S. Timm
M. S. Alam
F. Wappler
B. Athar
SUNY Albany
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TEA Aging Studies

1 Introduction

The CLEO III detector at the CESR facility at Cornell University will include a fast RICH detector which uses a photosensitive pad chamber to detect the Cerenkov photons[1, 2]. This detector will be filled with a gaseous mixture of methane (CH_4) and triethylamine ($(CH_3CH_2)_3N$), hereafter referred to as TEA. Triethylamine is a photosensitive compound with peak sensitivity to photons in the vacuum ultraviolet (120-180 nm) region of the spectrum. A prototype of this detector has successfully been tested at Syracuse University, and the detector is currently under construction there.

The goal of this experiment is to measure the long-term effects of exposure of the detector materials to triethylamine. This will be accomplished by placing these materials in an atmosphere of heated CH_4/TEA to accelerate the rate of reaction beyond that which would happen at room temperature.

2 Apparatus

2.1 The TEA bubbler

The TEA/CH_4 mixture is made by bubbling CH_4 through liquid TEA at 15° C. The vapor pressure of TEA at this temperature is 45 torr, so TEA will be about 6% of the combined gas. This is calculated via the Clausius-Clapeyron equation. Assuming that H_v, the latent heat of vaporization, is constant across the given temperature range, we can use this equation to calculate the vapor pressure at any given temperature.

$$P = p_0 \exp(\frac{-H_v}{RT}) \tag{1}$$

The constant p_0 is determined by measuring the vapor pressure at some known temperature. The known constants are at T_0=300 K, P=73.2 torr, H_v/R=4299 K[4], yielding $p_0 = 1.22 \times 10^8$ torr. This in turn implies that the partial pressure of TEA at 283 K is 55 torr. At -10° C, the normal operating temperature for our cold trap, the partial pressure is 10 torr.

The TEA is held in a one-pint Mason jar[1] with a Viton seal, and a set of shut-off valves allows us to bypass the TEA bubbler if necessary. All connections within this system are made with stainless steel pipe and Swagelok fittings. A blow-off valve is included in the system before the last shut-off valve to guard against accident conditions such as a wrong alignment of valves or a clogged-up TEA bubbler. This valve is set to blow off if the interior pressure is more than 5 psi above atmospheric pressure. It is vented through the cold trap into the fume hood.

There is also an input for nitrogen at the beginning of the system to allow for flushing the system and leak-checking. We generally flush the system with nitrogen for 1 hour before introducing CH_4 and TEA, and for at least 4 hours afterwards. In addition, nitrogen flows through the system throughout the heat only trials.

2.2 The reaction vessel

We are using the Lindberg furnace in room B-8 of the Physics building. A glass tube has been designed and constructed to fit inside the bore of that tube furnace. It is 7 cm outside diameter and 95 cm long. In normal operating conditions this volume will continuously have gas flowing through it so a large pressure build-up is not expected, however it is designed to hold a pressure at least twice atmospheric pressure (which would occur if the volume were heated to 100° C while closed). The walls of the vessel are rated to hold 51 psi. We plan to never have pressures higher than 30 psi. The vessel has a volume of approximately 3 liters. We set our gas flow to 3.5 liters per hour during normal operation. When filling the volume 20 liters per hour is our maximum flow. The flow is measured and regulated with a flow meter.

The glass tube is connected to the pipes on either end by a combination

[1]Not all Mason jars are equal. The correct jar is a Ball Perfect Mason #14 pint jar, which has an O.D. at the neck of 2.5 inches, not 2.56 inches as many modern pint jars do.

of Del-Seal flanges, which are a metal-on-metal seal. At the entry end a straight glass-to-metal joint is attached to the glass vessel with glassblowing and attached via a bellows connection to the gas system. At this end there is also an extra tee joint through which a K-type thermocouple is inserted and attached to an electronic thermometer. The positioning of this thermocouple allows us to measure either the temperature of the air inside the vessel or the temperature of the vessel wall itself. At the vent there is a glass-to-metal connector with a flexible metal bellows, which joins the copper vent pipe which is also connected to the glass cold trap by the same type of connector.

We use a cold trap of dry ice and ethylene glycol to re-liquefy the TEA. This cold trap sits in the fume hood and the CH_4 carrier gas is vented out into the fume hood. 6 lbs. of dry ice can keep 10 liters of ethylene glycol at -10° C for 10 hours or more.

2.3 Safety

The system has been leak-checked with Snoop and an electronic gas-leak sniffer. We also have a flammable gas monitor to monitor room-level gas build-up. However, since TEA is detectable by the human nose in concentrations of one part per million, and only hazardous at 200 parts per million, the human nose proves to be quite an effective monitor. Dr. Greg Peterson in our Physics department has examined our setup and found it to be sound, as have Vincent Franconere and Lisa Donohue of the safety division.

3 Calibration

We can calculate the estimated exposure by the following method. Reaction rates can generally be written in the following format:

$$rate = k[A][B] \tag{2}$$

where the above is a second order reaction, first order in the concentrations of elements $[A]$ and $[B]$. With organic amines such as TEA, a typical reaction is SN_2, second-order nucleophilic substitution on alkyl halides[5]. In a gas-phase reaction such as the one above, the concentration of reactants will also depend on temperature but will be a small effect compared to the explicit

exponential effect on the rate constant k, which can be written as

$$k = A \exp -(\frac{E_A}{RT}) \tag{3}$$

where T is the temperature in Kelvin, R is the ideal gas constant, 8.314 J/mol K, and E_A is the activation energy, which is not known for many of these reactions but for reactions of simpler compounds is often on the order of 84000 J/mol. Using this as a typical activation energy, we can see that heating the system from 20° C to 100° C will accelerate the reaction by a factor of 1600, and if E_A is higher than that the acceleration will be even faster. On the other hand, if E_A is lower than that, the absolute rate of reaction ought to be such that it will happen at room temperature in any case. Thus we can give a reasonable lower limit on the length of exposure that has been observed by our system. A chart of effective exposure time as a function of activation energy for three different temperatures is shown in Figure 1.

Although the rate constant increases as the temperature increases, liquid TEA tests are still important because a much greater concentration of TEA is present. Given that the density of TEA is 0.7245 g/cm^3[3] at and its molecular weight is 101 g/mol, the TEA concentration in liquid TEA is 38 mol/liter. As a gas at 100° C the concentration would be 0.032 mol/liter assuming ideal gas behavior, and since TEA is only 6% of our gas mix the concentration is 0.0019 mol/liter. This factor of 20000 decrease in the concentration is only partially offset by the acceleration of the reaction rate by a factor of 1600.

Our temperature probe has been verified to read 0 degrees C in a bath of ice water and 100 degrees C in boiling water.

4 Materials To Be Tested

4.1 Adhesives

Four types of adhesives are needed in the project. All can be handled without any special precautions. We use a small blob of the adhesive on an aluminum substrate to check for discoloration, loss of mass, and loss of adhesion.

Torrseal (from Varian Vacuum Products) is used for the joints between calcium fluoride windows. These joints should be checked both for gas tight-

ness and mechanical strength. We simulate calcium fluoride windows with glass of equal thickness (2 mm.) Torrseal combines a epoxy resin with diethylenetriamine as its hardener.

We test both Epon and EGA-148 for securing the Ultem hinge to the windows and the side rails, as well as the cathode boards. Again we test for gas tightness and mechanical strength. Epon 828 (from Shell Chemical) is a bisphenol/epichlorohydrin resin and we mix it with Versamid 140, a polyamide resin from Henkel in a 2:1 ratio by mass. EGA-148 (from IPN Industries, Inc.) combines a proprietary resin and amine mix. These glues were both candidates to replace a Hysol epoxy. We are also testing the Hysol epoxy that these two were scheduled to replace.

A silver conducting epoxy KS-0002 (from Hysol Division, Dexter Corp.) secures the high voltage cable to the printed circuit board. We need to test this for adhesion to the board, conductivity, and adhesion to Kapton.

Another adhesive will be used to connect the wires to the ceramic spacers. This will be either Delta Bond Blue 152 (from Wakefield Engineering) or Armstrong A-12 (from Armstrong). Delta Bond Blue 152 is a combination of aluminum oxide and epoxy resin and is hardened with a B-4 hardener.

All the above adhesives have been tested in liquid TEA at Syracuse[6] and survived for a reasonable time period. Epon, Delta Bond Blue, and Armstrong eventually did show some damage in liquid TEA.

We also test some of the adhesives which showed relatively rapid damage in liquid TEA, such as Devcon's 5-Minute Epoxy and RTV.

4.2 Wire Coatings

We test various wire coatings including PVC (known to degrade), Kynar, Tefcel, Teflon, XF0150, PEEK, and Kapton. We will also be testing wires of various metals including copper, stainless steel, and the gold/rhenium and gold/tungsten wires of the chamber.

4.3 Construction materials

We have samples of the various types of fiberglass which will be used in the side rails and end rails of the detector. We test hinge materials including Ultem, Kapton (used as electrical shielding in various applications), and circuit board materials such as G-10, FR-4, polyimide quartz, and polyimide

glass. We also test pieces of the actual cathode board from the prototype. In addition we test samples of the ceramic spacers and Viton A. Finally we test Noryl and a sample of silver paint on glass; both are known to degrade in TEA.

5 Experimental Procedure

5.1 Mass tests

For each glue, we have made 3 small samples on aluminum slabs. One is the room temperature (cold) control. The second is heated in the furnace under N2 and is called the hot control. The third is placed in the CH_4-TEA mixture and heated, and is referred to as the TEA sample. Every 24 hours for hot controls, and every 12 hours for TEA samples, we remove them from the furnace. We measure the mass with a precision balance, and examine by eye for surface defects and discolorations, and make sure that the adhesive adheres to the slab.

We can use adhesives known to degrade rapidly in liquid TEA to calibrate the speed of the effect. Mass tests are also performed on the wire coating samples and the construction material samples.

5.2 Strength tests

The adhesives we are testing are rated to be good to 2500-5000 pounds per square inch under tensile and shear strength. We test both properties by using each adhesive to glue together two pieces of $\frac{1}{4}$ inch diameter G-10 rod end to end. We put a sample under tensile stress until it breaks, then make another one and test it at 90% of breaking strength after various intervals of exposure to TEA. A similar mechanism is used to measure shear stress.

5.3 Leak tests

We have constructed an aluminum frame of a size small enough to fit in our furnace tube. We attached an Ultem hinge of the type used in the prototype RICH to the frame with EGA-148. We then attached two thin glass windows to the hinge with Epon/Versamid, and attached the windows to each other

with Torrseal. This frame forms the top of a small box, and was verified with a helium leak-checker to be leak-tight. This frame was used as our heat control sample. A second frame was constructed using Hysol adhesive in place of the EGA-148 and Epon/Versamid. This frame was used as our TEA sample.

6 Results

We have now completed one round of controls and TEA. In both cases we ran the furnace for 60 hours at or near 100 degrees C. This is roughly equivalent to an exposure time of 10 years. Our first set of samples comprised four glues, red RTV, Armstrong A-12, Torrseal and Epon 828/Versamid 140 (2:1) mix. We had two pieces of fiberglass that will be used in detector construction, and a small piece of the cathode board that was used in the prototype detector, which is made of FR-4 but is coated with copper and then gold-plated, with 5-minute epoxy plugging the holes. It also contained 7 wire coatings: Teflon, Kynar, Tefzel, XF0150, PEEK (PolyEtherEtherKetone), MIL-81044 (a cross-linked polyalkene), and Kapton. For the control samples we measured the mass at the beginning and end of the run and one intermediate interval. For the experimental samples that were in the TEA we turned off the furnace every 12 hours and checked the weight and appearance of the samples. The second batch of samples contained the rest of the materials described above that were going to be tested.

6.1 Adhesives

We had expected RTV to degrade rapidly. In fact it did not. This is a special type of RTV which is heat-resistant and that may be why. In any case there was no visible change in the samples of the heat resistant red RTV. We also tried translucent RTV and white RTV (the latter being part of the same batch which had degraded in the liquid TEA), and again saw no changes. 5-Minute Epoxy was advertised to degrade rapidly in liquid TEA. We observed that this glue turns from clear to orange, but this happens both in heat and in TEA. Also no visible change was observed for Armstrong A-12. Torrseal turned slightly off-white both in the heat-only control and in the TEA run. Epon/Versamid, normally a very light golden solid, turned a dark golden

brown in both the heat-only control and the TEA run. A call to the Henkel corporation confirmed that Epon/Versamid is likely to do this in the presence of heat and that part of the darkening is due to the reactions of the excess polyamide resin. The effect appears to be enhanced due to the presence of oxygen. In our second run we repeated the test with Epon/Versamid taking care to exclude oxygen from our furnace and also making samples containing many fewer bubbles, and the effect still occurred. The hot control EGA-148 sample fell off the slab, but a replacement test slab did not exhibit this behavior, nor did the TEA sample. There was no visible color change for EGA-148. The Delta Bond Blue sample was all right in heat, but in TEA we observed three different samples, from different mixes of that adhesive, all fall off the aluminum slab. Hysol was fine in the heat control but in the TEA two samples gradually turned a dark orange, beginning at the edges and working in.

6.2 Construction Materials

No visible changes occurred in the pink and black fiberglass samples or the piece of the prototype cathode board, either in the hot-only controls or in the TEA run. Pieces of Kapton, ceramic spacer, G-10, Viton A, Ultem, and the various circuit board materials showed no damage. An exception was FR-4, where the edges of the sample turned slightly brown in TEA. A piece of Noryl warped slightly in the heat trial but showed evidences of cracking and disintegration as well as warping in the TEA. Also a sample of silver paint painted on glass gradually flaked off in the TEA but was ok in the heat control. These latter two outcomes were expected based on earlier experience with liquid TEA and the prototype.

6.3 Wire Coatings

The Teflon and Kapton wire coatings showed no visible changes in either the heat-only run or the TEA run. The Mil-81044 wire did not change color in the heat-only run but turned a dark brown in TEA. The Kynar wire turned light brown in heat but again turned a dark brown in TEA. Both the wires that turned dark brown did so within the first exposure period. XF0150 turned slightly off-white in both conditions, ETFE turned a light brown in both conditions, and the PEEK tubing which begins as a clear amber became

cloudy and stiffer in both conditions. The red PVC-coated wire maintained its color through the heat trial although some melting was observed. It was scorched almost to a brown color in the TEA. The yellow Teflon-coated wire was slightly scorched in the heat run, but was thoroughly demolished in the TEA. A piece of speaker wire with a clear coat (most likely polyethylene) survived the heat, becoming a little cloudy, but vaporized instantly in the TEA run, condensing to a white powder in the vent of our apparatus and forcing us to temporarily halt the run.

6.4 Mass Loss

The mass results presented in Table 1, which was our first batch, are in general good to ± 2 mg. The uncorrected mass results are shown. The masses in the heat control run and the early part of the TEA run exhibit some shifts in the absolute mass scale. In our second batch of materials, described in Table 2, we acquired a better balance and believe the mass results are good to ± 1 mg. The fiberglass-based materials experience a slight loss of mass due to prolonged heating. In the heat controls, the Teflon and PEEK samples show loss of mass because part of the sample identification tag fell off of these samples. We do not observe any significant change in mass in the wire coating samples or in the adhesive samples. In the tables, samples which fell off the slab are indicated by *, samples which broke and were replaced are noted by †, and samples which lost their ID tag are indicated by ‡.

6.5 Strength Test

Seven adhesives were tested for strength. These were Epon/Versamid, Hysol, 5 Minute Epoxy, Delta Bond Blue, Torrseal, Armstrong A-12, and EGA-148. Since the area of the glue seal was 0.049 square inches and the glues are rated to thousands of pounds per square inch, we would expect the glues to break under 10-100 kilograms of force, and in fact they all did.

We applied tensile stress by gripping one end of the sample in a vise, hanging downwards, and then hanging weights on the other end of a sample by means of a hole bored near its end. Shear stress was applied by gripping one end of the sample in a vise and then hanging weights so that the force was applied perpendicular to the glue joint and as near to the glue joint as possible to minimize torque.

Table 1: Sample Mass (in grams) After Exposure to TEA

TEA SAMPLE	Exposure time (hours) 0	12	24	36	48	60
RTV	3.032	3.026	3.030	3.046	3.028	3.025
Armstrong A-12	3.243	3.242	3.245	3.244	3.244	3.244
Torrseal	3.438	3.432	3.435	3.435	3.432	3.433
Epon/Versamid	3.885	3.887	3.891	3.889	3.887	3.888
Pink Fiberglass	18.498	18.479	18.477	18.471	18.468	18.466
Black Fiberglass	3.989	3.985	3.988	3.992	3.984	3.983
Cathode Board	10.251	10.250	10.252	10.250	10.245	10.245
Teflon Wire	2.461	2.477	2.474	2.477	2.470	2.469
Kynar Wire	0.679	0.687	0.690	0.691	0.686	0.686
XF0150 Wire	2.932	2.933	2.937	2.975	2.931	2.929
ETFE (Tefzel)	1.807	1.805	1.809	1.808	1.808	1.807
Mil-81044	0.778	0.786	0.788	0.790	0.785	0.785
Kapton	0.308	0.310	0.315	0.312	0.309	0.309
PEEK	0.163					0.163

HOT CONTROL	Exposure Time (hours) 0	21	60
RTV	2.915	2.907	2.913
Armstrong A-12	3.355	3.351	3.355
Torrseal	3.295	3.288	3.291
Epon/Versamid	4.719	4.716	4.723
Pink Fiberglass	19.385	19.348	19.346
Black Fiberglass	4.089	4.062	4.065
Cathode Board	11.475	11.457	11.462
Teflon Wire	2.456	2.448	‡ 2.423
Kynar Wire	0.699	0.692	0.697
XF0150	3.025	3.015	3.020
ETFE (Tefzel)	1.907	1.901	1.905
Mil-81044	0.769	0.765	0.768
Kapton	0.327	0.328	0.329
PEEK	0.161	0.160	‡ 0.146

Table 2: Sample Mass (in grams) After Exposure to TEA

TEA	Exposure time (hours)					
SAMPLE	0	12	24	36	48	60
Delta Bond Blue A	4.2526	† 4.2472	4.2453	4.2441	4.2446	4.2445
Delta Bond Blue B		4.2607	† 4.2571	4.2563	4.2562	4.2561
EGA 148	3.1741	3.1690	3.1685	3.1684	3.1686	3.1684
Epon Versamid B	3.3704	3.3709	3.3713	3.3715	3.3714	3.3715
Clear RTV	3.2357	3.2305	3.2293	3.2287	3.2277	3.2273
White RTV	2.7902	2.8096	2.7905	2.7903	2.7878	2.7865
Hysol A	3.0790	3.0764	3.0763	3.0764	3.0760	3.0765
Hysol B		3.1640	3.1616	3.1619	3.1618	3.1618
KS-0002	3.7498	3.7400	3.7375	3.7346	3.7320	3.7310
5-minute Epoxy	3.2561	3.2535	3.2534	3.2538	3.2529	3.2534
Delta Bond Blue	5.4703	5.4654	5.4644	5.4642	5.4632	5.4603
EGA-148	4.6525	4.6475	4.6470	4.6463	4.6458	4.6451
Epon/Versamid	4.7567	4.7541	4.7524	4.7522	4.7508	4.7503
Hysol	4.8964	4.8708	*3.5482	3.5481	3.5469	3.5465
5-Minute Epoxy	4.7995	4.7952	4.7953	4.7953	4.7937	4.7939
Torrseal	4.5971	4.5907	4.5894	4.5889	*4.1439	4.1396
Armstrong	4.7209	4.7164	4.7150	4.7091	4.7086	4.7078
Red PVC	1.2233	1.2201	1.2194	1.2197	1.2218	1.2246
Yellow Teflon	0.3209	0.3299	0.3308	0.3302	0.3306	0.3311
White Fiberglass	4.4378	4.4307	4.4307	4.4306	4.4303	4.4302
Pink Fiberglass B	10.9212	10.9115	10.9095	10.9072	10.906	10.9049
Black Fiberglass B	2.2248	2.2224	2.2251	2.2223	2.222	2.2224
G10	11.4287	11.4211	11.4189	11.4174	11.4167	11.4169
FR4	1.305	1.3026	1.3026	1.302	1.3019	1.302
Ultem	3.512	3.5451	3.5434	3.5419	3.5412	3.5409
Noryl	4.2894	4.2819	4.2808	4.2805	4.2802	4.28
Epoxy Glass	0.944	0.9423	0.9423	0.9421	0.9419	0.9419
Polyimide Glass	0.7185	0.7162	0.7167	0.7162	0.716	0.7161
Polyimide Quartz	0.246	0.2453	0.2455	0.2453	0.2451	0.2455
Kapton	0.1123	0.1134	0.1126	‡ 0.1067	0.1065	0.1067
Ceramic Spacers	0.7288	0.7286	0.7285	0.7291	0.7291	0.729
Viton A	1.9849	1.9837	1.9831	1.9820	1.9803	1.9798
Glass Window	27.5125	27.5117	27.5118	27.5117	27.5118	27.5117
Silver Paint	5.4232	5.4214	5.4112	5.4109	5.4108	5.4108
Cathode Board	10.4084	10.4014	10.4014	10.3985	10.3970	10.3965

HEAT	Exposure time (hours)			
CONTROLS	0	20	40	60
Delta Bond Blue A	4.087	4.085	4.081	4.0826
EGA 148	3.169	* 3.164	3.162	3.1635
Epon Versamid B	3.179	3.179	3.177	3.1795
Clear RTV	3.524	3.500	3.497	3.4972
White RTV	3.465	3.462	3.456	3.4571
Hysol A	3.221	3.218	3.217	3.2179
KS-0002	3.593	3.584	3.581	3.58
5-minute Epoxy	3.251	3.251	3.25	3.2505
Delta Bond Blue	5.86	5.851	5.849	5.8496
EGA-148	4.759	† 4.78	4.773	4.7742
Epon/Versamid	4.615	4.61	4.607	4.6083
Hysol	3.855	3.850	3.843	3.845
5-Minute Epoxy	4.577	4.577	4.568	4.5704
Torrseal	4.772	4.767	4.765	4.7762
Armstrong	4.636	4.633	4.627	4.6297
Red PVC	0.874	0.870	0.867	0.8601
Yellow Teflon	0.522	0.521	0.520	0.5219
Speaker Wire	0.563	0.562	0.560	0.5627
White Fiberglass	4.423	4.415	4.415	4.4167
Pink Fiberglass B	10.396	10.386	10.380	10.3809
Black Fiberglass B	2.490	2.483	2.478	2.479
G10	12.930	12.918	12.913	12.9155
FR4	1.610	1.158	1.157	1.1567
Ultem	3.905	3.897	3.892	3.8953
Noryl	3.770	3.771	3.767	3.7693
Epoxy Glass	0.752	0.75	0.749	0.7509
Polyimide Glass	0.704	0.701	0.7	0.7019
Polyimide Quartz	0.238	0.238	0.237	0.2377
Kapton	0.056	0.056	0.056	0.056
Ceramic Spacers	0.560	0.560	0.558	0.5608
Viton A	1.995	1.994	1.992	1.9960
Glass Window	29.548	29.747	29.746	29.7471
Silver Paint	6.133	6.130	6.130	6.1311
Cathode Board	9.905	9.896	9.892	9.8935
Leak Frame	163.5			163.0735

Table 3: Loads used in the Stress Test (in kg)

Sample	Breaking Load		Test Load	
	Shear	Tensile	Shear	Tensile
Epon/Versamid	4	4	2	2
5 Minute Epoxy	10	14.5	8	13
Hysol	12	17	10.5	14.5
Torrseal	14.5	12	12.5	13
Delta Bond	19	13	12.5	10.5
Armstrong A12	19.5	17	17	13
EGA 148	25.5	13	17	10.5

In the table below, we list the strength at which our trial sample broke, the strength at which we tested the sample in each of the intermediate tests. At the end of the tests we will again apply weight equal or greater to the breaking strength until each sample breaks. It was our goal to test each sample at 90% of breaking strength but in the intermediate tests we did not test with any more than 13 kg. It is entirely possible that some glues actually gain strength with heat curing. In the hot control samples, the only sample that broke (EGA-148) was broken accidentally and a replacement sample that was added to the test survived quite well. In the TEA samples, the Hysol sample broke after the first 12-hour period. We measured this one to be good to 8 kg of shear stress and it broke when the full 10.5 kg was applied. A replacement Hysol sample did not break for the rest of the time. The Torrseal sample broke after the third 12-hour period (due to a tensile strength test) but its replacement sample has not yet broken. It is possible that this sample broke because the sample was not pointing straight down in the vise and some torque was generated on the glue joint.

All other samples performed quite well. It is rather puzzling that Delta Bond Blue adhesive held well in a G10-to-G10 seal but not when applied to the aluminum substrate in the adhesive test above. This may be due to the high aluminum oxide content of Delta Bond Blue causing it to make a bond to the oxide layer rather than the metal, which is broken by the heat/TEA combination.

6.6 Leak Test

The heat control leak test frame was exposed to 60 hours of heat controls at 100° C. In the last of the three 20-hour periods, the glass in the window cracked. This leak was patched but other leaks were also observed in the box, most notably at the EGA-148 seal but also at points along the Epon/Versamid seal as well. These leaks were of sufficient magnitude to be observed with Snoop.

The TEA sample frame, which used Hysol for the Ultem to metal and the Ultem to glass seals, was exposed to heat and TEA for 12 hours. After the first exposure period, a leak was visible in the Hysol seal by using "Snoop" which had not been visible before, and other leaks were forming.

At this point we can only conclude that the disparate coefficients of thermal expansion between the metal, glass, and glues cause the glue joints to break loose in time due to the large temperature variation.

6.7 Other Tests

We had a sample of the chromium and silver traces that will be applied to the CaF_2 window. These were etched on a glass window. Careful inspection with a microscope showed no visible effects from TEA exposure. Also we tested potential chamber wires of stainless steel, Gold/Rhenium, and Gold/Tungsten. Under microscopic examination they showed no damage.

We prepared two samples of the KS-0002 conducting epoxy on a glass substrate with lead wires. Initially the control sample had a measured resistance of 0.3 ohms and the TEA sample had a measured resistance of 0.2 ohms (as measured with a Wheatstone bridge.) After the exposure the control sample had resistance of 0.1 ohms and the TEA sample had resistance of 0.025 ohms. Such a decrease in resistance (implying an increase in conductivity) is expected, given the manufacturers specifications which state that it increases in conductivity when it is heat-cured. It should also be noted that KS-0002 maintained its adhesion to a Kapton piece during this test as well.

7 Schedule

Next week we will begin a continuous TEA run with all the samples that have been tested today, at 100° C, and hope to triple the exposure time achieved

to date. After that time we will do a lower temperature run (50°) focusing on the samples which showed strong effects at high heat.

8 Conclusions for first round

Since we began this study, several of the adhesives that we believed to be good in liquid TEA have begun to show effects. This includes Armstrong A-12, Delta Bond Blue, and Epon/Versamid. This round of tests has shown that there are also problems with Hysol. This leaves two crucial areas of the detector without a proven adhesive. The calculations in this paper and the experience that we have gained from this test show that liquid TEA is the ultimate test. We must decide whether or not we wish to go ahead and use the materials which have a clean bill of health in this study but are known to degrade in liquid TEA.

On the other hand, we are also beginning to see some of the effects that were also seen in liquid TEA, such as the degrading of Noryl, the teflon-coated wire, and the silver paint. So although calibration of the two methods may be difficult, it is not impossible.

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Figure 1: Effective exposure time as a function of activation energy for a 60-hour run at three different temperatures.