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Document Title

Performance of HPK Photodiodes with ShinEtsu Silicone Window

CHANGE HISTORY LOG

Revision	Effective Date	Description of Changes
1		Initial Release

1 Introduction

This document describes tests of the performance of Hamamatsu Photonics (hereafter, HPK) photodiodes with silicone optical windows, the commercial S5107 photodiode (PD) with 1 cm^2 active area (Figure 1), and the custom S8576-SP2 dual photodiode (DPD), which is the GLAST EM DPD with a silicone elastomer encapsulant in place of the standard, hard optical epoxy window (Figure 2). The silicone encapsulant is Shin-Etsu Chemical Co KJR 9022E silicone resin (hereafter, SE). This change in the optical window material from the GLAST EM DPD (S8576) was required due to the failure of the hard epoxy optical window to survive the qualification thermal cycling without cracking and delamination from the ceramic carrier.

We performed a number of tests of the performance of these PDs: the stability of the optical window over the GLAST qualification temperature range (–30C to +50C); its out-gassing properties; its compatibility with the material we have selected to bond PDs to CsI(Tl) crystals; the light yield of bonded PDs; the thermal stability of bonded PDs; the mechanical strength of bonded PDs. We have also begun tests of the radiation hardness of the SE window. Results of these completed tests are reported here.

We conclude that the SE window meets the requirements we have specified for the GLAST Calorimeter.

2 The problem

The hard epoxy window of the BTEM and EM dual PDs was not stable against thermal cycling. Diodes cycled over

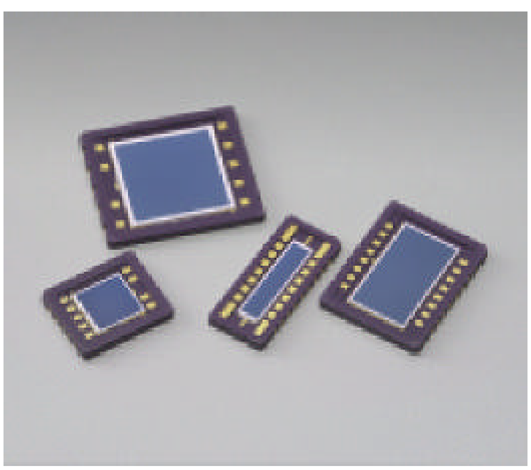

Figure 1. S5100 series PIN photodiodes. The S5107 (upper left) used in these tests is 10 x 10 mm active area.

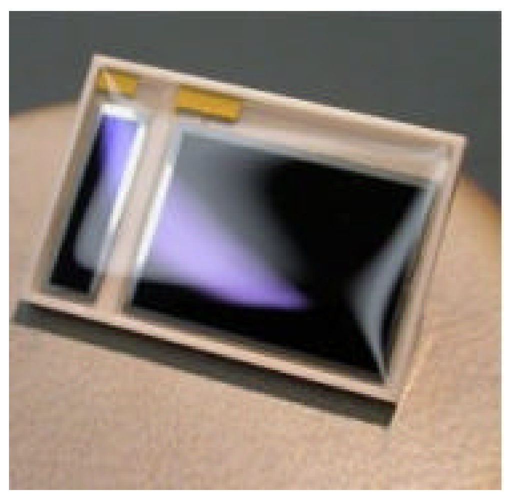

Figure 2: S8576-SP2 with SE silicone window

the Qualification range frequently showed "microcracks", severe cracks, and/or delamination of the epoxy window from the silicon wafer. With the severe cracking or delamination, electrical failure of the diodes could occur, as the wire bonds to the wafer were stretched or broken. We presume that these failures occurred because of a CTE mismatch between the hard epoxy and the ceramic carrier of the PD after more than 20 cycles. We note that such failures were somewhat surprising to HPK since they generally have no effect on the electrical or optical properties of the diode. In the most severe cases after many cycles significant delamination caused shearing of the wire bonds of the die to the carrier resulting in electrical failure.

The hard epoxy window was included in the LAT baseline diode because of experience with bonding diodes to CsI for the ISGRE experiment on ESA's Integral mission. The ISGRE experience indicated that more reliable bonds of PIN diodes to CsI were obtained when the diode bonding surface was flat. The required flatness was achieved by polishing the epoxy window, thus requiring a hard window material. This experience related to the hard epoxy resins proposed to bond the diode to the CsI. After much investigation in France and US, the CAL team decided that a silicone elastomer bonding material (Dow Corning 93-500) provided more reliable bonds and apparently was not sensitive to the concavity of the diode's unpolished optical window. Thus, the selection of the DC 93-500 bonding material permitted other options for the diode optical window material. The problems discovered in the thermal cycling of the S8576 diodes required a change to alternate encapsulant.

HPK offers an alternate optical window, the SE silicone window, with a significantly broader operational temperature range for surface-mount devices. We procured a number of the commercial S5107 PDs with SE window. This diode has 1 cm^2 active area and is mounted in a ceramic flat pack (see Figure 1). Additionally, HPK manufactured 10 custom S8576-SP2 dual photodiodes (the EM DPD with an SE window, Figure 2) for us. These S8576-SP2 diodes exhausted the available carriers at HPK for manufacturing additional test specimens. Other empty carriers were sent to France for investigations of alternate encapsulant solutions as potential backups.

3 Thermal stability of SE window

We tested the thermal stability of the SE window by cycling four S5107 commercial PDs and four S8576-SP2 custom DPDs over the Qualification range (–30C to +50C, 1 degree per minute, 30 minute dwells at extremes, dry nitrogen purge). The commercial PDs were cycled 180 times. The custom DPDs were cycled 100 times. We inspected each sample visually and measured its leakage current before and after the tests.

No cracking or delamination was visible under 10x magnification, and no change in leakage current was observed after cycling. We conclude that the SE window is stable under the GLAST temperature range.

4 Out-gassing

Out-gassing tests were performed at GSFC on samples of the SE material prepared by HPK. The samples were tested for Total Mass Loss (TML) and Condensible Volatiles (CVCM) after three different treatments: no bake out, bake-out for 24 hours at 125C in vacuum, and bake-out for 24 hours at 175C in vacuum. The maximum acceptable limits are 1.00% TML and 0.10% CVCM.

Results of the TML and CVCM tests are given in Table 1. The material fails CVCM without bake-out, but it readily passes the requirements on TML and CVCM under both bake-out regimes. Fred Gross, GSFC, is confident that the material will meet the requirements if the DPDs are baked for 24 hours at 175 C at one atmosphere. HPK has indicated that baking at 175C for 24 hours is not a problem for the DPDs. (The complete test report can be found in GSFC Document 28213.)

Treatment	**Average TML (Reqmt < 1%)**	**Average CVCM (Reqmt < 0.1%)**
No bake out	0.68%	0.43%
Bake out 24 hrs at 125C in vacuum	0.15%	0.05%
Bake out 24 hrs at 175C in vacuum	0.07%	0.01%

Table 1: Results of out-gassing tests on samples of SE material prepared at HPK.

We conclude that the SE window material satisfies out-gassing requirements.

5 Bond compatibility

It is essential that the SE silicone window be compatible with the adhesive we have chosen to form the bond between photodiode and CsI(Tl) crystal, the Dow Corning silicone encapsulant DC93-500. We created a number of test bonds between S5107 PDs and CsI(Tl) crystal samples, and we found no problems. The SE silicone does not prevent the Dow Corning silicone from curing, and once cured, the two silicones adhere strongly to one another. We conclude that the two materials are compatible.

We created the test bonds with the Spacer Method, discussed in some detail in Appendix 1. In brief, a measured volume of 93-500 is delivered to the surface of the PD, and a crystal is lowered to a fixed, measured height above the diode face. The 93-500 flows to fill the volume between the PD and crystal, but the sides of the bond volume are not constrained in any way. Surface tension holds the bond material in place until it cures. Prior to bonding, the crystal surface is roughened and primed with DC92-023. Because the SE window and the wire bonds it protects are fragile, we did not roughen the surface of the PD. The Dow Corning data sheets say not to prime cured silicone surfaces before bonding – which is consistent with advice we received from Ben Rodini of Swales – so we did not prime the PD window. (During later EM CDE production, Swales technicians applied primer to the SE silicone on three DPDs. The surface of the window immediately became cloudy, and the subsequent 93-500 bond was slow to cure. We conclude that indeed it is a mistake to apply primer to the silicone window of the DPD.)

The test bonds with S5107 PDs all cured properly, and there was obvious adhesion between silicones. We subsequently tested the optical and mechanical properties of the bonds, as discussed below.

6 Optical properties

We tested the optical quality of bonds made to commercial S5107 PDs and custom S8576-SP2 dual PDs with the SE window. As described in Sections 6.1 and 6.2, bonded diodes meet the optical requirements for the CAL.

6.1 Light yield

The FM CDE specification requires scintillation light yields of >7000 e/MeV in the big PIN and >1100 e/MeV in the small PIN. To test whether the SE window is sufficiently transparent to CsI(Tl) scintillation light, we measured the light yield in six DPDs bonded to three EM CDEs at Swales. As part of the verification of the EM CDE build, we placed all EM CDEs in a muon telescope with calibrated electronic gain. From the amplitude of the muon peak and the known most-probable energy deposition of sea-level muons, we calculated the light yield of each big and small PIN diode of each CDE.

One of the bonds to an SE DPD was visibly delaminated, and that CDE was rejected from the EM build. We therefore compared the light yield of the five good SE bonds to the ensemble of all EM bonds. The average light yield from the five good bonds is 7950 e/MeV, while the average over the entire ensemble of EM CDEs is 8500 e/MeV. The average light yield from the five small PINs is 1350 e/MeV, while the average over the entire ensemble of EM small PINs is 1500 e/MeV. Thus the light yield from the EM DPDs with SE windows is ~90% of the light yield from the standard EM DPDs, with their hard epoxy window.

As a cross check, we can compare the amplitude of the 662 keV line from ^{137}Cs measured in the S5107s bonded to CsI(Tl) crystal cubes (discussed in Section 5) to that measured in earlier test samples of BTEM DPDs bonded to CsI(Tl) crystal cubes. Note that this is only a rough check, since the geometries and colors of the PD carriers are different (S5107 is dark brown; BTEM is white). We find that the amplitude in the S5107s is ~90% of in the BTEM DPDs, which is surprisingly consistent with the result from the EM CDEs.

Bonds made to EM DPDs with SE windows therefore meet the FM light yield requirements.

6.2 Thermal stability of optical bond

To verify the stability of the optical bond throughout the thermal environment for GLAST, we measured the light yield of a number of bonds through at least 100 thermal cycles. We found no significant changes in light yield, and therefore no evidence for optical failure of the bond. We conclude that the bonds are thermally stable.

The test samples used for this study were nine S5107s bonded to CsI(Tl) crystal cubes (discussed in Section 5). The crystals were wrapped in Tetratex and adhesive aluminized Mylar. They were temperature-cycled in a Tenney thermal chamber over the range –30 to +50C at a rate of change of 20C per hour, with dwell times of 1 hour at each temperature extreme. The Tenney chamber was constantly flushed at a moderate (but unmeasured) rate with dry nitrogen, and the temperature and humidity were logged with a Dickson paper recorder. The relative humidity was at all times below 6% (the Dickson meter did not register <4% RH). After a specified number of thermal cycles, the test samples were removed from the Tenney chamber and illuminated with a ^{137}Cs source. We used the centroid of the 662 keV line to measure the light yield (in arbitrary units).

In the past four years, we have made more than 150 similar PD + CsI test samples for thermal cycling, with a variety of bond materials and PDs. Almost without exception, good optical bonds in these samples show a decline of ~5% in light yield during the first 10 thermal cycles, followed by a plateau of constant light yield with subsequent thermal cycling. We attribute this consistent, initial decline to degradation of the crystal surface from minor hydration and adhesion of the Tetratex wrapper. In contrast, bonds that fail during thermal cycling show a drop of ~20% in light yield.

Figure 3 shows the light yield, in units of the fraction of the signal prior to thermal cycling, for the 9 thermal test samples and one control sample (labeled "C029") that was not thermally cycled. The test samples all show the typical decline of ~5% and plateau in light yield, while the control sample remains essentially constant. It is therefore apparent that DC93-500 bonds between CsI(Tl) and the SE window are stable against thermal cycling.

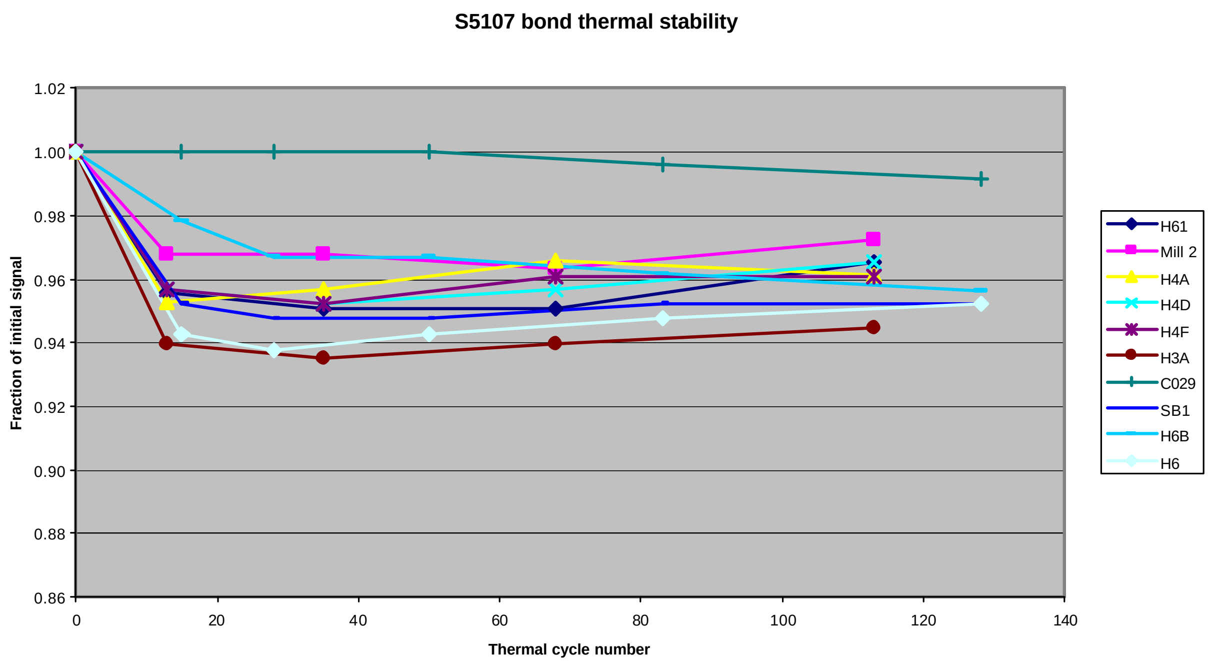

Figure 3: Light yield as a function of thermal cycle number. After more than 100 cycles, bonds retain ~95%of their original signal, which is consistent with previous results with PDs with hard epoxy windows.

7 Mechanical strength

The diode bond must be strong enough to survive handling loads and the stresses induced by differential expansion between the CsI and diode carrier over the Qualification temperature range. We have specified that the FM DPD bond have a tensile strength of 10 N or greater and a shear strength of 0.12 N/mm^2 or greater. We measured the tensile or shear strength of a number of test bonds between S5107 PDs and CsI, and found that they significantly exceeded the strength requirements, as described below.

7.1 Tensile strength

We measured the tensile strength of five bonds with a Chatillon Dynamic Load Test Stand. The test configuration is shown in Figure 4. The piston was attached to the back of the PD with cyanoacrylate, and an increasing tensile load was applied to the diode until the optical bond failed. The loading force was logged to a controlling computer. We inspected each bond after failure to determine whether the failure occurred within the DC93-500 bonding material or at the interface with the diode or crystal.

Table 2 gives the tensile strength of the five test samples and a description of where the failure occurred – in the DC93-500 bond material itself, or at the interface to the diode or crystal. Four of the five samples failed at >150N, which is more than 15 times the tensile strength requirement. The fifth failed at 20-40 N (the precise value is uncertain because the readout was noisy just as the load was applied). While this still exceeds the strength requirement, it is obvious that this was not a good bond – the failure was at the CsI face and unrelated to diode window material. We do not have an explanation for this bond weakness. Indeed this was the first bond we made to an SE window, so perhaps there was

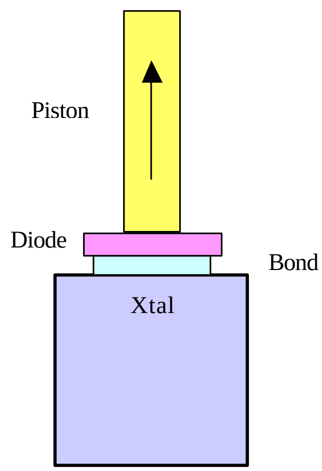

Figure 4: Cartoon of tensile strength test configuration. A measured load is applied to the piston normal to the plane of the PD, increasing until the diode bond fails.

some variation in the bonding process in that particular instance. All of the bonds failed at either the diode or crystal interface, which indicates that the adhesion between the two silicones or between the DC93-500 and the primed CsI(Tl) is not as great as the cohesion of the DC93-500 itself.

Crystal ID	**Diode ID**	**Failure Description**	**Tensile strength (lbf)**
H4D	1-05	Clean break at xtal face	20-40
HUD	1-11	Break at xtal face, one 2 mm wide stripe of 93-500 on xtal face	160
H4F	1-06	Clean break at xtal face	220
H6	1-08	Clean break at diode face	310
H6B	1-12	Clean break at diode face	240

Table 2: Tensile strength of bond samples

7.2 Shear strength

We measured the shear strength of four bonds with a Chatillon Dynamic Load Test Stand. The test configuration is shown in Figure 5. The piston was aligned with the plane of the PD, and an increasing shear load was applied to the diode until the optical bond failed. The loading force was logged to a controlling computer. We inspected each bond after failure to determine whether the failure occurred within the DC93-500 bonding material or at the interface with the diode or crystal.

Table 3 gives the shear strength of the four test samples and a description of where the failure occurred – in the DC93-500 bond material itself, or at the interface to the diode or crystal. All four samples failed at >0.8 N/mm2, which is more than 6 times the shear strength requirement. All of the bonds failed at the diode interface, which suggests that the adhesion between the two silicones is the weakest interface.

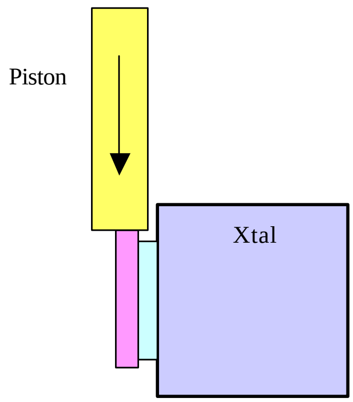

Figure 5: Cartoon of shear strength test configuration. A measured load is applied with the piston in the plane of the PD, increasing until the bond fails.

Crystal ID	**Diode ID**	**Failure Description**	**Shear strength (N/mm^2)**
H4F	1-17	80% clean break at diode face, 20% at xtal face; partial cohesive failure	1.2
H3A	1-14	Clean break at diode face	1.7
H61	1-10	Clean break at diode face	0.91
SAM	1-07	Clean break at diode face	0.80

Table 3: Shear strength of bond samples

8 Radiation hardness

We created four samples to test the radiation hardness of the SE window. Each test sample consists of a CsI(Tl) crystal bonded to two PDs, a reference PD with a hard epoxy window and the test S5107 with SE window. Although the tests have not yet begun, we will measure the light yield in the two diodes of each sample as a function of radiation dose up to 10 kRad. The experiment should begin in mid February, when NRL's Co^{60} well becomes available.

9 Conclusion

We conclude that the SE window meets the requirements set for GLAST DPDs. The SE window remains stably attached to the silicon die and ceramic carrier over the GLAST temperature Qualification range (–30C to +50C). After bake-out, it is low out-gassing. It bonds well with the DC93-500 silicone encapsulant, forming an optically clear and mechanically strong bond.

While these tests provide reasonable confidence that this new optical window material meets all the GLAST requirements so that we can proceed with the procurement, they do not replace the formal evaluation and qualification of the flight DPD which will occur in France. We will receive in late February 184 prototypes of the flight DPD, S8576-01, for evaluation in France. At the same time, unfortunately, we must proceed with the flight DPD specification to meet the first CAL flight module schedule milestones. We expect to have results from the prototype evaluation before the encapsulant is actually used for the first flight units. The fabrication of the ceramic carriers for the DPD consumes the 1st two months from receipt of order at HPK.

1 Appendix: Bonding methods

We created these bonds on a bench top in the lab at NRL. The room is not clean, and it is not strictly climate-controlled. The temperature ranged from ~21C to ~24C. The relative humidity ranged from ~20% to ~45%. We record temperature and humidity readings for this room taken every morning (about 06:30 am), as well as daily min and max values.

We made a good effort at keeping clean, standard lab practice. We used only stainless steel tools, glass dishes, and plastic micropipettes. We cleaned tools and dishes with pure ethanol. We wore powder-free nitrile gloves.

We used Dow Corning silicone encapsulant DC93-500 with primer DC 92-023. We mixed the 93-500 in the prescribed 10:1 ratio, typically 5 g base to 0.5 g hardener, measured with 0.01 g precision. We mixed the encapsulant in a glass petri dish by hand with a stainless steel spatula for ~5 minutes, and we pumped on the mixture in a bell jar for typically ~20 minutes until all bubbles were gone. We typically released the vacuum two or three times to help free the bubbles. We pumped for five minutes after all bubbles were gone. We kept the unused portion of the mix in its glass petri dish to ensure that the mix cured.

We delivered the 93-500 with a “plastic” micropipette (Microman model M1000 with CP1000 polypropylene capillary and polyacetal piston tip). We applied the 92-023 by wiping the crystal surface with a soaked clean-room swab.

We prepared the diode surface with a very gently wiping with a clean-room swab moistened with 100% ethanol.. We allowed the diode surface to air dry.

We prepared the crystal surface by polishing for a few minutes with 100% ethanol on a cotton ball in random circular motions, then roughening with 240-grit aluminum oxide paper soaked in 100% ethanol for ~30-45 sec in random circular motions, then cleaning with a clean-room cloth soaked in 100% ethanol. This process appeared to leave a small amount of CsI powder embedded in the grooved surface. We primed the crystal surface with two passes of a clean-room swab soaked in DC92-023.

In the spacer method, a measured amount of 93-500 is delivered onto the diode as it is held horizontal, and the crystal is lowered to a known, fixed distance above the diode surface. Surface tension and capillary action cause the 93-500 to fill the volume between the diode and crystal, and if the proper amount of encapsulant is used, it conforms to the rectangular shape of the diode.

The procedure we used is as follows.

1. Place ethanol-cleaned diode on a horizontal surface between standoffs that form the spacer to suspend the crystal above the diode. Standoffs are stacks of two or three glass slides with paper shims to create a plane ~0.6 mm above the diode face.
2. Prime CsI crystal face with 93-023. Xtal has been roughened with 240-grit paper and cleaned with ethanol. Wait 1 hr.
3. Apply DC93-500 to primed diode to make the total bond + primer height ~0.6 mm. The 93-500 forms a bulge on the surface of the diode.
4. Place one edge of the crystal against the glass standoff and hinge the crystal down into place above the diode and 93-500.

Presumably because of its high viscosity, it is difficult to deliver a precisely measured amount of 93-500 with the micropipette. The delivered amount is systematically less than the dialed-in amount, and there is some significant variation from one delivery to the next (at least a few percent, probably less than 10%). We had to wipe the pipette tip to remove the excess 93-500 clinging to the outside of the tip after filling.