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An Old Material, a New Technique: Casting Paraloid B-72 for Filling Losses in Glass

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Abstract

A new technique has been successfully developed for casting Paraloid B-72 (B-72) in thin sheets or films, without air bubbles. These sheets are then shaped, cut, and used as fills for losses in glass. Solutions of B-72, clear or tinted with colours, are cast into open moulds, which are then kept partially sealed in a chamber. This eliminates the formation of air bubbles, and after several days the films can be removed for use.

Titre et Résumé

Un produit de longue date, une nouvelle technique : le coulage du Paraloid B-72 pour le comblement de lacunes dans les objets en verre

Une nouvelle technique a été mise au point pour réaliser le coulage du Paraloid B-72 (le B-72) sous forme de feuillets ou de pellicules minces qui ne contiennent aucune bulle d'air. Les feuillets sont ensuite mis en forme et découpés et utilisés comme matériau de comblement de lacunes dans les objets en verre. Les solutions de B-72, limpides ou colorées, sont coulées dans des moules ouverts, lesquels sont partiellement étanchéisés et conservés dans une enceinte particulière. Cette mesure permet de prévenir toute formation de bulles d'air et, après quelques jours, les pellicules d'adhésif peuvent être retirées des moules pour utilisation ultérieure.

Introduction

Materials used for filling losses in glass objects should be selected with care. This is particularly true for fragile archaeological glasses where the glass is very thin (0.5-1.0 mm) and the glass structure is often weakened. Some of the commonly used synthetic resins or casting techniques can result in more damage to a glass (Down 1996; Tennent and Koob 2010).

Direct casting resins

Epoxy or polyester resins have been traditionally used for filling losses in glass. Their use and characteristics are well referenced in the literature (Jackson 1983; Bradley and Wilthew 1984;

Bradley 1990; Down 1996; Nudes de Silva 1998; Shashoua and Ling 1998; Down 2001). They offer a refractive index close to glass, high Tg, perfect transparency, and a large variety of reconstruction technique possibilities (simple backings, open or two-sided moulds, direct or indirect castings). Despite having many excellent properties, epoxy resins have limited reversibility and tend to yellow over time. Even the most stable resins (Hxtal NYL-1 and Epotek 301-2), under good exposure conditions will show degradation (Down 1984; Down 1986). Polyesters also have a history of yellowing. Another disadvantage to direct filling with epoxy or polyester is that the entire object usually has to be dismantled to replace any fills. The work has to be started from the beginning, cleaning all the breaks, assembling, and gap filling. This can be very time consuming and poses more risk of damage and strain to the glass. The potential damage to glass from commonly used fill methods and the limited stability and reversibility of epoxy and polyester make other materials and techniques desirable.

Reversible, detachable fills

Numerous less-invasive techniques have been developed, designed to be reversible. One of these is the use of Japanese paper (Henau 1980-81; Fontaine 1988-89; Perbu, Heulin, Leclerc et Leconte 1990; Fontaine 1993; Fontaine 1999). The fills are very light and the technical process reduces the number of manipulations of the fragile glass object. However, this technique is not always visually acceptable, if the glass is clear and transparent, and it only provides minimal structural support.

Detachable fills have also been developed to be both reversible and minimize the strain and potential damage caused by direct casting and finishing on the glass. Several methods have been recommended using plaster, wax or modeling clay fills which are moulded, cast in epoxy, and then adhered in place (Davison 1998; Koob 2000; Koob 2006).

Another technique that has been recommended is to produce an epoxy film or sheet of resin away from the object (Lechat 2007; Lacroix 2009; Fontaine and Benrubi 2010). The profile of the fill is made by positioning the flexible film (before it is completely set or by reheating slightly with a hot airgun) on a form reproducing the shape and curvature of the missing area; then the film is cut to fit exactly the area to be filled. Plexiglas or Perspex sheet can be used in a similar manner (Hogan 1993), but is difficult to work with and often requires coloured coatings to match the glass.

In all these cases, the fills are fixed in position using an adhesive made of Paraloid B-72 (B-72) (Koob 1986). This results in less strain on the glass, and offers long-term reversibility. Although the techniques described above do address some of the problems with using epoxy resins for filling losses in glass, they do not prevent the fills from yellowing. This means that eventually the objects will have to be retreated which always creates a potential for new damage, especially with fragile glasses.

Filling with Cast B-72

A new technique has been developed at The Corning Museum of Glass for loss compensation in glass. B-72, one of conservators' best-known acrylic resins, is now being cast in film and sheet form to use for filling losses in glass objects.

B-72 is an acrylic resin which needs to be dissolved in one or more solvents, and is not practical for direct casting on objects to fill losses because so much shrinkage occurs during the setting process. However with careful preparation, the technique is simple, minimally invasive, and can produce fills that can be transparent, translucent or opaque. B-72 fills can be made thick enough to provide structural support for glass fragments, or it can be an aesthetic non-invasive fill similar to a tinted Japanese paper fill, with the added advantage of being transparent. In addition, the B-72 fills will never yellow like epoxy or polyester resins.

Casting procedure

The B-72 is first dissolved in acetone, and the mixture is then modified with the addition of ethanol or a small amount of xylene (see the case studies for the amounts used) to slow evaporation. Ethanol is relatively non-toxic, and is preferred. The mixture is stirred well, and pigments or dyes are added as necessary, along with an opacifier such as fumed silica or titanium dioxide, to achieve the desired color and transparency to match the glass being restored. The mixture is then poured onto polyethylene sheeting or silicone release paper, or in an open-faced silicone rubber mould (Figure 1), to the



Figure 1: removing flexible film from mould



Figure 2: open-faced silicone moulds in polyethylene bags

desired thickness (keeping in mind that solvent loss will cause significant thinning or shrinkage of the film). Once the B-72 is poured out, it must be immediately covered or placed in a vapour chamber to slow down the solvent evaporation. The poured out B-72 can be placed under a watch glass, Petri dish, or inside a plastic bag and left covered for 1-4 days, depending on the size and thickness of the film and the exact solvent mixture. This restricts, but does not stop, the solvent from evaporating out of the film. This very slow evaporation of the solvent allows the film/sheet to form uniformly, and without air bubbles. After several days, the B-72 film can be removed, and because it is still flexible (Figure 2), it can be easily cut with scissors or a scalpel, and be shaped to specific curvatures. Large fills and ones with deep curvatures are more difficult to achieve; the film needs to be slumped over a base of the right shape until it is rigid enough not to collapse in on itself. Hardening of the B-72 can be aided by placing the film or sheet in an incubator oven at 40-50° C. Thin applications of B-72 will harden faster, while thicker applications (2-3 mm) take longer. The amount of opacifiers and pigments or dyes added also affects hardening time.

The B-72 fills are then easily attached to the glass with a very small amount of B-72 adhesive or very careful wetting of the edges with acetone.

Manipulating the appearance of the film

The B-72 films can be coloured with dyes or pigments. Dyes should be dissolved in a solvent (ethanol) before being added to the B-72. The resulting film will have clarity and a transparency almost equal to results obtained with an epoxy.

Similar results can be achieved with pigments which are more stable and thus preferable (Figure 3a). A very small amount of pigment is mixed first with ethanol then the B-72 is added and allowed to rest for 10 min. This way the heavier pigments can drop to the bottom leaving a uniform clear color at the top. The B-72 is then slowly poured into the mould making sure to stop just before the accumulation of pigments at the bottom. If the mixture is not allowed to rest the larger, heavier pigments will be visible in the film (Figure 3b). Pigments can also be reground and sieved to help eliminate the larger pigments.



Figure 3: a: B-72 film with pigments not visible;

b: B-72 fill with pigments visible

The transparency of the film can be adjusted with the addition of pigments or fumed silica to the resin mixture. It is best to dissolve the fumed silica is a small amount of solvent before adding to the resin mixture. Adding it directly to the resin mixture can cause the fumed silica to

clump resulting in a "lumpy" film. The addition of fumed silica will increase the viscosity of the mixture and will result in a thicker film. It can also shorten the time the film needs to be covered and make the film more rigid (Degussa 1989; Degussa 1995).

Most of the B-72 solution consists of solvents that have to evaporate off in order for the film to harden. This means that the resulting film will be significantly thinner than the initial pouring. Thicker films can be achieved by adding additional B-72 to a mould with a film that has hardened or partially hardened.

The texture of the film can be changed by disrupting the surface while the film is still slightly sticky. This can be done with a finger (Figure 4) or textured tool or fabric that will not stick to the resin. If the film is no longer sticky, the surface can be made sticky with a tiny amount of solvent.

Case Study 1

One of the largest successful fills made from cast B-72 was made for a Roman ewer (Corning Museum of Glass 2007.1.26). Before treatment it was in nearly 100 pieces and had many areas of loss, including most of the rounded bottom. The fragments around the missing bottom were very thin (0.2 mm in some areas), small, and numerous, making the task of filling the loss difficult. Common fill techniques such as casting epoxy in place or from a detachable plaster fill were not possible because of the fragility of the vessel. Instead a fill was made of a cast sheet of B-72.



Figure 4: Small textured fill from case study 1



Figure 5: before (left) and after (right) treatment, Corning Museum of Glass 2007.1.26

The fill was made in the following manner:

A 30% solution of B-72 in acetone was made up. Funed silica and very small amounts of dry pigment were added to match the solution to the color and transparency of the glass. A small amount of xylene (a few drops for a 100 ml solution) was added and then the solution was set

aside for at least an hour to rehomogenize. A rectangle of silicone release paper was folded to make a shallow open box. The B-72 solution was poured into the box which was then sealed in a zip-lock polyethylene bag. The bag with the box of B-72 was put in an out of the way place and left undisturbed. After about 4 days the B-72 film was removed from the bag and pulled free of the paper. At this point the film, which was free of bubbles and still quite flexible (but not sticky), was slumped over a glass flask with a curvature



Figure 6: Shaping B-72 film on flask in oven

similar to that of the bottom of the vessel. The B-72 film was left on the flask for most of a day until it was rigid enough to not collapse in on itself. It was then roughly cut to the desired size (approximately 8 cm x 3 cm) and shape and placed on the bottom of the vessel over a weekend. After the weekend the fill had fallen in on itself somewhat. This was corrected by placing the fill back on the flask and heating it to $40-50^{\circ}$ C for a day to drive off more of the solvent (Figure



Figure 7: Detail of large finished fill

6). The fill was then cut to better fit the area of loss and continued to be heated on and off for two more days. Finally the fill was joined to the vessel with small amounts of acetone brushed onto the edges of the fill.

A few additional small fills were made to support the fragments with very small break edges. Two of these fills were lightly textured by disrupting the surface of the film with fingers slightly wetted with acetone.

Case Study 2

The object of the second case study is an Islamic glass of a translucent turquoise green colour with dark brown surface decorations (Corning Museum of Glass 68.1.3). The vessel consisted of more than 200 fragments, most of them between 0.5 and 2 cm in size. The walls of the glass averaged 1 mm in thickness while the base was slightly thicker. Detachable epoxy fills were made for the larger fills at the bottom where the glass was thicker and less fragile. However, the upper fragments were so small and so thin that gap filling and joining had to be done simultaneously. Detachable epoxy fills were not practical in this case. Cast sheets of B-72 were used to make the fills.

The resin mixture used was 20 ml of a 30% solution of B-72 in acetone to which 4 ml of ethanol was added. This object required numerous fills which needed to have the same transparency, thickness, and color. For the initial castings, the B-72 was spread out on a polyethylene sheet or in a silicone release paper shaped in a box. However, it was difficult to get castings with the same thickness and therefore color with this technique. Instead open silicone moulds were made from square and rectangular pieces of Plexiglas. The filled moulds were then sealed in a polyethylene bag. By using the same resin mixture, moulds, and drying conditions, uniform films were made.

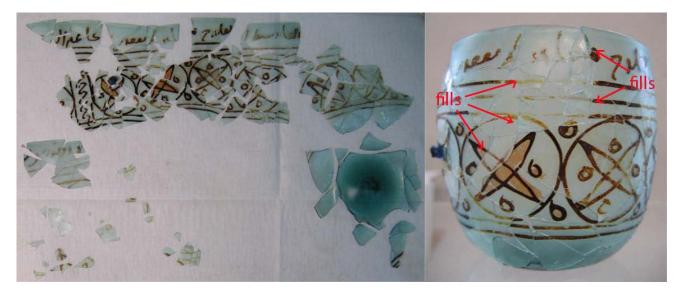


Figure 8: before (left) and after (right) treatment, Corning Museum of Glass 68.1.3

After 48 hours, the B-72 films were hard enough to be removed from their moulds. They were still flexible enough to be placed on the object over the gap. The film was cut to the appropriate shape with scissors or a scalpel and the cut out shape was placed in the missing area and joined to the breaks of the glass by simply applying a drop of acetone with a soft brush in between the break and the film.

Most of the fills were structural and helped with the assembly of the glass fragments. It helped to make the process far less laborious and saved a considerable amount of time. The small glass fragments with little join surface were held with scotch tape just for the time needed to cut out a

fragment of B-72 film and put it in place. This only required a few seconds and the B-72 fill acted as a bridge to hold the two glass fragments together.

For the larger fills, two films were joined by applying a bit of acetone between them. The join line between the two fills is very discreet as the object in itself has many break lines.

Some final retouching was done on the fills, primarily as an experimental trial. Acrylic paints were mixed with Primal WS-24 diluted to a 10% concentration, and some decoration lines were completed successfully on the B-72 fills.

Limitations, Further Experimentation, and Future Applications

Thick films

Fills made from cast films of B-72 are very useful for some applications, but like any other fill technique, won't work for every situation. So far no fills for glass more than 1-2 mm thick have been made. Although this is probably possible by adding fumed silica and "topping up" partially hardened castings, further experimentation would be needed to find the best method to do this. Thick transparent fills would be especially difficult to produce because adding fumed silica would make the film less transparent.

Flexibility and rigidity

Another difficulty with this fill technique is the timing of when to remove the fill from the vapour chamber. If it is removed too soon the film will be too flexible and will stretch or collapse in on itself. In some cases the fills may distort after placement because not enough solvent was driven off. A good method to prevent this is to heat the fill (to 40-50° C) on a mould with the right curvature. If it is removed too late it may be too rigid to conform to the curvatures needed for the fill. For small fills the rigidity is usually not a big problem, but for larger fills or fills with extreme curvatures a rigid film will be unusable. Since the addition of fumed silica and pigments alters the hardening time, each resin mixture will be different and some experimentation will usually be necessary to find the ideal amount of time the resin should remain covered.

Fills can be re-shaped using heat (e.g., from a hair dryer) to take advantage of B-72's thermoplasticity. In addition, silicone moulds can be reused again and again, and since B-72 is inexpensive, new castings can be easily made.

Archaeological/field applications

One possible future use of B-72 films is in field conservation. Films of different colors and thicknesses could be made in advance and brought to locations where supplies and time are limited. The pre-made films could be re-activated or softened with heat or in solvent chambers and then cut and shaped to fill losses.

B-72 is not appropriate for fills (or even as an adhesive) if the glass is to be stored in very hot conditions, exceeding B-72's 40 °C Glass Transition Temperature (Tg). The addition of fumed silica may increase the Tg slightly, and should be studied further. Other resins such as Paraloid B-67 or B-48N, both of which have higher Tg's, may have possible applications.

Fumed pigments

Industrial developments in the production of fumed metallic oxides have already produced materials useful for modifying colours and rheological (flow) characteristics for adhesives and coatings. Fumed titanium dioxide has a much smaller particle size than standard titanium dioxide pigment, and imparts a translucency to a B-72 cast film, that is otherwise not obtainable. This has been used for certain applications, where a white, hazy, translucent film is desired. The production of other fumed metallic oxides may provide additional colours.

Conclusion

B-72 fills are inexpensive, stable, yet easily reversible and can be made to fill losses on ancient, historic or modern glasses. They are especially useful for very fragile glasses that would not tolerate the more invasive gap filling methods traditionally used for glass. This technique is particularly suitable for archaeological glass. The latter often undergoes significant alterations that make it vulnerable to manipulation and restoration products. Gap filling with B-72 allows minimum intervention on the glass while limiting excessive handling. B-72 has all the characteristics required for a filling material such as reversibility, stability, health safety, ease of application, as well as the possibility of colouring and retouching. It also has the advantage of being accessible to all conservators.

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Materials and Suppliers

Fumed silica; Fumed titanium dioxide Degussa Corporation 65 Challenger Road Ridgefield Park, NJ 07550

Resins:Paraloid B-48NMethyl methacrylate and butyl acrylate copolymerParaloid B-67Isobutyl methacrylate polymerParaloid B-72Ethyl methacrylate (70%) and methyl acrylate (30%) copolymerPrimal WS-24Acrylic colloidal dispersionConservation Resourceswww.conservationresources.com

Solvents: Acetone CH_3COCH_3 Ethanol C_2H_5OH Xylene $C_6H_4(CH_3)_2$ Fisher Scientific www.fishersci.com

Acrylic paints; Dyes; Pigments; Plexiglas; Silicone moulding materials Hardware stores or art/conservation suppliers

Author Biographies and Contact Information

Stephen P. Koob received an MA in Classical Archaeology from Indiana University (Bloomington, Indiana) in 1976, and a BSc in Archaeological Conservation and Materials Science from the Institute of Archaeology, University of London (London, United Kingdom) in 1980. He spent the next 5.5 years working as conservator of the Agora Excavations with the American School of Classical Studies in Athens, Greece. In 1986, he joined the Smithsonian Institution in Washington, DC, working as a conservator (specializing in ceramics and glass) at the Freer Gallery of Art and Arthur M. Sackler Gallery. He left the Smithsonian in 1998 to come to The Corning Museum of Glass, where he is now Chief Conservator.

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Sarah Benrubi has an MA in Conservation (2003) from the École National Supérieure des Arts Visuels (ENSAV) La Cambre in Brussels, Belgium with a specialization in ceramics and glass. She has also completed internships at The Corning Museum of Glass in Corning, New York and The National Museum in Cardiff, United Kingdom, as well as a postgraduate internship at the Royal Institute for Cultural Heritage (IRPA) in Brussels. Since 2005, she has been working as a private conservator and a collaborator on projects at the IRPA in the Glass Conservation department. In 2009, she became an Assistant Instructor for the Ceramics and Glass Conservation department at the ENSAV La Cambre in Brussels.

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Stephen P. Koob a obtenu une maîtrise en archéologie classique à l'Université de l'Indiana (Bloomington, Indiana) en 1976 et un baccalauréat ès sciences en conservation des objets archéologiques et science des matériaux à l'Institute of Archaeology de l'Université de Londres (Londres, Royaume-Uni) en 1980. Au cours des cing années et demie qui ont suivi, il a travaillé comme restaurateur avec l'École américaine d'études classiques à Athènes (Grèce), sur le site des fouilles de l'Agora. En 1986, il entre au service de la the Smithsonian Institution à Washington, DC, et il occupe le poste de restaurateur (spécialisé dans les céramiques et le verre) à la Freer Gallery of Art et à la Arthur M. Sackler Gallery. Il a quitté la Smithsonian en 1998 pour passer au Corning Museum of Glass, où il est maintenant restaurateur en chef.

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Sarah Benrubi est titulaire d'une maîtrise en restauration (2003) de l'École nationale supérieure des arts visuels (ENSAV) La Cambre de Bruxelles, en Belgique, avec une spécialisation en céramique et en verre. Elle a aussi fait des stages au Corning Museum of Glass, à Corning, dans l'État de New York, et au National Museum de Cardiff, au Royaume-Uni, ainsi qu'un stage postdoctoral à l'Institut royal du Patrimoine artistique (IRPA), à Bruxelles. Depuis 2005, elle travaille comme restauratrice privée et collabore à des projets de l'IRPA au service de restauration des verreries. En 2009, elle est devenue formatrice adjointe au département de restauration des objets de céramique et de verre de l'ENSAV La Cambre, à Bruxelles.

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