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Silicone "Cookies" for Blowfish Detectors (draft)

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Introduction

The detectors in the Blowfish neutron detector array have photomultiplier tubes (PMT) coupled to their cells via silicone wafers or "cookies"[1]. During a PMT upgrade, it was noted that some cells had lost their cookie. Also, there are plans for a future upgrade of Blowfish to add 40 more cells. More cookies need to be made. This document provides instruction on how we fabricated our own silicone cookies.

1 Requirements

The material for the silicone cookies is Dow Corning **Sylgard 184**[2][1]. This is a two part kit consisting of a base and curing agent mixed in a 10:1 ratio. This silicone elastomer has a refractive index of 1.43, which is a close enough match to the glass of the PMT and the plastic of the cell. It is quite resistant to radiation damage. It is very sticky yet does not bond to smooth surfaces and has significant durometer hardness, which makes it ideal for our purpose of reusable optical interface. It does not require air to cure (i.e. can cure in a totally sealed environment), so it can be sandwiched between two surfaces. Curing can be accelerated by heating, up to 150 C. Otherwise, the preliminary cure takes 24 hours at room temperature. However, it takes 7 days to attain its full strength. Room temperature curing has the advantage that bubbles have enough time to percolate out.

Of particular concern is that *Sylgard does not cure properly when in contact with certain types of plastics and all forms of rubber.* All hardware used to form the Sylgard as it cures must be made of materials other than plastics and not have any plastic coating. Glass and solid metal parts and equipment are ideal. Care should be taken to clean all parts of oils and other organic materials used in the manufacture of the parts. Do not assume a brand new part or tool is clean.

From the functional perspective, the cookies must have smooth and flat surfaces for optimum contact with the PMT and cell faces, and should contain few bubbles and other inclusions that may block or scatter light away from the PMT photocathode. This rules out many types of mold materials and manufacturing methods. One cannot make a cylinder, then slice it because this technique always results in striations. The only guaranteed types of materials are glasses and metals. Stock aluminum is not polished enough, and is easily scratched. Glass is typically used for optical purposes, hence glass tends to be very smooth. Some means of retaining the initially fluid Sylgard is needed, so it would be convenient if the glassware was a prefabricated unit, similar to glass cookware. However, the latter tend to be rather nonuniform, even if smooth.

Hence, it was thought that the base of a form for the silicone cookies should be a sheet of glass. The sides could be some sort of aluminum attachments, perhaps even aluminum foil wrapped around the edges. It turns out an unpainted, unvarnished aluminum picture frame with spring clips is an adequate form. At least 16 cookies can be made from a sheet coming from an 8x10 frame.

The two faces should be fairly parallel, so whatever work surface the form rests on should be level.

[1]Purchased from E.B. Peerless in Calgary.

If room temperature curing is used, a vacuum system is not needed to de-bubble the mixture before pouring, for thin layers of silicone.

Some thought may be needed for a form for individual cookies. Of particular concern is the smoothness of the sides. If a cutter results in streaked, ragged, or otherwise unsmooth sides, an alternative method method may be called for. To minimize sticking of the glass plate to the bottom of the form and to provide a place for excess liquid to flow off the top, the form should have a lip around the cookie form on both sides.

2 Manufacture

2.1 Preparation

There are 3 issues that require preparation. The approximate quantities needed for the pour should be computed. The frame needs to be prepared the day before the pour. The work area must be level.

2.1.1 Quantity: Volume versus weight

Dow Corning recommends that Sylgard be made from a 10:1 base:hardener mix, **by weight**. It would thus be best to measure the components on an electronic weigh scale with at least 0.1g resolution. How much is mixed depends on the size of the form and the thickness of the cookie. A loss of 10% is also expected to cling to the beaker. Since the specific gravity of Sylgard is 1.05, so the volume of the form converts nearly directly to the overall weight:

$$weight = 1.05 \times width \times height \times thickness \text{ in g}$$

So, for an 8x10 mold with 0.4cm thick cookies, the ideal weight must be

$$weight = 1.05 \times 8(2.54) \times 10(2.54) \times 0.3 = 4 = 216.8g$$

With the 10% overhead, the weight for mixing comes to

$$weight = 216.8 \times 1.1 = 238.5g$$

With a 10:1 component ratio, that means one should start out with the mixing weight of the base of

$$weight_{base} = 238.5(10/11) = 216.8g$$

and then top that up to a mixing weight of 238.5g with curing agent. Table 2.1 provides some example weights for frames of various dimensions.

However, it is impractical to portion out exactly the amount of base needed because it is rather viscous. Fortunately, the thickness of the cookie is not critical, so a 10% overshoot or undershoot with the base component is tolerable. The curing agent is significantly thinner, and needed in much smaller quantities, so it can be dispensed with a pipette (which gives good control) to the proper (recalculated) final weight.

frame (inches)	thickness (mm)	ideal weight (g)	mixing weight base (g)	mixing weight total (g)
8x10	3	162.6	162.6	178.8
8x10	4	216.8	216.8	238.5
3.5x5	3	35.6	35.6	39.1
3.5x5	4	47.4	47.4	52.2
4x6	3	48.8	48.8	53.7
4x6	4	65.0	65.0	71.5
11x14	3	313.0	313.0	344.3
11x14	4	417.3	417.3	459.0

Table 2.1: Examples of silicone weights for frames of various dimensions.

2.1.2 Preparing the frame

The following tools and supplies are required:

- lint-free paper towels
- isopropyl alcohol
- aluminum foil
- spatula marked "Base"

An 8x10 inch aluminmum picture frame was purchased[1] rather than having one custom built in the shop. It consists of a glass plate, aluminum frame, cardboard backing and metal spring clips (see Fig. 2.1). The spring clips push the glass plate (and backing) against the inside edge of the frame.

The glass plate should be removed from the frame and cleaned. Peal off any gross quantities of hardened silicone first. For thinner areas, firmly rubbing with a clean paper towel should remove the silicone. Any remaining film or unhardened silicone can be removed with isopropyl alcohol on a paper towel, again with firm rubbing. Then reassemble the frame.

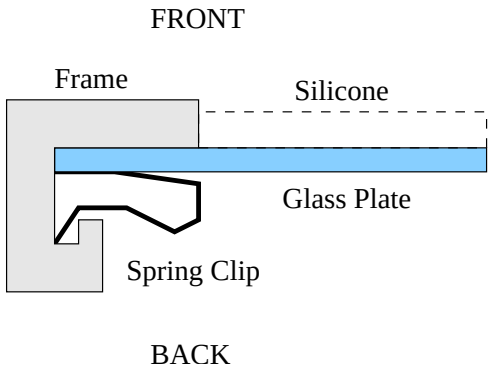

Figure 2.1: Cross-section view of frame used as form

Even though the Sylgard mixture is initially quite viscous, it still flows quite well. All joints and cracks should be sealed previous to the initial pour with a small quantity of Sylgard. See 2.3 for mixing instructions, however, only make a 1 g batch and use the spatula marked "Base" to dispense the base onto a piece of aluminum foil (instead of beaker).

[1]The supplier was **Michael's**, a craft supply retail chain. Be sure that either the backing is solid (not corrugated cardboard) or get a replacement backing board. **Michael's** had such items.

Due to the recessed geometry of picture frames, applying the silicone prior to assembly risks smearing the plate during the assembly. The seal has to be applied after assembly.

Using a square ended spatula, draw a bead along the inside edge of the frame, where it contacts the glass. Also draw a bead in the inside corners of the frame, where the frame parts join. Actually, the term "draw" is a bit misleading, since the spatula cannot dispense fluid continuously. Nor can one drop a large enough quantity of fluid to draw from, since the seam must not spread out onto the plate. One technique is to dab the tip of the square spatula in 1 to 2 mm of fluid, then dab one small section of the joint to be sealed. To minimize the chance of accidental contact with the plate, the motion should be horizontal towards the top of the edge, contact, slide down to the joint (see Fig. 2.2). Excess along the top of the edge will flow down to the seam. The quantity tends to be slightly more than needed so that after a series of dabs, the spatula can be run over the seam to smooth it out and fill any voids. This provides enough sealant to the joint while minimizing the spread onto the plate.

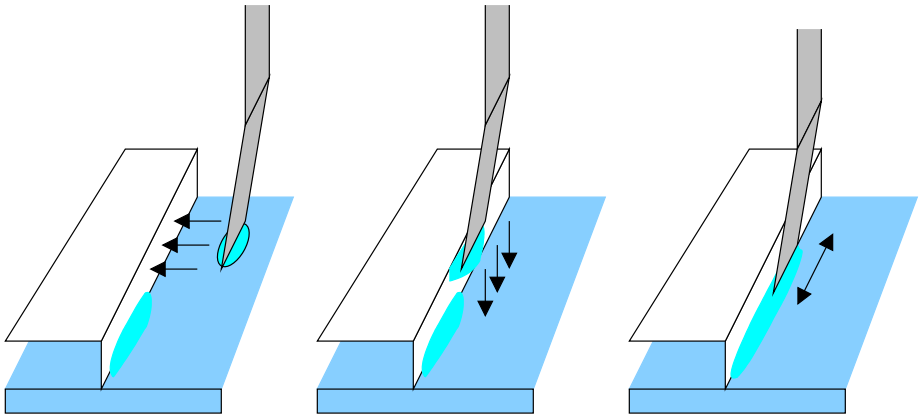

Figure 2.2: Drawing a bead on the frame

Leave the assembly overnight to at least partially cure before attempting a full pour.

2.1.3 Preparing the work area

The following tools and supplies are required:

- lint-free paper towels
- small carpenter's level or clean ball bearing or marble
- shims

In general, the work area should be clean, with as little dust as possible.

Since one face of the sheet of silicone is left open, the uniformity in thickness of the cookies will depend on a level glass plate. No work surface is perfectly level, so the plate should be leveled by shims under the frame. A small carpenter's level placed on a lint-free paper towel can be used to measure the level. A clean (with alcohol) ball bearing or marble can also be used.

2.2 Mixing the Compound

The following tools and supplies are required:

- Sylgard 184 Base
- Sylgard 184 Curing agent
- glass beaker
- steel spatula
- steel spatula marked "Base"
- glass pipette marked "Curing agent"
- pipette bulb
- weigh scale with at least 0.1g precision

1. turn on weigh scale and allow to warm up for 10-15 minutes
2. place empty beaker on scale and *zero* the scale
3. open the jar of the base, and either
    a) for large quantities, pour the amount that is needed down the spatula to within 10%.
        - **Never** stick a non-designated spatula into the jar.
        - Try to *avoid getting the base on the wall of the beaker*, since any unmixed base flowing from such a spot can cause inhomogeneities in the final product.
    b) for small quantities (e.g. less than 5 grams), use the round "Base spatula" to "scoop" base from the jar and release base into small container in drops. One drop is about 1 gram.
4. wipe any excess off the outside of the jar and close it.
5. recalculate the required overall weight (weight of base + 1/10 weight of base)
6. open the vial of the curing agent
7. depending on the quantity
    a) for large quantities use a pipette bulb
        i. place the pipette bulb on the top end of the pipette (note, it does not fasten to the pipette, so do not hold the assembly by the bulb alone).
        ii. squeeze the bulb then insert the tip of the pipette into the vial.

iii. slowly release the bulb until the needed amount of curing agent is drawn or 10 ml, which ever is smaller. *Do not allow liquid into the bulb.*

iv. quickly remove the bulb and cover the end of the pipette with your thumb. This holds the liquid in the pipette.

b) for smaller quantities of base,

i. it may suffice to just immerse the pipette, withdraw from the vial, and drop curing agent one drop at a time

ii. one drop corresponds to about 0.5 grams and the capillary effect can draw more than that.

8. withdraw the tip of the pipette from the vial and move to the beaker

9. while watching the scale, release your thumb and let the liquid flow out of the pipette until the required overall weight is obtained. Do not be alarmed by an additional 10% of curing agent (i.e. 1:100 extra weight). Try to *avoid getting any curing agent on the wall* of the beaker.

10. for larger volumes of curing agent, do multiple draws (steps 7(a)ii to 9) until the required overall weight is attained.

11. Stir the mix with the square spatula thoroughly. Sylgard has a two hour working time, so do not hesitate to mix for longer than you might thing is necessary. 5 minutes is not unreasonable. It is impossible to stir the mix without introducing bubbles, so don't bother trying to minimize bubbling. Start stirring with a folding motion to draw curing agent down. Then stir in small circles, moving around the beaker, and do so at varying elevations in the beaker. Occasionally, use a scraping motion to draw unmixed material away from the beaker walls.

2.3 Pouring the Form

The following tools and supplies are required:

- frame with glass, assembled

Note, **never** use a mold release agent, since there is a good chance the agent will inhibit proper curing. The procedure is then:

1. Pour the mixture into the form, starting from the center and spiralling outwards in a rectangular pattern. The corners may initially need more than the central areas.

2. If there is still a noticeable tilt, shim up the end with excessive silicone until the surface looks level.

3. Place dust cover. Make sure dust cover does not touch the silicone.

4. Do not touch for at least 24 hours.

2.4 Cleanup

On their own, each silicone component poses a slip hazard, and may pose some health risks if ingested. All tools and parts, and any excess or spillage, should be cleaned up. Sylgard generates hydrogen gas when in contact with oxidizers (e.g. water). Do not use water for cleaning anything on which there are uncured Sylgard components.

The following tools and supplies are required:

- cleaning jar, labeled for curing agent
- isopropyl alcohol
- lint-free paper towels
- aluminum foil
- shim

2.4.1 Cleaning the pipette

1. pour about 10ml of isopropyl alcohol into the designated jar
2. immerse tip of pipette in alcohol
3. use pipette bulb to repeatedly flush the pipette with less than 10 ml of alcohol. Be careful to **not** get liquid in the bulb.
4. repeat 1 to 3 until there is little beading of alcohol inside the pipette
5. use a lint free paper towel to wipe pipette
6. store pipette with tip wrapped in foil (do not mix up foils)

2.4.2 Cleaning the spatula

1. wipe off excess silicone with dry lint-free paper towel. This may require a couple of towels.
2. pour some alcohol onto a lint-free paper towel
3. give spatula a final wipe
4. 24 hours later, rub off any remaining silicone

2.4.3 Cleaning the beaker

1. cut a piece of aluminum foil twice as large as the beaker opening
2. lay out aluminum foil with edges curled up
3. place beaker on foil inverted, so that excess silicone will run onto foil. Shim up the side opposite of the spout to tilt the beaker (this forces liquid on the bottom to flow towards the spout)
4. leave for 30 minutes
5. after 30 minutes, wipe remaining excess from beaker with lint-free towels, and set aside the foil with excess silicone to cure overnight before disposal.
6. rinse beaker with small quantity of isopropyl alcohol
7. wipe dry with lint-free towel
8. if there is still a film, repeat rinse and dry
9. cover top of beaker with a clean piece of Al foil

2.5 Cutting the cookies

The following tools and supplies are required:

- circular cookie cutters
- 2 plexiglass sheets for storage
- scalpel or craft knife
- isopropyl alcohol
- distilled water
- mild liquid dishwashing detergent

The silicone sheet must first be extracted from the form and glass plate, and then cookies can be cut on one of the pre-cleaned sheets of plexiglass. Avoid touching the silicone since the cure may not be complete. The Physics Machine shop milled a steel cutter specifically for this purpose, basing it along the lines of a stamping tool (see Fig. 2.3 and Fig.). This proved to be insightful since the silicone turned out hard enough that significant pressure is required, probably enough to damage a sheet metal or plastic cutter. An inadvertent design feature are holes drilled through the back of the cutter. The original intent was to allow for a prod to push cookies out that stuck to the cutter. Silicone cookies tend to stick to the cutting board. However, in the process of pressing the cutter, an air tight seal is formed at the edge. The holes act as a release for air trapped in the cutter volume.

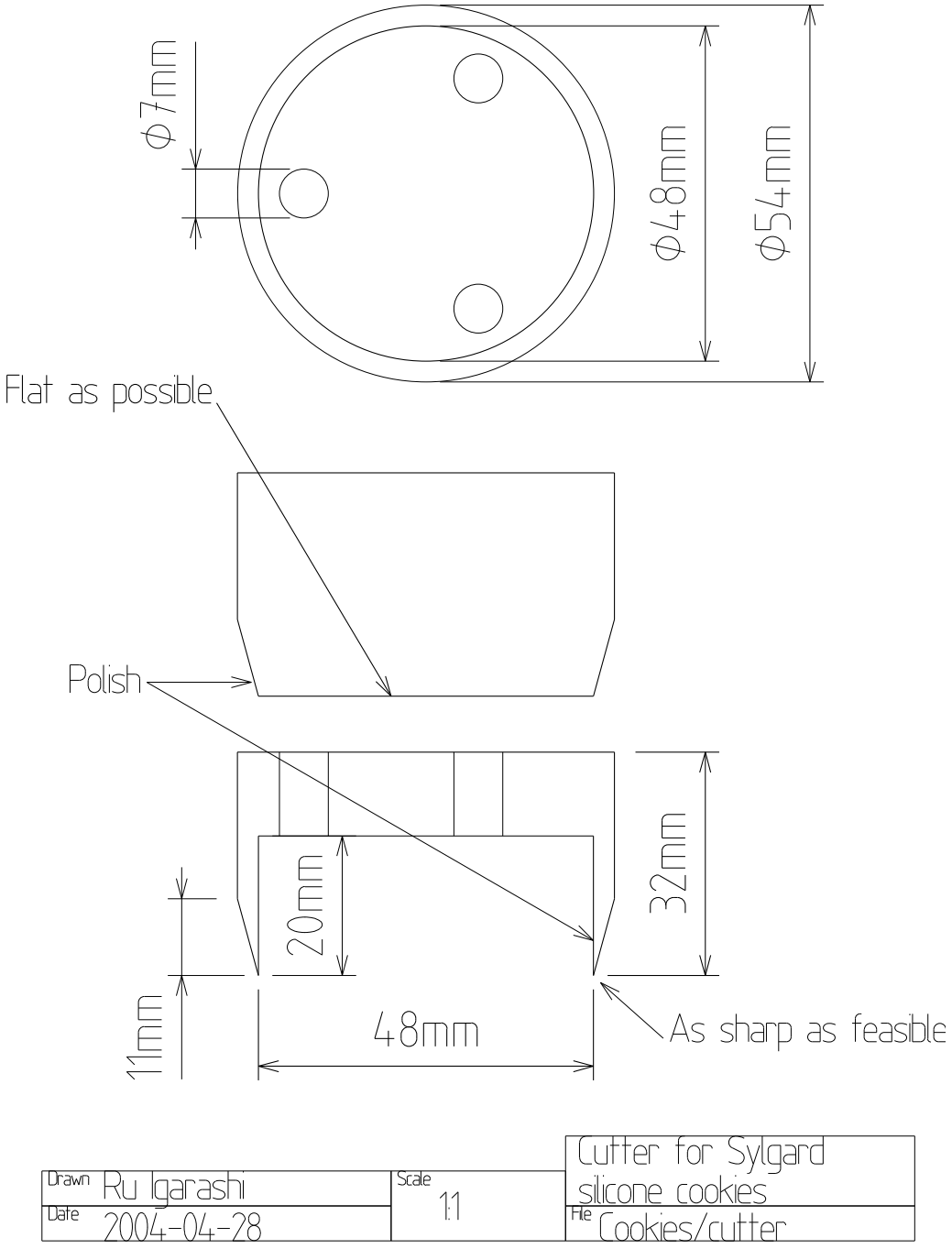

Figure 2.3: Cookie Cutter

Figure 2.4: Photo of cookie cutter, blade side up

2.5.1 Releasing the silicone

1. Gently cut around the edge of the frame with the knife
2. Use the knife to pry at one corner until the silicone begins to peal off the plate. The corner won't be part of any cookie, so if any handling is necessary, do it at the corners.
3. Carefully peel the silicone off the plate. Watch for the first signs of tearing. If you see any, redo the cutting around the edge.
4. Transfer the silicone to the cutting board. It should not stretch much in transit if it is as thick as 4mm, but be aware that you want to minimize deformation (the cookies will turn out deformed when they start to relax)

2.5.2 Cutting the cookies

Wait at least 2-3 days before cutting the cookies. While the silicone is softer, the optical surfaces are also easier to damage (pitting, printing) from simply touching them. After a few days of hardening, the surface is hard enough to resist contact damage.

It is important to clean all tools and hardware before cutting. The cutting board and storage boards need to be free of dust, debris, and residue. The cutter must be totally clean (even the side you push down on).

1. swab detergent liberally on both the inside and outside of the edge of the cutter. Do this for every cookie.
2. place the cutter on the silicone where the next cookie will be cut out. Be sure to give at least 2 mm clearance from any edge of the silicone.
3. press down evently. You should feel the silicone give way. You may hear or feel the cutter make contact with the cutting board.
4. rotate the pressure around the circumferance of the cutter, and continue until you feel or hear contact with the cutting board.
5. where possible, carefully pull back excess silicone to see if the cutter is indeed at the bottom. If not continue pressing.
6. If it has reached the bottom, remove the cutter. This may peel the cookie off the cutting board but usually does not.
7. If the cookie is stuck to the cutting board, carefully pull the excess silicone, where possible, then try to lift one edge of the cookie. Beware of tearing: if the cookie *looks* like it is still attached to the bulk silicone, assume it has and repeat the cutting procedure concentrating on the attached area, but be very careful to position the cutter into the existing cut.
8. Once the cookie is free, clean it, first with soapy distilled water, then rinse with alcohol.

9. Transfer it to the storage plexiglass. Try to ensure air bubble are not trapped.

2.5.3 Cleanup

When all cookies have been cut:

1. Clean the unused side of the cutting board and use that as a cover for the storage plexiglass.
2. Wash the detergent off the cutter, and insert protective foam.

Bibliography

[1] "Blowfish Detector Upgrade Manual", B. Bewer, J. Robb, W. Wurtz, U of S Subatomic Physics Internal Report SPIR-114, 2003.

[2] Dow Corning Sylgard® 184 Silicon Elastomer Base & Curing Agent product guide.