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Subject: Curing Contraction Ratios for Hysol 9313 and 3M 2216
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Summary

The WISDOM pupil slicer mirrors are mounted via bonding to Invar tabs. Because contraction of the bonding adhesive during curing is the primary cause of residual stress in the mirror, quantifying this contraction ratio is necessary to predict the success or failure of the bond design. Five samples of Hysol 9313 and two samples of 3M 2216 were mixed, cured, and measured with the properties and contraction ratio results shown in Table 1. Additionally, viscosity of 0% fill samples were compared, with Hysol 9313 exhibiting much lower overall viscosity.

Table 1. Average Volumetric Contraction Ratio Across 880 mm^2 Face of 1 cm^3 Adhesive Sample

	Volumetric Contraction Ratio		
	0% Siltex 44 Fill	20% Siltex 44 Fill	40% Siltex 44 Fill
Hysol 9313, non-outgassed	**6.3 %**	**5.0 %***	**3.5 %***
Hysol 9313, outgassed	**5.7 %**	~	**4.1 %**
3M 2216, non-outgassed	**4.7 %**	~	~
3M 2216, outgassed	**1.7 %**	~	~

**see results section for discussion*

These contraction ratios far exceed the contraction limits of 0.78% and 0.96% for Hysol 9313 and 3M 2216 respectively, deduced via analysis and assuming a rule of thumb of 1ksi for allowable bonding stress (see 28 October 2015 engineering memo "FEA Analysis of Stress Induced In Zerodur By Curing Contraction of Hysol 9313 and 3M 2216").

Given the number of unknowns that remain, it is recommended that a test piece of Zerodur be bonded to an Invar mounting tab to determine whether the glass will survive bonding, and to further refine the analysis model.

Methodology

A testing fixture with ten square-shaped pockets of 10.0 cm^3 volume (area 880.5 mm^2, depth 11.36 mm) was manufactured of Al 6061 (see Figure 1). Epoxy was filled at room temperature and ambient humidity (30 to 60% in the course of curing) to the top of these pockets using surface tension and the reflectance of light off the adhesive surface to determine flatness with the surface of the fixture.

Three of the Hysol 9313 samples were non-outgassed, consisting of Siltex 44 (silica powder) fill ratios of 0%, 20%, and 40%. The other two Hysol 9313 samples were 0% and 40% fill, outgassed via a small vacuum pump for approximately 10 minutes. Both 3M 2216 samples had 0% fill ratios, one outgassed and one not. Siltex 44 was not used to fill the 2216 epoxy because its viscosity would have become unworkable (it starts out very viscous). Fumed silica fill, 0.2-0.3 µm particle size, was also experimented with but resulted in an epoxy mixture far too viscous for practical use.

Figure 1. Testing Fixture With Measuring Locations, Results Shown

After curing for a minimum of 3 days, each adhesive surface was measured in seven discrete locations, roughly consistent across all the samples, shown in Figure 2. The measurements were performed with an electronic drop indicator (Mitutoyo part number 543-783, 0.0005 inch resolution). These measurements were averaged to obtain the contraction ratios previously listed in Table 1.

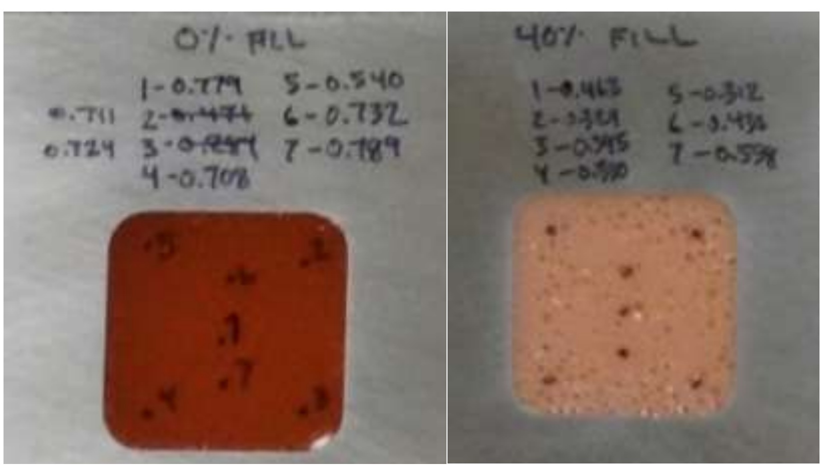

Figure 2. Measuring Locations on Adhesive Surfaces

As a bonus test, the viscosities of Hysol 9313 and 3M 2216 (both 0% fill) were observed by dropping 0.1 grams onto an inclined surface, as shown in Figure 3.

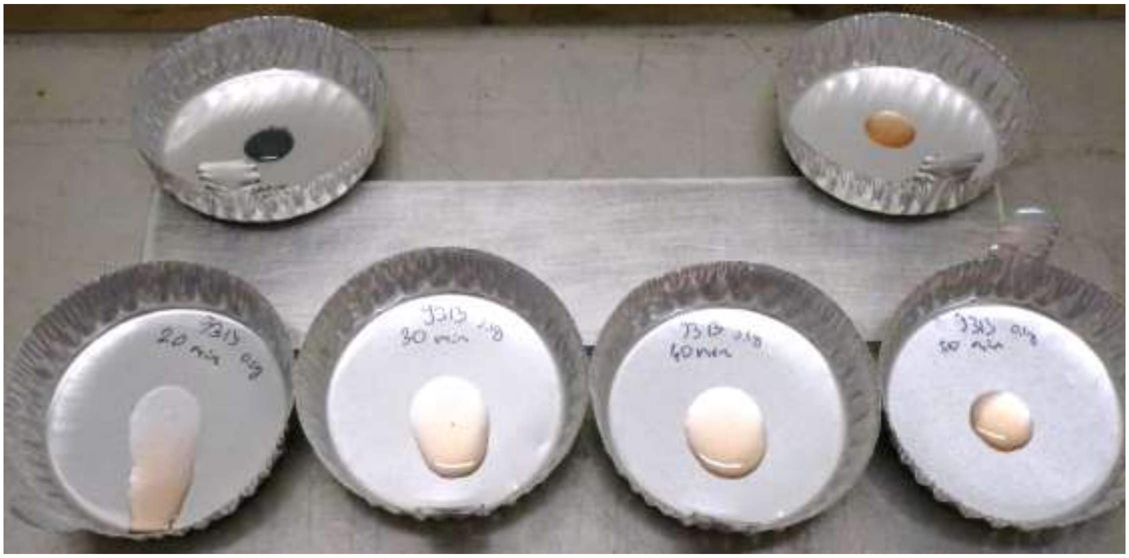

Figure 3. Viscosity Tests: Hysol 9313 at 5 Ten-Minute Intervals (20-60 Minutes Post Mixing), 3M 2216 at 20 Minutes After Mixing

Results

Table 1 summarizes the contraction ratios measured after the samples cured. It must be noted that the high number of air bubbles in the non-outgassed, 20% and 40% fill Hysol 9313 samples made reliable measurements very difficult and thus these measurements should be taken with a grain of salt. Furthermore, the inability to perfectly level the test fixture, variations in the fixture's profile tolerance, and the inability to fill each sample to exactly 10.0 mL introduced uncertainty. Measured contraction ratios are probably only reliable to within 1% or so. It's also worth noting that the surface area of the adhesive exposed to air may also affect contraction – these test samples do not simulate the bonding geometry employed in the pupil slicer design.

Even so, several conclusions can be made. First, Hysol 9313 has a volumetric contraction ratio of approximately 4-6% depending on fill and whether it has been outgassed. This is quite a bit larger than 3M 2216 contraction ratios at approximately 2-5%. Second, all of these contraction ratios exceed the allowable ratios of 0.78% and 0.96% for Hysol 9313 and 3M 2216 respectively, deduced via analysis outlined in the 28 October 2015 engineering memo "FEA Analysis of Stress Induced In Zerodur By Curing Contraction of Hysol 9313 and 3M 2216". Though this does not necessarily mean the glass will break, it does mean that if the model is accurate, the 1ksi stress rule of thumb is exceeded.

3M 2216 proved to be much more viscous than Hysol 9313, to the point that even no-fill 2216 is likely too viscous for our application. 0% fill Hysol 9313 appeared to reach a good level of viscosity 30 minutes after mixing.

Future Work

Due to schedule constraints, further analysis and testing of adhesive configurations is not possible at this time. Rather, a test piece of Zerodur will be bonded to an Invar mounting tab, using Hysol 9313 with 40% Siltex 44 fill and outgassed. This test piece will be cooled and heated through the operational temperature range (-15 to +30 C). Whether or not the glass or bonding breaks will allow us to refine our analysis model and further assess the suitability of Hysol 9313 as an adhesive for the pupil slicer mirror bonding.