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LIGO Laboratory / LIGO Scientific Collaboration

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Use of PI-2525 Resin with T-9039 Solvent on Vacuum Parts for LIGO

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The purpose of this project is to investigate methods by which a vacuum-safe resin coating can used to manufacture UHV-compatible parts in LIGO. We use poli-hymide resins because of their low outgassing properties. The resin initially used was Cycom 3001, now going out of production. Presently we are using resin PI-2525. Because of their high curing temperature, these polymers can withstand high temperature baking (above 350^{o} C).

These resins have been developed for the silicon microchip industry and as insulating varnish for transformer wires. In these applications the resin is used in very thin films. In LIGO it would be used as coatings to isolate and freeze the windings of actuation and sensing coils. Much thicker layers form, especially between successive windings in a coil. Past tests have shown that the curing process of thick films can cause foaming (in the industry this effect is called Yellow Powder Syndrome. It is generated because the curing process generates water molecules that in thin films can diffuse to the surface and boil off, but in thick films remain trapped in the volume in the form of bubbles). Foaming in the resin is totally unacceptable in UHV environment, as it can trap dirt or liquids (as well as trapping the water and solvent vapor that generate the foaming), which cannot be cleaned or removed.

We investigate different resins, baking profiles, and solutions to produce a smooth, even coating. In particular, we attempt to avoid "Yellow Powder Syndrome," in which baking a resin causes it to break up into a dull powder.

Diluting the Resin:

We are testing the resin PI-2525 from Dupont. The resin is conserved in a freezer at -25 ° C to -30 ° C. The manufacturer's literature [1] suggests dissolving the resin in a solution prior to baking in order to prevent powder formation. As solvent, we use the T-9039 thinner from HD Microsystems, composed of n-Methylpyrrolidone and Propylene Glycol Monomethyl Ether [2]. The mixture is approximately 4:1 solvent to resin by volume (the solution and resin have similar densities). Jack Craig at Dupont prescribes that, in order to properly dissolve the resins in their solvents, it is necessary to mix them for a couple hours and allow them to sit overnight. The mixing is performed at room temperature by a modified rock tumbler (figure 1). The tumbler's chamber has been modified to hold a standard glass laboratory bottle, in which the resin and solvent are mixed.

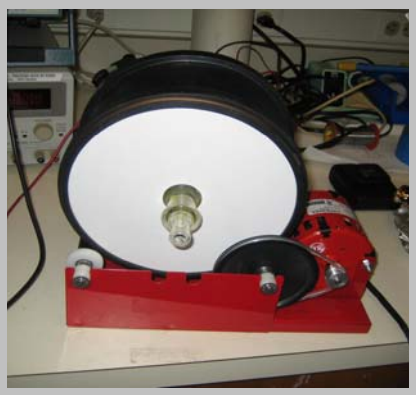


Figure 1

Upper Left: The bottle is placed into the modified mixer cavity and held in place by plastic framework.

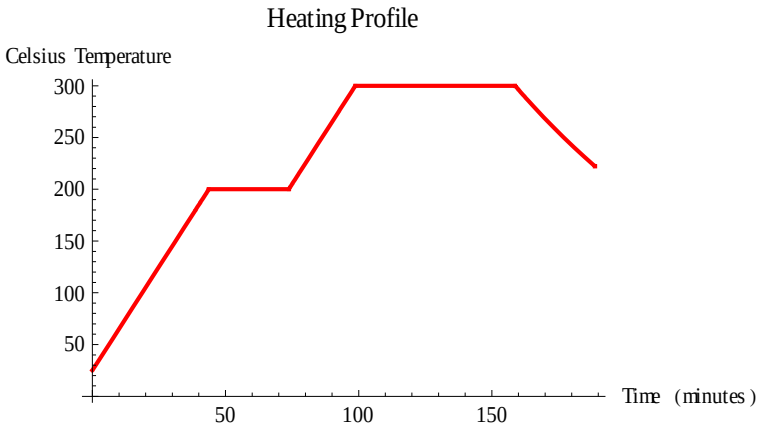
Lower Left: The cavity is placed on the mixer and rolled by the motor until the solution is well-mixed.

Lower Right: PI-2525 must be dissolved in a special solution—attempting to use ethanol creates a hard residue and ruins the sample.

Curing the Resin:

Curing is done in a programmable oven. The resin is poured into a slanted aluminum tray (the slanting causes a variation in thickness along the sample which, for each sample and heating profile, will allow us to measure the maximum achievable thickness without foaming) and placed in the oven.

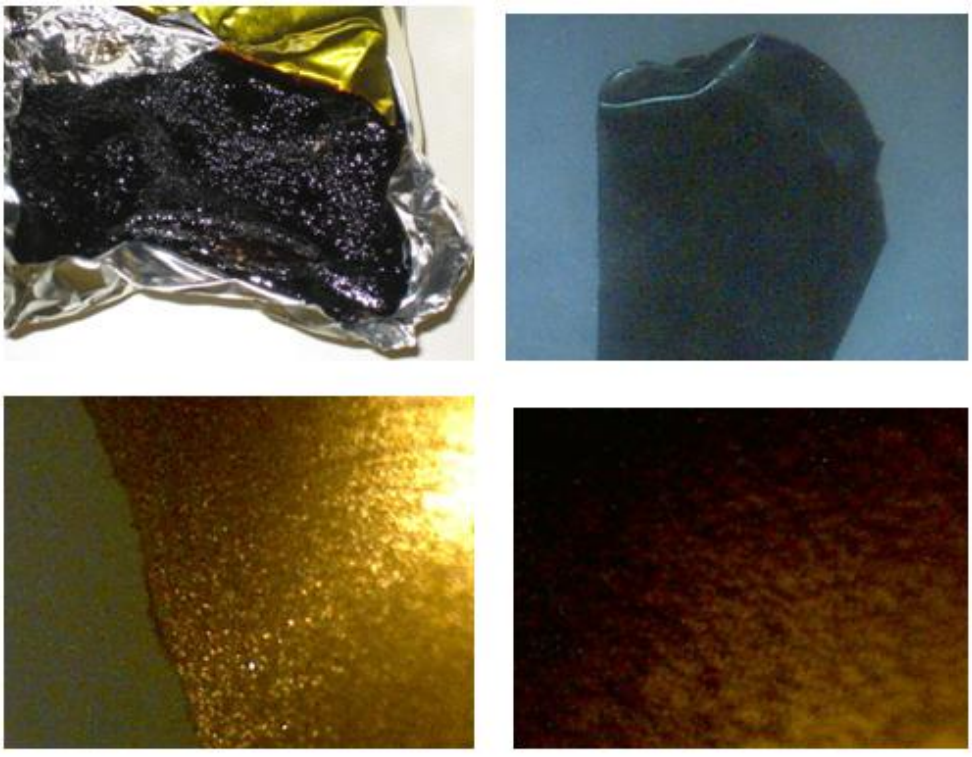
We started with pure resin. The initial heating profile used is diagrammed below. Allowing the sample to cool for inspection in between sections of the heating profile does not appear to affect the process.



The initial heating profile used for resin PI-2525

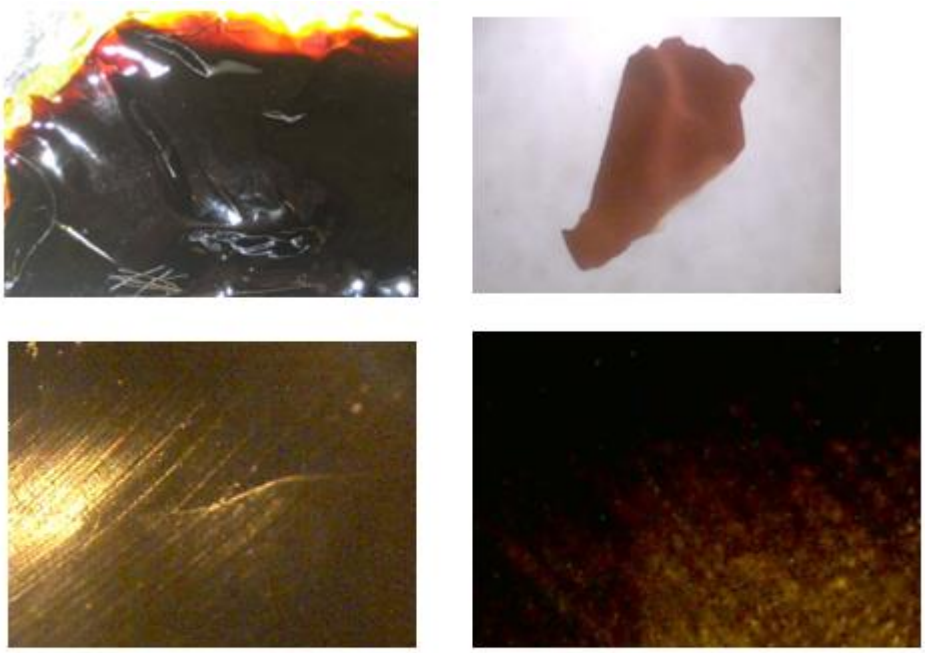
First Samples:

The first sample produced using PI-2525 with no solvent generated moderate bubbling, although no powder was present, even below the surface (bubbling was observed with Cycom 3001 produced under similar conditions):



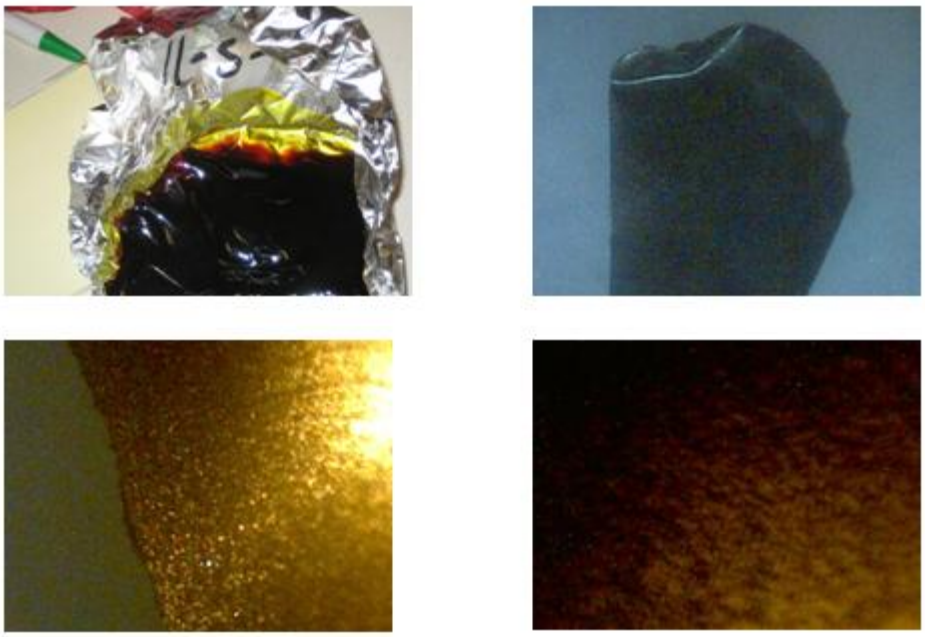
Above: Clockwise from upper left: Normal magnification, 10x magnification, 200x magnification, 60x magnification. Bubbling can be clearly seen, but there is no powdering, and the surface is fairly smooth even at high magnification.

The next sample produced was one with a very slow heating profile, with the entire heating taking 20 hours:



Above: Clockwise from upper left: Normal magnification, 10x magnification, 200x magnification, 60x magnification. While the sample appears very smooth to the naked eye, there are ridges visible at 60x and higher. These may have been produced in removing the sample from its aluminum tray.

The next sample was run with a relatively modest time budget. Its holds were simply doubled from the original heating profile (60 minutes and 120 minutes). The results were very similar to the second sample:

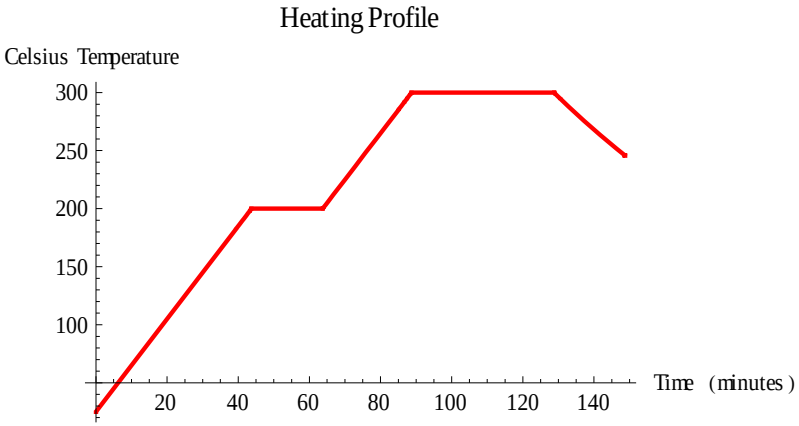


Above: Top Row—the sample at normal magnification and at 10x.

Bottom—the sample at 60x magnification. The sample is very smooth; there do not seem to be any deficits as compared to the second sample.

Tests with PI-2525 Resin Diluted with T-9039 Solvent:

The PI-2525 resin was tested both with and without the T-9039 solvent. Several runs were performed, with holds at 200° C and 300° C as short as 20 and 40 minutes respectively:



	Hold 1	**Hold 2**	**Solution**	**Notes**
Run 1	60 min	120 min	No	Good, no bubbling or warping
	60 min	120 min	Yes	Good, no bubbling or warping
Run 2	40 min	80 min	No	Good, no bubbling or warping
	40 min	80 min	Yes	Good, no bubbling or warping
Run 3	20 min	40 min	No	Good, no bubbling or warping
	20 min	40 min	Yes	Good, no bubbling or warping

The PI-2525 resin proved uniformly superior to the original resin, not exhibiting the Yellow Powder Syndrome in samples as thick as about a millimeter. Runs were made with dissolved and undissolved samples together in the oven. The two samples were principally the same. In this set of tests, no bubbling was observed, probably due to a thinner layering of the sample. It may also be that the greater time at room temperature prior to curing (as the other sample was being dissolved) contributed to the superior samples. All of the solution evaporated during the baking run. The diluted sample yielded a somewhat thinner,smoother and more evenly distributed finish:




The above samples were created using PI-2525 and the heating profile of the figure above. The sample depicted in the two photos on the right was dissolved in T-9039 thinner, and the sample on the left was not. Both samples peel away from the aluminum tray smoothly and without breaking.

Attempts to measure the hardness of the baked resin have proved unsuccessful. The resin is below the hardness which a standard press can measure.

Recommendation:

The PI-2525 resin can be effectively cured to parts for use in the UHV environments necessary in LIGO. Based on our tests and the manufacturer's recommendation, we recommend the following formula:

- Thoroughly mix T-9039 and PI-2525 in a ratio of 4:1; allow the mixture to sit overnight.
- Apply the mixture to part and heat in oven:

- Heat from room temperature to 200° C at 4° C / min.
- Hold at 200° C for 40 minutes.
- Increase temperature to 300° C at 4° C / min.
- Hold at 300° C for 40 minutes.
- Allow part to cool to room temperature.

- T-9039 must be stored in a chemical cabinet. PI-2525 should be kept refrigerated to increase its shelf life.

- The container of resin must be brought to room temperature before opening in order to prevent water from condensing on the resin. A fan is used to accelerate the warming process. The original bottle of resin was divided into single-use samples of about 30 g of resin so that the entire resin would not have to be repeatedly warmed and cooled.

References:

HD Microsystems, "PRODUCT BULLETIN PI-2525, PI-2555, PI-2556 & PI-2574 Polyimide," August 2005

HD Microsystems, "Material Safety Data Sheet: T-9039," December 2004