two hrs at 70 deg C; and 3) 72 hrs at room temperature.

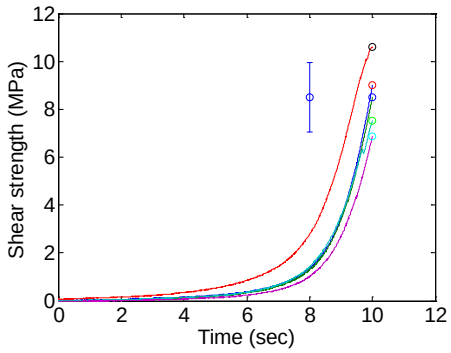

FIGURE 5. Shear strength profiles for glass-glass bonding using a commercially-available optical cement.

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. Figure 5 shows the shear strength profiles for 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.

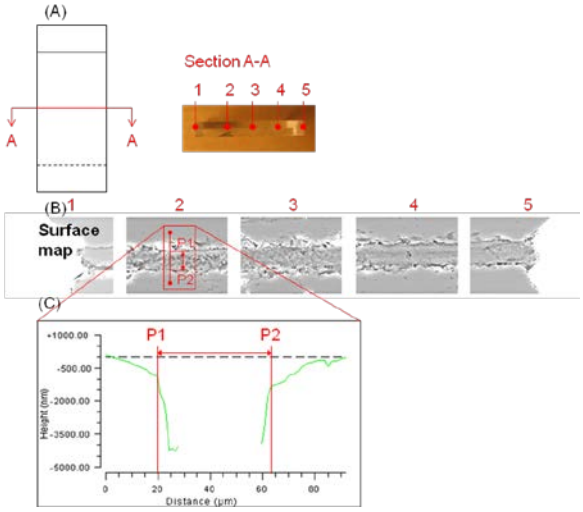

FIGURE 6. Optical cement bond thickness measurements. The mean thickness for the five measurements was approximately 40 µm.

SWLI images of the bond thickness for the optical cement are presented in Fig. 6. The same 1 µm lateral resolution as for the HCB thickness measurement was available. Figure 6(B) shows the bonding interface at each location labeled in Fig. 6(A). A uniform bond thickness is observed. The mean bonding thickness of the five locations was approximately 40 µm as shown in Fig. 6(C). This bond

thickness (~40 μm) is much higher than for HCB (<1 μm), while the bond strengths are comparable.

Aluminum-glass (HCB)

The native oxide layer on metals enables a hydroxide catalysis bond to be formed with a glass sample. Like the glass-glass bond, curing time variation tests were conducted to evaluate the maximum bond strength for aluminum-glass bond. The samples were cured at room temperature for a range of times from 24 hrs to 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.

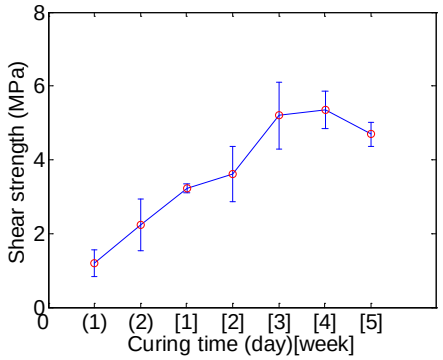

FIGURE 7. Shear strength for HCB aluminum-glass assemblies at various curing times.

Figure 7 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 deg C for 7 hrs), it is reasonable given the large PV flatness value for the aluminum samples.

CONCLUSIONS

In this work, the shear strength for glass-glass (water white, low iron glass microscope slides) and aluminum-glass bonds produced using hydroxide catalysis bonding was evaluated. It was determined 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 hrs at room temperature followed by 1 hr at 100 deg 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. 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.

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