Home Made Silicone Rubber Embedding Molds

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Silicone rubber embedding molds may be inexpensive In North America, but are not necessarily so elsewhere in the world. Further, commercial molds are available only in standard sizes and only with standard sizes and shapes of cavities for specimen embedding. If, however, one needs to embed specimens of nonstandard size or shape, wants more embedding cavities per mold than usually available, or is working where commercial molds are expensive or not easily available, then all is not lost. Embedding molds for specimen blocks can easily be made in the lab from silicone rubber. Further, making embedding blocks allows the use of different silicone rubbers which would be of value with unusual embedding resins, or when embedding must be done in unusual environments (such as extreme cold or heat).

The Primary Mold

Making embedding molds first requires constructing a "negative mold" for casting the silicone to make the final mold. This primary (first) mold can be constructed of Plexiglas or wood covered with a very smooth surface such as melamine or PVC plastic. "Wood" made of compressed and glued wood chips is not recommended for this first mold. PVC or hard PVC (Acryinitrile-styrole-Acrylnitrile), or resins like Duroplast, Durovit, etc., can also be used for this, but for this note, it is assumed that Plexiglas is used,

The primary mold is sized according to how large the embedding (final) mold is to be, allowing for any size change during curing, so that the mold will fit into any oven used during embedding. This dimension is important!

To construct the primary mold, glue three Plexiglas plates as sides to a base plate. The fourth side will remain free. Add reinforcement to the base and three side plates joints by gluing strips along the outside of the joints.

As the slide-in plate must fit with precision in this three sided box, the three side plates and the baseplate must not have glue on or at the inner sides or edges. Such would interfere with the exact horizontal plane (of the slide-in plate) needed when curing the silicon rubber mass, as well as with the proper and tight fitting of the slide-in-plate with respect to base-plate and side plates when pouring the silicone mass. The surface of the Plexiglass parts should be thoroughly cleaned (fat free) before gluing by wiping with alcohol (*e.g.*, 100% EtOH). Rather than using "regular" glue, use chloroform mixed with Plexiglas "sawdust" or commercially available plexiglass glue to glue the sides. This will dissolve the Plexiglas and form a "weld", rather than a simple glue joint. "Regular" glue might result in excess glue coming out of the joint, and will give messy edges to the final product (including in the embedding cavities, see below). Gluing Is facilitated by using a disposable syringe to apply the glue.

The fourth side is left free, so that a slide-in plate with the forms for the embedding cavities can be placed in the primary mold. After placing the slide-in plate in the mold, this fourth side is held to the mold with clamps and/or with strong self adhesive insulation tape. It is a good idea to wrap the ends with plumber's Teflon tape.

The embedding cavities are made by gluing forms to the slide-in plate. These forms are sized and shaped according to the embedding cavities required. Make the forms about 4% larger than needed to allow for shrinkage during the curing of the silicone mass and/or the embedding resin during polymerization. The forms may be made from any of several different materials, such as metal, Plexiglas, BEEM capsules filled with polymerized resin, and so forth. Be sure to remove the BEEM capsules from the polymerized resin before gluing the resin blanks to the primary mold base-plate. Use as many forms as desired and make as many embedding cavities as needed (see figure 2).

Make identification numbers for the cavities by engraving numbers or letters in normal writing on the tops of the cavity forms. This results in reversed writing on the embedding mold, and in normal writing on the final resin casts. Identification marks can be inscribed in reversed writing on the primary mold or cavity forms so they can be read on the embedding mold.

The primary mold is then assembled: the slide-in plate with cavity forms is slid into the primary mold and the fourth side is then held by clamps or tape.

Casting the Specimen-Embedding Mold

The embedding mold is cast from silicone rubber. I have found that Elastosil RTV-2 M 4503 with T 35 hardener (Wacker Silicone Co.) at 5% (wt:wt) works well, but other types will work also. Local availability will determine what is used.

The silicone chosen should have these properties:

- ➜ Low to medium viscosity, so it is easily pourable, with a pot-life of at least 15-20 minutes;
- ➜ Moderately "hard", but flexible for easy removal of specimen blocks;
- ➜ Light or pale in color to aid in orienting specimens during embedding (Transparent is also good, then a color background can be used);
- ➜ Should withstand temperatures of 100 to 140°C for rapid curing of resin and "tempering" the molds. Tempering emulates aging of the silicone and increases its stability and life. The silicone and conditions will vary if the specimens are to be embedded at unusual temperatures.

With the Elastosil mentioned above, and the resin polymerized at 65°C, specimens can be embedded, polymerized, and removed by flexing the mold 50 or more times. Polymerizing at 90° to 100° C will shorten the life of the mold.

Follow the manufacturer's specifications for the silicone rubber and hardener/catalyst that you use. Mix in a container (glass or polyethylene) with two to three times the volume of the silicone solution you will be making.

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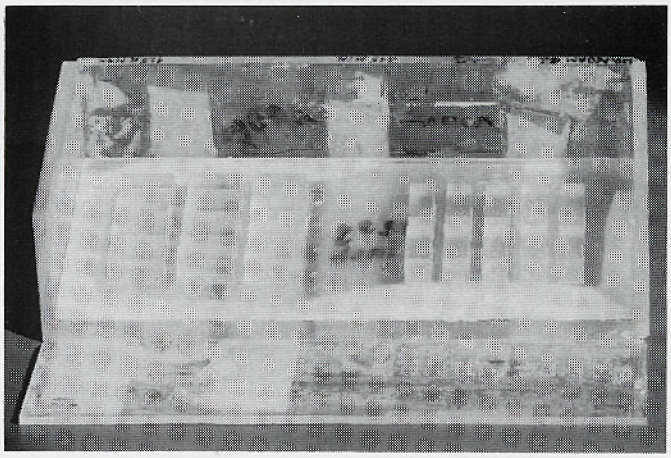

Figure 1A: Primary mold with slide-in plate for making rectangular embedding molds in place. The size of the slide-in plate is 14.8 x 7.3 x0.5 cm.

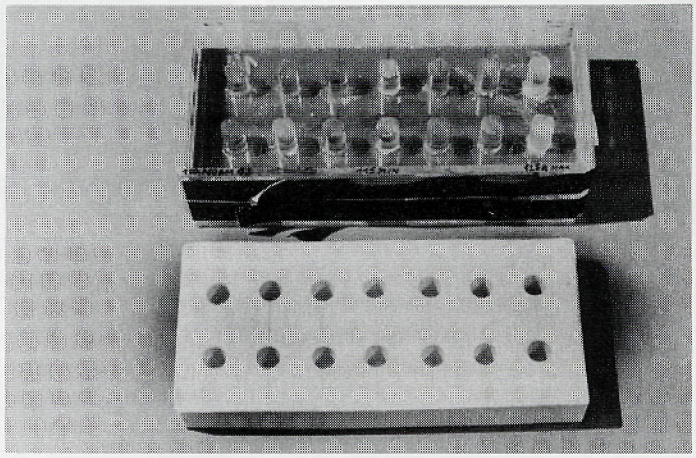

Figure 1B: Assembled primary mold and finished embedding mold for making BEEM-capsule shaped embedding cavities.

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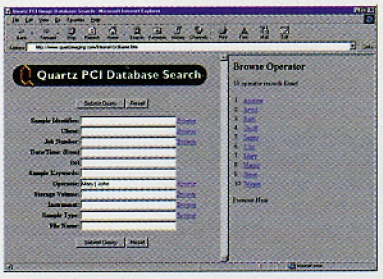

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Use gloves! The hardener often contains tetra-organo-Zn compounds that can be sensitizing or irritating.

Mix vigorously. The hardener presents an "oily" appearance, and likes to stick to the sides and bottom of the mixing vessel. Be sure that the mixing is complete, and no oiliness is seen.

When mixing is complete, degas the silicone to remove all air bubbles. This is easiest to do in a large vacuum desiccator, empty of desiccant. Line the bottom of the desiccator with paper towels, or spray with a PTFE releasing agent. Don't wipe off spilled silicone, let it cure, and then peel it off. Pumping can be done with a rotary pump, house vacuum, or a hand vacuum pump. The volume of the silicone will increase as it degasses. Admit air before it overflows the container. The bubbles, and so the silicone, will collapse. The trick is to provide a sufficiently large container (2-3 times the volume of mixed silicone rubber). The silicone mass will increase to a point of maximum extension, with lots of bubbles, not like a hydrous solution but more like a very viscous bubble gum mass. After reaching this maximum extension, the silicone mass will collapse back to its pre-evacuation volume. Now it is time to close the valve and admit air (more or less slowly). Don't evacuate any longer since volatile components would be exhausted from the mixture. This could interfere with optimal curing of the mold! If the container is too small to provide the "self collapsing" of the silicone mass, you have to close the valve to stop overflowing the container, then admit air, and then repeat pumping cycles as often as it would be needed to reach the "self collapsing" point of the mass! Proceed as above despite seeing "bubbles" on the surface - they will disappear on pouring.

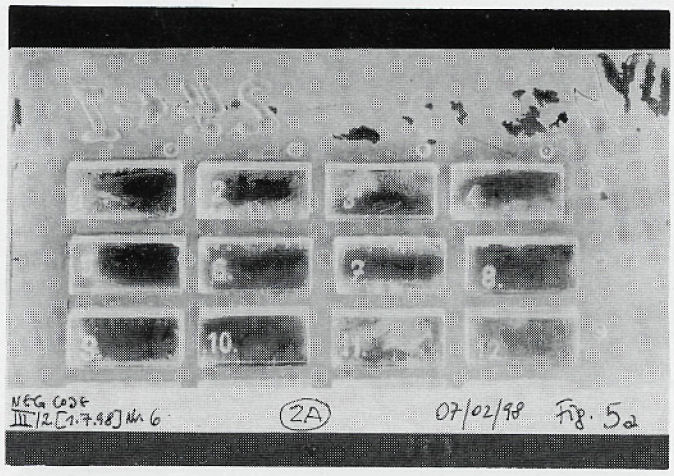

Figure 2A: Detail of slide-in plate for making rectangular embedding cavities, showing specimen block ID numbers engraved in base plate blocks.

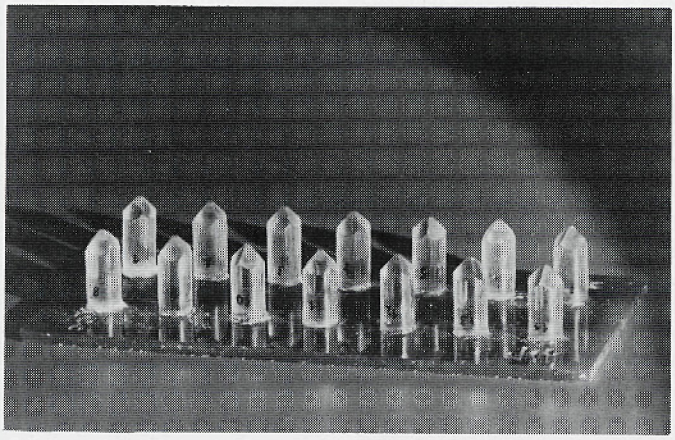

Figure 2B: Slide-in plate used for making BEEM-capsule shaped embedding cavities.

The silicone will be very viscous, but reasonably pourable. A small amount will remain in the container. If needed to fill the mold, this can be carefully scrapped into the mold. Be very careful during pouring not to introduce air bubbles into the silicone. Start pouring slowly, and speed up towards the end of the pour. Start pouring where there are no cavity forms. After the silicone has covered all of the forms, pouring can be speeded up and moved around the mold. Note: be certain that the silicone has completely spread around the bases and edges of the cavity forms before they are covered by more silicone! Otherwise air bubbles will be trapped at these points, with defective embedding cavities.

After pouring, as a product of initial curing, there will likely be air bubbles in the surface of the mold. These will not affect the embedding cavities and can be ignored. They, except for the larger bubbles, will eventually disappear during curing. If desired, these bubbles can be popped with a needle, or "exploded" with a fine jet of compressed air.

To cure the silicone, let the mold stand covered in a perfectly horizontal position in a dry, temperature-controlled environment for 15 to 20 hours. The curing process may be hastened by heating moderately in an oven at 50 to 100°C. This usually is not wise when using plexiglass! After this, soft "fluidy" areas are an indication of poor mixing of silicone and hardener.

Remove the polymerized embedding mold from the primary mold by removing the fourth side of the primary mold, then carefully remove the finished silicone embedding mold from the sides. Remove the slide-in plate and peel off the silicone mold from the molding forms of the slide-in plate. If the slide-in plate is stuck to the base plate, carefully separate the silicone from the sides, and peel the silicone form from the slide-in plate by carefully lifting the mold's edges.

Durability of the embedding mold can be improved if, after condensation, curing is tempered at 120° to 140° C for about 12 to 48 hours - after allowing the polymerized mold to sit for one to two days to finish outgassing curing products.

Note: These conditions, especially the time, may vary depending on the silicone rubber used. Also, polymerization may be accelerated in an oven, its temperature depending on the silicone used.

These silicone molds will last for 20 to 30 embedding and de-embedding cycles, and 50 cycles is easily achieved. The following will lengthen lifespans:

➜ Do not overfill the embedding cavities with resin. The walls dividing the cavities will be destroyed more quickly with overfilling;

➜ Spray the molds after or before use (at least every tenth or fifteenth use) with a thin layer of releasing agent - but don't spray every time, or there will be remnants of the releasing agent left on the specimen blocks. ■

Note: Compound names are for products available in Europe. The same, or closely similar, products are available under different names, from perhaps different companies, elsewhere in the world. In North America, try SylGuard from Dow Corning Chemical Co.

Photographs (Micrographs) by Dr. Arno Laminger, Salzburg, Austria

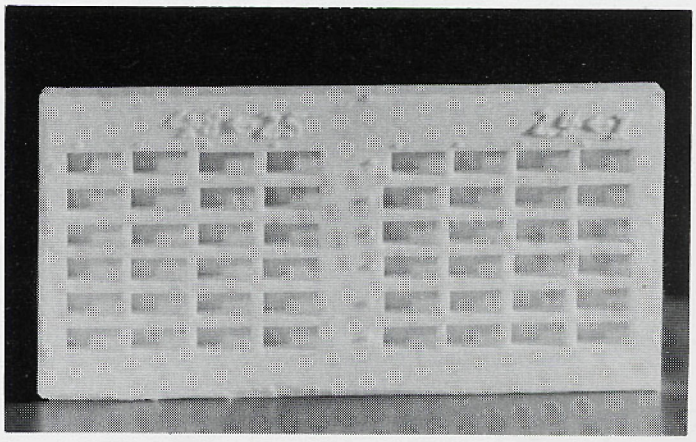

Figure 3: Finished specimen embedding molds showing two possible cavity numbering schemes