Experimental investigation of adhesive bond strength between metal and optical glass

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ABSTRACT

Within the general astronomical community as well as at the University of California Observatories, there has been a long history of using epoxy to mount optics within instruments such as spectrometers and telescopes. The Ken & Gloria Levy Spectrometer, part of the Automated Planet Finder (APF) telescope located at Mt. Hamilton's Lick Observatory, relies on epoxy-bonded joints to attach the instrument's large cross-dispersing prism and echelle grating to its Invar space-frame structure. Design constraints dictated that these large optics each be attached at only three points, and that the bond areas be as small as possible while maintaining an adequate strength factor of safety. Previous UCO instruments, such as the Keck Telescopes' primary mirror segments and the ESI Spectrometer, used Hysol's 9313 epoxy product for this purpose. Concerns over long-term reliability of such joints led us to re-examine this issue. We empirically investigated the roles played by epoxy selection and techniques such as surface preparation and the use of a primer, in creating a robust metal-to-glass bond. Bond strength data was generated, leading us to select a previously unused epoxy, and to implement particular techniques to ensure bond quality. Most notably, we found that bond strength data as typically reported on adhesive manufacturers' datasheets was not a reliable indicator of long-term bond reliability between metal and optical glass.

Keywords: Adhesive, Bonding, Epoxy, Optics, Primer, Strength, Stress, Surface Preparation

1. INTRODUCTION

The Ken & Gloria Levy Spectrometer incorporates a determinate space-frame structure of struts and connecting nodes to position its major optical components in three-space. Each of these components attaches to the structure at only three nodal locations, a necessary and sufficient condition to accurately define their placement while helping to isolate them from external moments that could be detrimental to optical performance.

Mounting the spectrometer's two large monolithic glass optics, a 23-kg [50.7 lb] Ohara BSL7Y prism and a 56-kg [123.5 lb] Schott Zerodur® echelle grating, posed a particular design challenge since no intermediate structural carrier was intended to hold them. Instead, round metal pucks were adhesively bonded directly to them, the pucks incorporating threaded holes to accommodate bolted connections to the instrument structure. Our group has relied on bonded pucks before, to mount the Keck Telescope primary mirror segments as well as the large prism in the Keck ESI Spectrometer[1].

Placement of the three pucks on each optic was subject to a number of constraints. Since the pucks were coincident with nodes, struts from other parts of the space-frame would connect to them. Struts could not intersect the optic or interfere with the light path through the instrument. Pucks could not be located where they might cause unwanted reflections or vignetting. Finite element analysis (FEA) was carried out to confirm that the puck configuration would not result in excessive stress on the adhesive bond or the glass, as a consequence of the optic's self-weight.

The diameter of the cylindrical pucks was sized such that calculated peak joint stress would not exceed 1,380 kPa [200 psi]. This was viewed as a conservative value that was expected to offer a roughly 10X factor of safety with respect to the working strength of both the glass and the epoxy bonds. Puck material selection was driven by the desire to match its coefficient of thermal expansion (CTE) to that of its mated optic. This minimizes temperature induced differential stress between the glass and metal. In this instrument we paired Invar 36® (CTE of 1.5 ppm/°C) with the Zerodur® echelle grating (CTE of 0.05 ppm/C°), and titanium 6Al-4V (8.6 ppm/°C) with the BSL7Y (7.2 ppm/C°) prism.

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It was considered prudent to be overly conservative about bonded joint working stress given the wide range of temperatures present at Lick Observatory, where this instrument is to be sited. Large and frequent temperature excursions exercise bonded joints due to differential stresses created by the typically large CTE mismatch between substrate and adhesive.

We have recently received reports of localized stress cracking of the Keck primary mirror segments in areas immediately adjacent to bonded attachment points. Given this fact and the dynamic nature of the instrument's working environment, we decided to conduct an experimental investigation with the goal of either confirming the robustness of our traditional gluing method, or to suggest improvements in materials and techniques such that we would have a high degree of confidence in the reliability of our bonded joints.

2. METHODOLOGY

2.1 Selection of Epoxy Candidates

Many factors affect bond strength. Given the time and resources available, we limited the scope of this investigation to room temperature experiments comparing five commercially available epoxy formulations and one primer. These included Hysol 9313, with which we had previous experience, Hysol 9361, Armstrong A-12, Milbond (distributed by Summers Optical), and 3M 2216 B/A Gray. Milbond Primer was recommended by Summers Optical for use with their epoxy, but we tried it with other epoxies as well. These products were chosen not only because they were commonly used in optical assembly, but more importantly, would fully cure at room temperature. This was an important requirement, given our reluctance to expose two large, expensive optics to elevated temperatures and extra handling.

We were particularly interested in evaluating Hysol 9361. According to datasheet information, it was comparable in strength to Hysol 9313, but had an 11 degree-C higher maximum service temperature and 1/3 the modulus of elasticity. FEA predicted that the lower modulus material would be better able to absorb differential movement created by CTE mismatches between puck, optic, and adhesive, resulting in lower joint stress.

2.2 Sample Preparation

Test samples were prepared, each consisting of a cylindrical metal puck (25mm [1 inch] diameter x 25mm height) face-bonded to a glass cube. Titanium pucks were paired with BSL7Y blocks, Invar with Zerodur®. We standardized on practices that common sense and past experience had shown to be successful. A gluing fixture was used to control bondline thickness to a constant 0.25mm [.010 inches], a value that previous analysis had indicated would offer a good balance between strength and stress buildup[2]. We prefer using a gluing fixture because it does not introduce geometric discontinuities or trap foreign materials within the bondline, as happens with the commonly used technique of mixing glass microspheres into the epoxy or using nylon monofilament as a permanent spacer (Figure 1).

Figure 1 - Gluing fixture used to prepare test samples. Note the three outboard screws used to set adhesive gap.

Surface preparation and cleaning also followed established practice. Metal bonding faces were flattened by rubbing them against fine grit emery paper taped onto a surface plate. They were then bead-blasted using medium grit glass media, producing a uniform matte surface finish. Glass bonding faces were ground to a 30-micron finish. Before gluing, these surfaces were cleaned three times in succession, with ethanol followed by acetone, using lint-free wipes.

Due to its short pot life, the epoxy components were measured out, mixed, and vacuum de-gassed immediately prior to use. A thick layer of epoxy was applied to both the metal and glass faces using a plastic spatula, being careful to wet the entire bonding area without introducing air bubbles. As the puck and block were slowly brought together, their final spacing was controlled by the gluing fixture, and the excess epoxy was squeezed out of the joint. This had the effect of flushing out any remaining air that otherwise might have been trapped in the interface. The excess epoxy was cleaned off and all bonds were allowed to cure undisturbed for a minimum of 7 days at room temp before testing was performed.

The primer presented its own difficulties; we had to experiment to find a suitable application method. Recommended primer thickness was 8-25 microns [0.0003-0.001 inches]. It could easily be applied with a small brush, but not with the uniformity or minimal thickness required. We tried spraying it on with an airbrush, also without success. Our solution was to brush on a thick coating, let it fully cure, and then sand to the required dimension. Thickness was readily determined by color comparison with a known good specimen. When thick, the primer appears bright yellow, but eventually becomes a dull olive green when the desired thickness is achieved. This was a slow and messy process, but produced excellent, repeatable results. Prior to gluing the primed surface was cleaned in the same manner as mentioned above for the unprimed surfaces.

2.3 Test Apparatus

The test apparatus was designed to apply tension to the bonded joint in a direction normal to the bond area, similar to a standard tensile pull test. The glass block was clamped down to a workbench. The attached puck had machined features at its non-bonded face, to which a pinned connection could be made. The device consisted of a lever with the fulcrum placed so as to amplify force by a factor of ten: 1 unit of downward directed input force resulted in 10 units of upward pull force on the test specimen. Compliant joints and pinned connections were used to ensure that the test sample was exposed only to tension. A more rigid configuration could have introduced moment loads that would have had the effect of peeling apart the joint, a far more detrimental condition. We intentionally designed our spectrometer space-frame with compliant flexures at bonded joints to avoid this situation.

Calibrated brass weights initially supplied the input force, but were tedious to use, and limited the resolution by which force could be increased. The weights were eventually replaced with a load cell, which allowed for finer resolution and the ability to slowly and continuously increase the applied force.

The load cell also allowed us to detect micro-creeping of the joint, since small dimensional changes reduced the magnitude of the initial force applied. The apparatus had an overall spring stiffness – the ratio of its deformation to input force applied – of approximately 7 microns/kg. The load cell's digital output display had a resolution of ½ kg, so creep movement as small as 4 microns [0.00016 inches] could be detected (Figure 2).

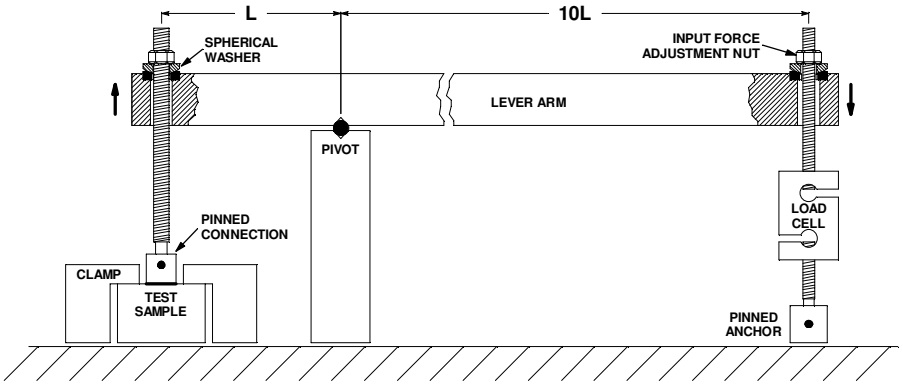

Figure 2 – Test Apparatus

2.4 Testing Protocol

Testing essentially consisted of applying tension to the joint, and slowly increasing it until the bonded faces were pulled apart. Approximately 40 test samples were made, representing five epoxies and the two puck/glass pairings. For the Milbond and the Hysol 9361 adhesives, samples were prepared both with and without Milbond primer.

Early in our testing we learned the importance of increasing the load slowly. Some joint failures, particularly those occurring at an adhesive/substrate interface, are preceded by significant micro-creeping, which increases the likelihood of failure and decreases the stress level at which it occurs. This happens on a relatively long timescale, often on the order of minutes or tens of minutes. If force is increased too quickly, this time-dependent phenomenon is not captured, resulting in an erroneously high measured value for joint strength.

Each sample was initially loaded to a joint stress of 8,000-10,000 kPa [1,160–1,450 psi] for 1 hour. Thereafter, the stress was increased in increments of approximately 800 kPa [116 psi], each time held for a minimum of 5 minutes, up to 2 hours. This time variation was at the discretion of the experimenter and was influenced by such factors as the presence of creeping, stress level, and expected stress at failure. The incremental time period tended to start out short, and increase over the life of the test, which made efficient use of available testing time and generated measurements that were most accurate at failure.

3. RESULTS

All samples were tested to failure. When it occurred, the stress level of the previously successful test load was recorded as the measured bond strength.

Bond failures were characterized by the nature of the failure mechanisms observed and by the proportion of the bond footprint each of these affected. Failures occurred at the surface of the glass substrate (adherend fracture), at the interface between adhesive and substrate (adhesive failure), and within the adhesive itself (cohesive failure).

Failures were dominated by one of these mechanisms, but generally exhibited mixtures of all three. Quality control standards usually characterize cohesive failure as a success, and adhesive failure as unacceptable. Adherend fracture would have been the best possible result, but in the course of these experiments glass failure rarely exceeded a couple of percent of the bond area. Metal substrate failure never occurred.

As mentioned before, joint creep was noted during some of the tests. It could occur when a new specimen was initially loaded, but often did not appear until some stress threshold was reached. Creep sometimes lasted only a few seconds, but if it continued beyond a minute, joint failure was invariably dominated by adhesive failure. The earlier this ‘long-term’ creep appeared in a test, the lower the final measured bond strength. Creep seemed to be exclusively associated with adhesive failure; it was not seen in those samples where the dominant failure mechanism was cohesive.

Whether or not primer was used, no adhesive failure was ever observed at an adhesive/metal interface. Perhaps this is due to the metal’s surface chemistry, or to the nature of the surface roughness pattern we imparted to it.

Substrate materials did not seem to be a factor in joint strength; we saw no significant difference in test results between Invar/Zerodur® and Titanium/BSL7Y bonds.

The most important result of the testing was the finding that the strongest bonds were consistently achieved using Hysol 9361 in conjunction with Milbond primer. These samples were also the only ones whose failure mode was dominated by cohesive failure (80-90% of area). Bond strength of this group fell in the range of 20,000-23,000 kPa [2,900-3,300 psi].

4. CONCLUSIONS

• We were able to repeatedly create bonds whose failure mechanism was almost fully cohesive, and therefore close to optimal. Further work might increase bond strength another 20 percent, but improvement beyond that would probably

depend on finding an adhesive with higher bulk strength. We were satisfied with the bond strength achieved, which should offer a significant bond strength safety factor.

• Testing would have been more productive if we had had access to an Instron® universal tester, which could be programmed to input continuously increasing load, as slowly as necessary, while measuring the resultant bond strain.

• Fillets created by excess epoxy at the junction of puck and optic should be avoided. This seems counter-intuitive, since it is common practice in machine design to use fillets at changes of part cross-section or direction in order to avoid stress concentrations. In our bonded joints, the adhesive itself is the source of stress concentrations since its CTE is one to two orders of magnitude higher than the substrates it bonds. The effect of this large mismatch is lessened with a thin bondline, but the relatively large dimensions of a fillet could generate damaging stresses on the glass substrate near the puck, especially when ambient operating temperature differs significantly from that experienced during bonding.

• Metal-to-glass adhesive bond data is difficult to find. Adhesive datasheets typically report bond strength based on a standardized test: ASTM D 1002, "Standard Test Method for Apparent Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tension Loading (Metal-to-Metal)". Those test conditions deviate far from ours, to the point of not being meaningful. In particular, the ASTM test calls for a "rate of loading of 80 to 100 kg/cm^2 (1200 to 1400 psi)/min…" As discussed previously, this rate would be far too fast for accurate measurements.

• Final advice – be cautious and view these results skeptically. The testing reported here was not statistically rigorous, and did not cover a wide variety of epoxies. Nor did it consider other types of adhesive chemistries (UV-cured, polyurethane, etc.) now commonly used in optical assembly.

5. BONDING PROCEDURE

This is the bonding procedure we followed. It was performed in a dust controlled environment:

• Roughen puck and optical surfaces (Figure 3).

• Clean puck and optical surfaces with ethanol and acetone.

• Apply primer masking rings to optic – these are sized to create a primer footprint 6mm larger than the puck (Figure 4).

• Apply primer to puck and optic (Figure 5).

• Allow primer to cure a minimum of 7 days at room temperature.

• Sand puck primer to required thickness.

• Remove masking ring from optic and sand primer spots to required thickness.

• Clean bonding areas with ethanol and acetone.

• Apply epoxy masking rings to optic, using a locating jig – the rings are sized to closely fit around the puck.

• Apply masking tape to cylindrical surface of puck.

• Prepare epoxy and apply to both puck and optic surfaces.

• Place pucks on optic – use locating/gluing jig to ensure correct placement and adhesive gap (Figure 6).

• Allow epoxy to cure a minimum of 7 days at room temperature (Figure 7).

• Remove fillets created by excess epoxy – facilitated by the presence of the previously applied masking tape (Figure 8).

• Remove remaining masking tape and clean area with alcohol – use soft tools to avoid scratching glass (Figure 9).

• Perform a pull test (we stressed each joint to 2,760 kPa [400 psi] and held it for one hour).

• Ship it.

• Time for a beer!

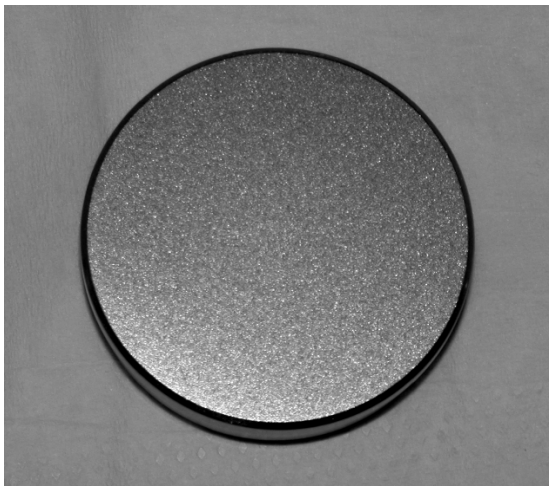

Figure 3 - Puck with uniform matte finish.

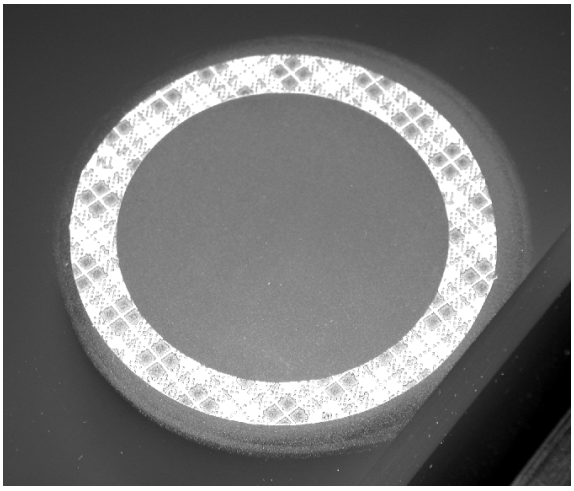

Figure 4 – Optic with masking ring used to define primer footprint. Note the ground finish in this area.

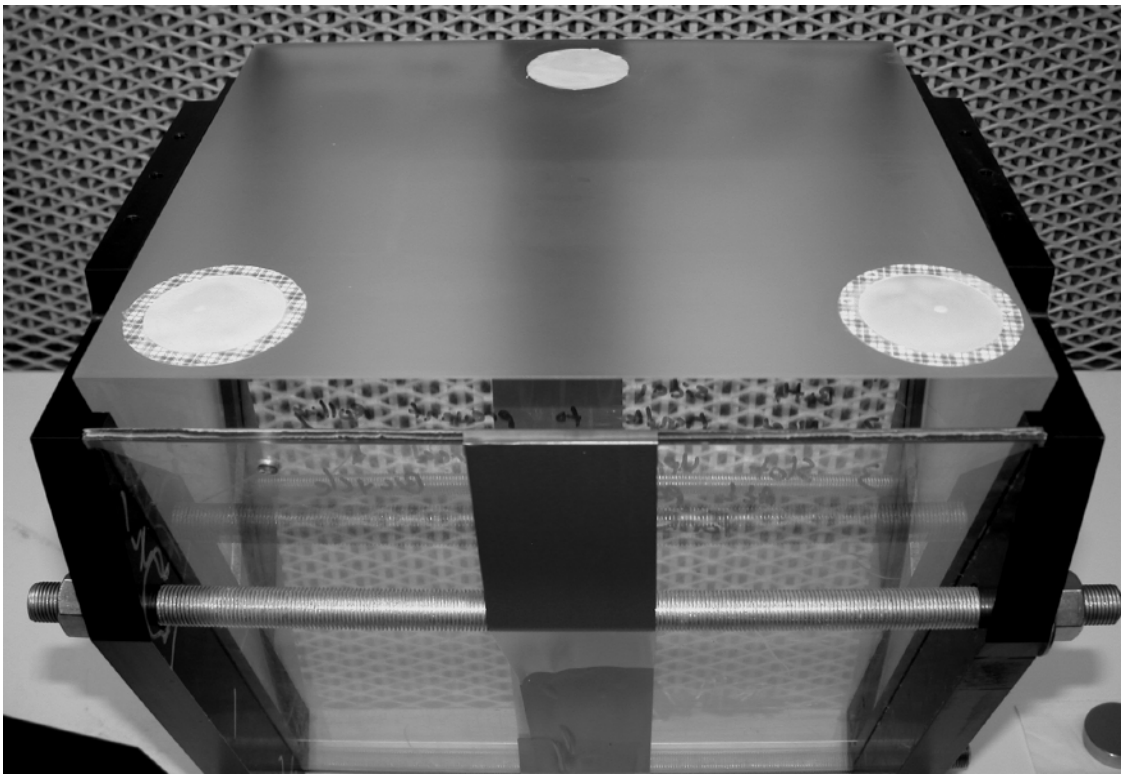

Figure 5 – Prism in its handling fixture showing primed bonding areas prior to sanding. Primer masking ring has been removed from the rearmost area.

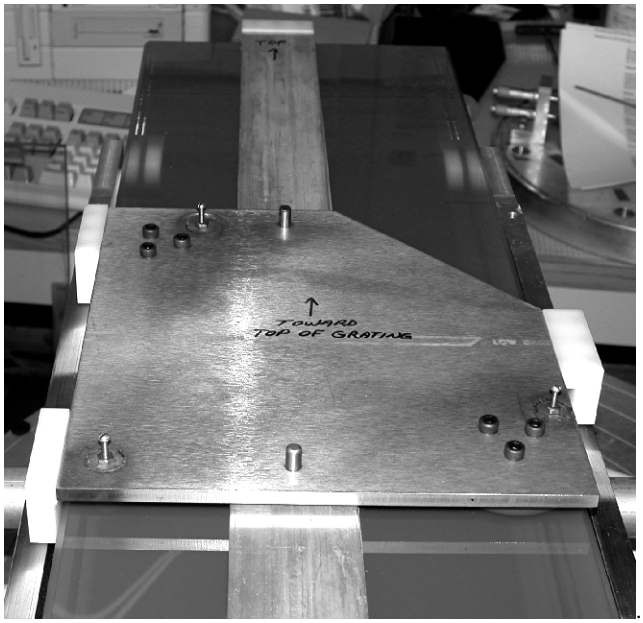

Figure 6 – Locating/gluing jig being used to position grating pucks.

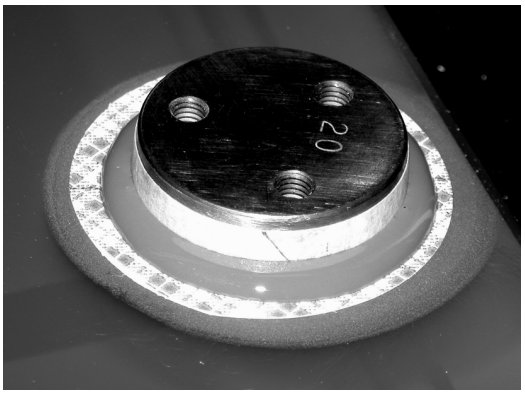

Figure 7 – Bonded puck with masking tape

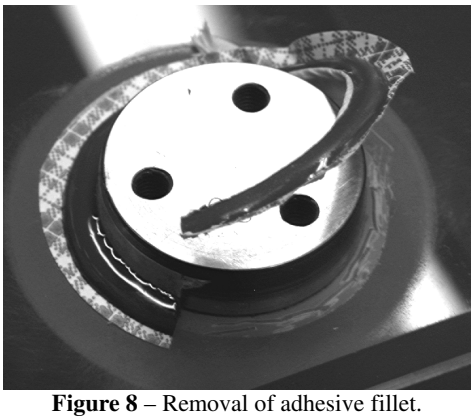

Figure 8 – Removal of adhesive fillet.

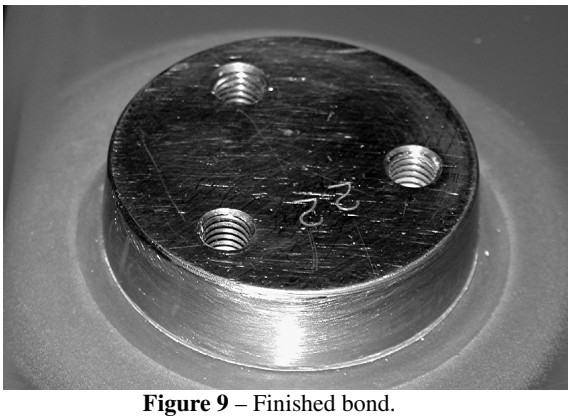

Figure 9 – Finished bond.

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