

# AN EVALUATION OF FOUR BARRIER COATING AND EPOXY COMBINATIONS IN THE STRUCTURAL REPAIR OF WOODEN OBJECTS

*Lisa Ellis and Arlen Heginbotham*

## ABSTRACT

This investigation was undertaken to determine the suitability of two synthetic resins for use as barrier layers in the bonding of wood with epoxy. The two materials in question, Paraloid® B-72 and Acryloid® B-67, were chosen because of their potential to be practically reversible in low polarity solvents. The two polymers were compared, as barrier materials, to two proven barrier coatings, hide glue and Butvar B-98, by measuring their strength in shear according to ASTM standard D 905-98. Investigations were also undertaken to determine the amount of time necessary for barrier layers to dry prior to application of epoxy. Finally the practical reversibility of the barrier coatings was empirically evaluated. Paraloid® B-72 was found to be a suitable barrier material in all respects, while B-67 failed both strength and reversibility tests.



## INTRODUCTION

In the conservation of wooden artifacts, it is often necessary to repair broken wooden elements which serve a structural or load-bearing function. Such repairs must have high strength, yet be reversible in the future. Where the break in question is recent and the mating surfaces are clean and undisrupted, animal hide glue is widely accepted to be a suitable adhesive, though in practice, reversal of intact hide glue bonds can be problematic. In cases where the mating surfaces are dirty, damaged, or a gap filling adhesive is needed, animal hide glue may have greatly reduced strength and an alternative adhesive may be required. Bulked epoxy resins have found wide use in such instances, and some have the additional advantage that after setting they can be carved, sawn, sanded, and finished, allowing them to be used simultaneously as both adhesive and fill material. One commercially available product of this sort which is widely used by furniture conservators is Araldite 1253, a carvable paste epoxy, bulked with titanium dioxide, amorphous silica, iron oxide, and phenolic resin (Ciba, 2001). The primary disadvantage of using epoxies in conservation is that, once cured, they can be extremely difficult to reverse.

Barrier coatings are widely used in conservation to add a measure of reversibility to an otherwise irreversible adhesive bond. The barrier material is applied as a thin film to both mating surfaces prior to application of the primary (irreversible) adhesive. Subsequently, if reversal is required, the barrier layer can be softened or dissolved, releasing the bond. In the conservation of wooden artifacts, animal hide glue has been used as a barrier material for epoxy joins due to its high strength, ease of use, and its familiarity among furniture conservators. Hide glue, however, has certain disadvantages as a barrier material. First and foremost, it is not always reversible in a safe and practical manner. Reversal depends on moisture and/or heat, both of which can cause damage to wood and associated finish materials. Some promising work has been reported using microwave radiation to reverse hide glue bonds (Neher, 1997); however, the equipment necessary is quite expensive and the technique has not gained wide acceptance. In addition, hide glue is known to weaken when exposed to extremes of humidity (Buck, 1990) and may degrade over long periods of time.

Recognizing the difficulty in reversing repairs made using hide glue as a barrier, and seeking an appropriate alternative, Anderson and Podmaniczky tested the suitability of Butvar B-98 [poly(vinyl butyral)] as a barrier layer for epoxy joins in wood (Anderson and Podmaniczky 1990). B-98 is often referred to simply as a poly(vinyl butyral), but it is actually a co-polymer of poly(vinyl butyral), poly(vinyl alcohol), and poly(vinyl acetate) in a ratio of approximately 40:10:1 (Horie, 1987, 101–102; Monsanto, 1994). This polymer was recently shown experimentally to be a suitably stable material for the consolidation of dry archaeological wood (Spirydowicz, *et al.*, 2001). Reporting on the results of their testing in the 1990 article, “Preserving the Artifact: Minimally Invasive Conservation Treatment at the Winterthur Museum,” Anderson and Podmaniczky noted that while barrier coatings should help make epoxy repairs more easily reversible, they must also maintain the overall strength of the bond. The results of their work demonstrated that Butvar B-98 dissolved in ethanol is a suitably strong barrier material when used in conjunction with the bulked epoxy, Araldite 1253.

Butvar B-98, while a good alternative to hide glue as a barrier material, also has significant limitations with regard to its reversibility. B-98 is soluble in polar solvents such as alcohols and in certain mixtures of polar and non-polar solvents (Monsanto, 1994; Spirydowicz, *et al.*, 2001). Unfortunately, many varnishes and paints traditionally used to coat wooden artifacts are also sensitive to this range of solvents, making it difficult or impossible to dissolve a B-98 barrier layer without damaging an adjacent surface coating. This is particularly true because extended exposure (to liquid or vapor) may be necessary to allow the solvent to penetrate deep into the repair and dissolve the barrier. Even after Anderson and Podmaniczky’s important study, therefore, there remained a need for a well-tested barrier coating of high strength which could be reversed in low-polarity solvents.

In this study, the authors chose B-72 and B-67 for comparison with the other proven barrier adhe-

sives because they have advantageous dissolution properties, they are readily available, and they are well known and widely used by conservators. Paraloid® B-72, a copolymer of ethyl methacrylate and methylacrylate, is a Feller Class A material and is not known to become insoluble or degrade over time (Horie, 1987, p.106). It is soluble in low polarity solvents such as xylenes which will not dissolve most historic furniture finishes. Its inclusion in this study seemed obvious: it is a mainstay in the conservator’s studio, and its strength, when used in combination with epoxies in the bonding of stone, has recently been clearly established and published (Podany, *et al.*, 2001). Acryloid® B-67, poly(isobutyl methacrylate), is also considered a Feller Class A material even though it is known to cross-link over time (Horie, 1987, 108). B-67 was considered in this study because it is reversible in low-aromatic hydrocarbons which present less of a health hazard than the fully aromatic solvents necessary to reverse Paraloid® B-72.

The authors determined to conduct comparative shear strength testing with all four of the mentioned barrier coatings (hide glue, B-98, B-72, and B-67) with Araldite 1253 bulked epoxy. It was hoped that if B-72 and/or B-67 proved to be of comparable strength to the other two proven barrier materials, then the results of this work would provide conservators with more options in choosing a barrier coating when factors such as the solubility of an original finish need to be considered. In order to confirm that the adhesive bonds using barrier layers were in fact reversible as intended, the authors also conducted empirical reversibility testing.

#### METHODOLOGY

This study was organized into four components. First, a barrier application protocol was established and the amount of time required for barrier layers to dry was determined experimentally. Second, the shear strength of the adhesive bond made with each of the barrier materials and Araldite 1253 epoxy was determined quantitatively according to ASTM standard D 905-98. Third, the patterns of failure in the test samples were analyzed.

Both strength testing and failure analysis were conducted according to ASTM standard D 905-98. Fourth, the practical reversibility of the barrier materials was tested empirically.

### Barrier application protocol

The following solutions of barrier materials were chosen for testing:

- 10% (w/v) solution of Butvar 98 in ethanol
- 17% (w/v) solution of Paraloid® B-72 weight/volume in acetone
- 17% (w/v) solution of Acryloid® B-67 in Shell Mineral Spirits 135
- Titebond Liquid Hide Glue direct from manufacturer's container

The solutions were formulated as such for two reasons; they needed to be concentrated enough to leave a significant amount of material on the surface of the wood, but also had to be able to be applied in a continuous, even coat with a brush. Anderson and Podmaniczky used a 20% (w/v) solution of Butvar B-98 in ethanol in their study. In our experience, however, this proved too viscous to brush on conveniently and a concentration of 10% in ethanol was used instead. The 17% (w/v) solution of Paraloid® B-72 in acetone was chosen because it had been used successfully in the 2001 study by Podany *et al.* and was found to be easy to apply. The 17% solution of B-67 in Shell Mineral Spirits 135 was chosen to be comparable to the B-72 solution. The Titebond hide glue was chosen because it is a widely available, reasonably standardized formulation and was found to be of comparable strength to typical hot animal hide glues in moderate humidity environments (Buck 1990). Its viscosity was found to be suitable directly from the manufacturer's bottle.

The barrier coatings were applied, as consistently as possible, to samples of hard maple similar to those called for in the ASTM shear strength testing method. For each kind of coating, a new brush was dipped into the jar containing the solution; the bristles were then brushed against the rim of the jar; the brush was flipped over and excess solution brushed away again. The sample was then

brushed once along its length and again across the grain. The coatings applied this way appeared to completely saturate the surface of the wood, leaving no bare or dry areas.

After the first application had dried, the barrier coatings were evaluated visually. While the hide glue layer appeared coherent and glossy over the entire surface, the three synthetic resin layers did not; therefore, a second coat of each synthetic resin was applied over the first. Upon drying, all three of these samples appeared to have a reasonably thin, yet coherent and glossy film over the test surface. We therefore decided that in preparing the sample blocks for shear strength testing, the hide glue barrier layer would be applied in a single layer, while the three synthetic resins would be applied in two layers.

### Solvent Evaporation from the Barrier Coatings

We next tried to determine the length of time required for the barrier layers to dry, prior to the application of the epoxy adhesive. It is generally accepted that there should be little to no solvent remaining in the coating as retained solvent can act as a plasticizer within the resin and thus weaken the barrier film (Podany, *et al.*, 2001, 27).

### Testing Method

A simple test was designed to establish when the solvent had evaporated from barrier layers. Small wafers of hard maple (the wood called for in the ASTM shear strength testing method) were painted with the barrier coatings and then weighed periodically until there was no more detectable weight change. The maple was cut into 20 samples measuring approximately 2" x 3" x 1/8". Five pieces of wood were set aside to be used as controls to track the changes in weight of the substrate caused by fluctuations in the ambient relative humidity. The 15 remaining samples were divided into three groups. Each group was coated with two coats of the synthetic resin barrier solutions, the second coat following the first by three days. Hide glue was not tested for solvent evaporation time because of anticipated complications due to its continual weight change with fluctuations in ambient

relative humidity. Changes in weight for all forty samples were recorded in the same order, using an Ohaus® Precision Standard scale, which is accurate to one milligram. Since the weight of solvent added in each coat was typically about 0.3 grams, the scale was effectively accurate to approximately 0.3% for measuring solvent loss. The five wafers of each group were weighed individually and their weights were then averaged. Initially, weight measurements were taken every half hour. The interval between measurements increased with time, until five days after the second application of barriers, measurements were taken twice a day. We felt that once the weight stopped changing measurably, it could be concluded that the barrier adhesives were essentially free of solvent.

In order to better understand the dynamics of solvent evaporation from wood substrates, the same test was carried out using two different substrates, Douglas fir and 4 mil Mylar® polyester film (essentially non-absorbent). These results were compared to those recorded for hard maple.

### Results of Solvent Evaporation Tests

The solvent evaporation testing on maple showed that all three non-aqueous barrier coating materials would be essentially solvent free within five days of the application of the second coat. As expected, the faster evaporating solvents (acetone and ethanol) yielded dry films more quickly than the slower evaporating Shell Mineral Spirits 135. Table 1 shows the times required for 98% and 100% evaporation of solvent from the second coat of barrier solution. Based on this result, sample blocks of maple which had been coated with the barrier layers and allowed to dry for three

days, then re-coated and allowed to dry for five days were considered suitable for strength testing according to ASTM standards.

Two interesting phenomena were observed during evaporation testing. First, it became clear that the nature of the substrate played a large role in the evaporation rate of solvent from the resin layer. Figures 1 and 2 illustrate the extremely different evaporation rates for three different substrates when initially coated with resin solutions. Figure 1 shows the progress of drying for 17% (w/v) B-72 in acetone when applied to Mylar®, maple, and Douglas fir test panels. On the Mylar® substrate, which is essentially non-absorbent, over 99% of the solvent applied had evaporated within one minute. In contrast, on the test wafers of maple, a dense and even-grained wood, it took approximately 21 hours for 98% of the solvent to evaporate. With the fir substrate, which is lighter than maple and has distinct hard and soft zones in each annual ring, it took approximately 29 hours until 98% of solvent had evaporated. Figure 2 shows the progress of drying for 17% (w/v) B-67 in Shell Mineral Spirits 135 (a much slower-evaporating solvent than acetone) on the same three substrates. On the Mylar® substrate, 98% of the solvent applied had evaporated after only one hour and 40 minutes. On the test wafers of maple, it took approximately 60 hours until 98% of solvent had evaporated, and with the fir substrate, it took over 91 hours until 98% of solvent had evaporated. It is interesting to note that in the drying curves for B-67 on maple and fir substrates, the fir samples initially dried more quickly than the maple samples, but were overtaken by the maple after about a day. The cause of this phenomenon is unknown,

	98% Evaporated	Terminal Weight "100% Evaporated"
B-72 [17% (w/v) in acetone]	2.5 hours	28 hours
B-98 [10% (w/v) in ethanol]	26 hours	51 hours
B-67 [17% (w/v) in Shell 135]	75 hours	124 hours

TABLE 1 Time required for solvent evaporation from second coat of barrier material on hard maple substrate.

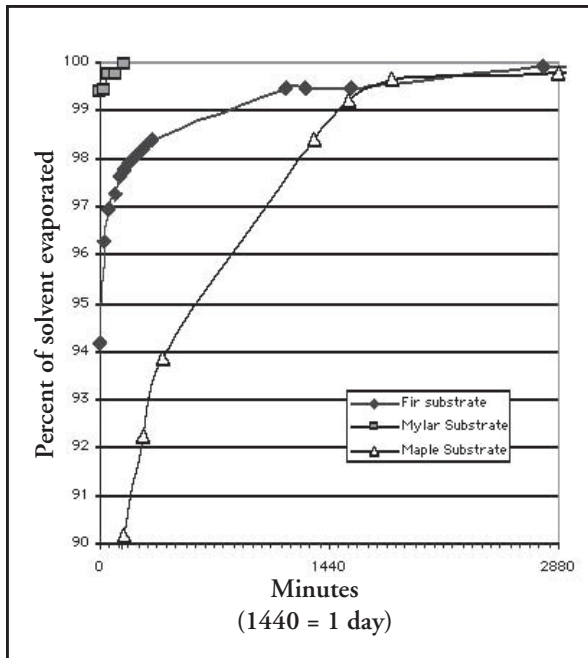


FIG. 1 Evaporation of solvent from 17% (w/v) B-72 in acetone applied to three different substrates.

but it suggests that the mechanisms of solvent evaporation from wooden substrates are complex, and may depend on a wide range of variables such as the anatomical characteristics of the wood, the condition of the wood surface, the affinity of the particular solvent for both the resin and the wood, the volatility of solvent, and the film thickness.

The second phenomenon noted during evaporation testing was that, on wooden substrates, the first and second coats of resin solutions dried at different rates. With the fast-evaporating solvents, acetone and ethanol, the second coat of barrier material clearly dried more quickly than the first. Presumably this is because the first layer of resin seals the wood so that when the second coat is applied, the solvent is not absorbed into the wood to the same degree. Figure 3 shows the percent of solvent evaporated vs. time for the first and second coats of 17% (w/v) B-72 in acetone applied to maple substrate. While the first coat does not reach 98% evaporation until 21 hours after application, the second coat is 98% dry after only 2.5 hours. In contrast to the results with fast-evaporating solvents, the second coat of barrier in slow-evaporating mineral spirits dried more slowly than the

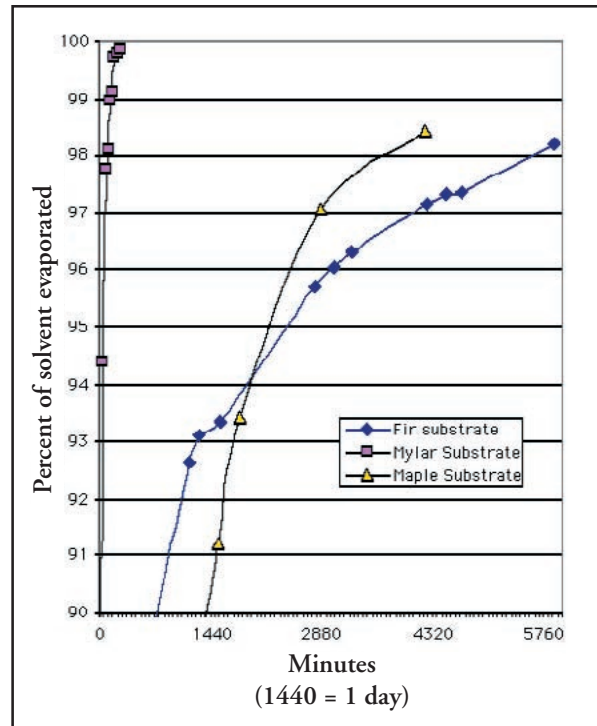


FIG. 2 Evaporation of solvent from 17% (w/v) B-67 in Shell Mineral Spirits 135 applied to three different substrates.

first. Figure 4 shows the percent of solvent evaporated vs. time for the first and second coats of 17% (w/v) B-67 in Shell Mineral Spirits 135 applied to maple substrate. While the drying rates are much more similar than with B-72 in acetone, it is clear from the graph that the first coat evaporated more quickly than the second. This presumably indicates that some of the solvent in the second coat penetrated the first coat and was absorbed into the wood. The increased overall thickness of resin after the second coat may then have contributed to an overall slower drying of the coating layer.

The degree to which barrier layers on wood should be allowed to dry before final assembly with epoxy is difficult to judge with certainty. Both for convenience and because contamination of the surface by airborne pollutants could result in a weakened bond, it is better to glue up joints soon after the surfaces are prepared. However, premature bonding when using barrier coatings could result in a joint which is initially weak due to plasticizing effects of retained solvent. It might also result in

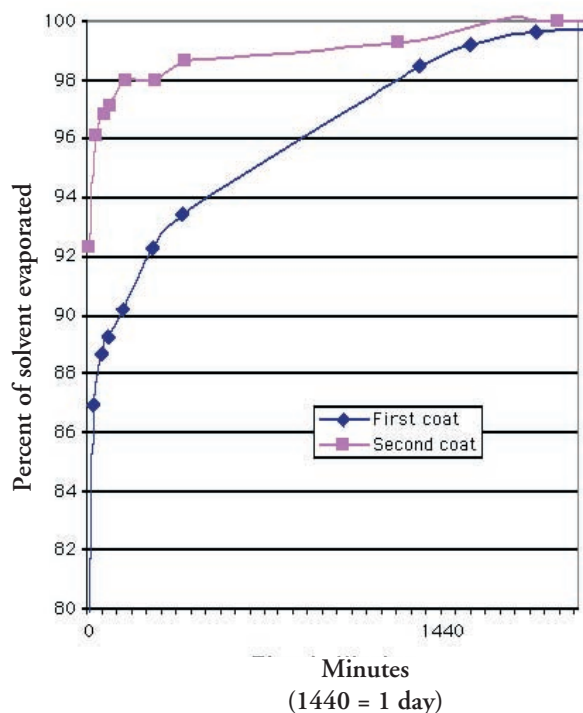


FIG. 3 Comparison of solvent evaporation rates from the first and second coats of 17%(w/v) B-72 in acetone when applied to hard maple.

a bond which is weakened even after the eventual drying of the solvent (the retained solvent could impair the bonding of the epoxy to the resin, or the shrinkage of the resin film during drying could cause internal stresses within the joint). Based on our results, it should also be considered that when applied to wood, a significant portion of the solvent may be retained, not in the resin layer, but in the wood itself. In this case, any remaining solvent would not be likely to contribute to plasticizing or weakening the barrier layer. In any event, more testing is clearly warranted to determine the relationship between solvent retention and the strength of barrier coatings on wood substrates.

### Strength Testing

Unfortunately, there is no ASTM standard for determining the strength of combinations of barrier coatings and adhesives. Where possible, this study used the testing methodology as specified by ASTM Designation D 905-98 "Standard Test Method for Strength Properties of Adhesive Bonds in Shear by Compression Loading" and

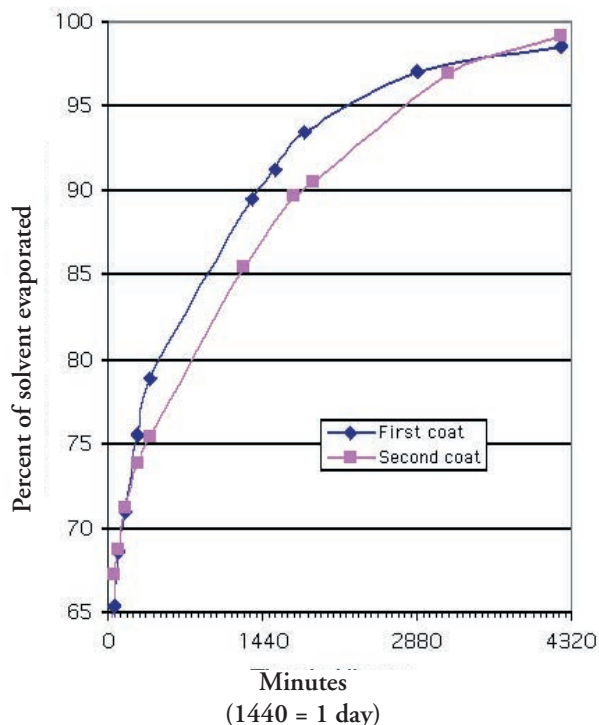


FIG. 4 Comparison of solvent evaporation from the first and second coats of 17%(w/v) B-67 in Shell Mineral Spirits 135 when applied to hard maple.

modifications were made where necessary. Modifications were in some cases based on those made by Podany *et al.* in their 2001 study and by Anderson and Podmaniczky in 1990.

### Determination of specific gravity

ASTM Designation D 905-98 specifies the use of hard maple for shear strength testing of adhesives. It further stipulates that the maple used fall within a certain range of specific gravity. In order to measure the specific gravity of the maple obtained for this test, two small pieces of the wood were oven dried, according to the specifications of ASTM Designation D 143-94, "Standard Test Methods for Small Clear Specimens of Lumber." The two samples were weighed, and then placed in an oven at 103° C until their weight loss ceased changing. The moisture content of the wood was then determined by dividing the samples' loss in mass by the oven-dry mass. These results were used to calculate the specific gravity of the blocks as shown in Appendix X1 of ASTM D 905-98 (American Society for Testing and Materials 2001, 25). The

specific gravity of the hard maple stock fell in an acceptable range.

### Sample Preparation

The size of the samples specified by ASTM Designation D 905-98 was too large for the Getty Conservation Institute's Instron tensile testing machine which is limited by a 10 kN load cell. Based on the ultimate strength of trial samples of different sizes prepared for this purpose, it was calculated that the machine would be able to run samples of approximately one quarter of the specified size. The final configuration of the test blocks is shown in figure 5, resulting in a bond area of one square inch.

The  $\frac{3}{4}$ " x  $9\frac{1}{4}$ " hard maple stock was cut into  $1\frac{1}{4}$ " strips across the grain on a table saw. Surfaces to be glued were prepared by sanding lightly with 320 grit abrasive paper. The wooden strips were divided into five groups, four of which were coated on one face with each of the respective barrier materials in the manner described under barrier application protocol. The fifth group was not coated with any barrier material and served as a control. After drying, pairs of strips from each group were bonded together using Araldite 1253 in the manner described below. The two components of the epoxy were measured before mixing by weight according to the product data sheet, provided by manufacturer Vantico, which specifies the optimal resin/hardener weight ratio at 100/82.

One of the strips, already coated with the barrier, was covered with the epoxy paste and laid, face up, in a jig built for this experiment. The second strip was placed into the jig above the first, overhanging by approximately  $\frac{1}{4}$ ". The upper strip was pushed down in the jig so that the adhesive layer measured 0.030 in. (30 mils) thick. This adhesive layer thickness was chosen to approximate a typical gap-filling bond as might be required in wooden artifacts conservation. The samples were removed from the jig and left to cure for eight days. Excess epoxy was removed from the edges of the strips first, using a shoulder plane.

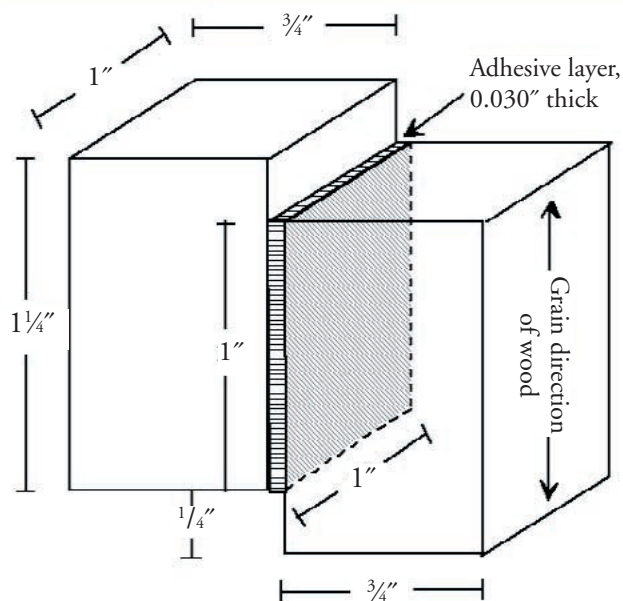


FIG. 5 Dimensions of hard maple samples prepared for shear strength testing.

Once the samples had been cleaned of excess adhesive, the bonded wooden strips were then cut into smaller pieces, approximately 1" wide, on a table saw. Ten small test blocks were prepared and labeled for each barrier material. The bond area of each sample block was then calculated by measuring the width and length of the bonded area. This data was recorded for use in calculating the final load at failure for each test block.

### Shear Strength Measurement

The samples were tested in batches according to sample type by the same operator during a four-hour run. In order to keep the samples in the same conditions, they were held in sealed polyethylene bags until just before testing. Ten samples per sample type were run. Both parts of each sample were labeled in pencil according to adhesive type and numbered in sequence, for post-testing analysis of the break edge.

A Model 4201 Instron with a 10 kN load cell, belonging to the Getty Conservation Institute, was used to test the samples (fig. 6). Instron Series IX Automated Materials Tester software, version 8.06.00, was used to run the tests and to partially analyze the data. Before each sample was run, the operator entered the width and thickness of each

sample. The Instron's moving cross-head was configured to push down on one of the two bonded sample blocks while the other was held in a fixed position creating a shear stress on the adhesive bond (fig. 7). The cross head was set to move down at a constant rate of 5 mm/min., the ASTM standard specified speed, until the sample failed.

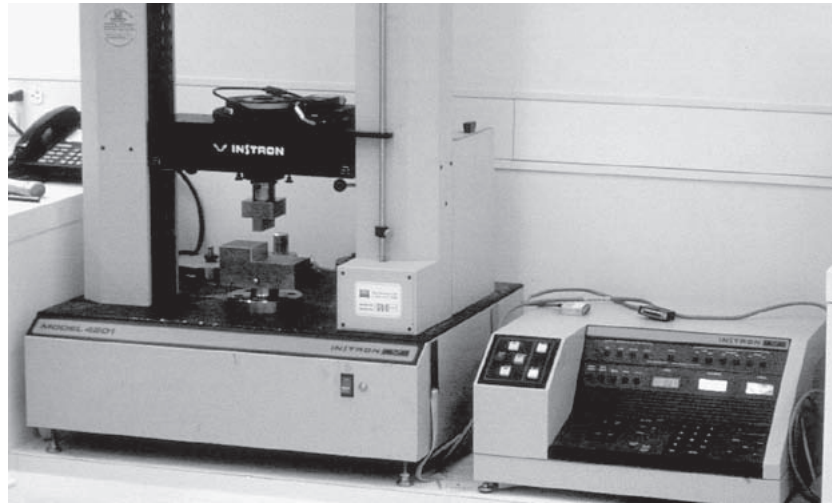


FIGURE 6

For each sample, the Instron Series IX Automated Materials Tester software then calculated the maximum load, displacement of the cross-head at maximum load, and stress at maximum load, as well as the mean and standard deviation of the samples grouped together. The software also produced graphs showing these results.

The quantitative results of the Instron testing were then subjected, by group, to the "Q" test for outliers at the 90% confidence level. This test is part of ASTM designation 3980. The test considers the number of samples and the distribution of the results: any individual result deemed too incompatible with the spread of the others is excluded within a determined confidence level.

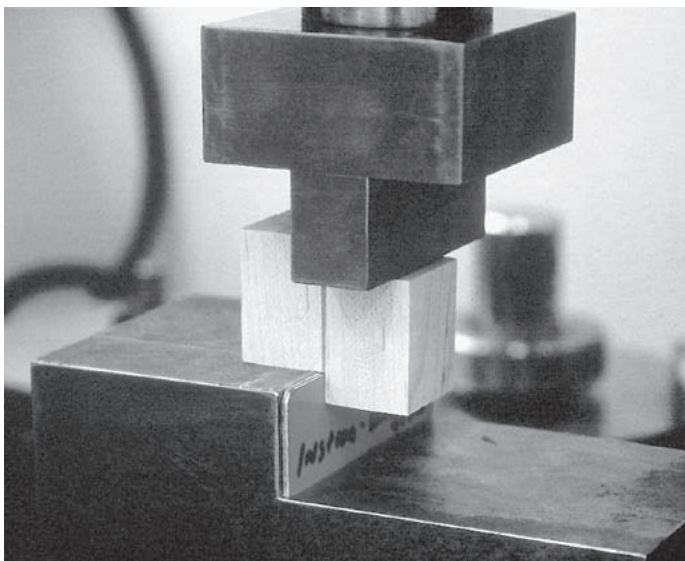


FIGURE 7

### Results of Shear Strength Testing

Table 2 and Figure 8 show the results of the Instron shear strength testing. Araldite 1253 epoxy bonds prepared with B-98 and B-72 barrier layers proved to be as strong or stronger than bonds prepared with no barrier. Bonds prepared with liquid hide glue barrier layers were weaker on average, but nearly as strong as bonds with no barrier, while bonds prepared with B-67 barrier layers were much weaker than any other category. These results indicate that B-98, B-72 and liquid hide glue barrier layers yield high strength epoxy bonds and can be considered suitable barrier materials for use with wood and epoxy adhesive. Conversely, the use of B-67 was shown to result in consistently weak bonds and is clearly unsuitable for use as a barrier material. The failure of B-67 to produce a sufficiently strong bond was disappointing since it could have provided a barrier method reversible in solvents of low polarity (safe for objects) and low toxicity (safe for conservators). This failure may be directly related to the low polarity of the B-67 polymer. It may be that epoxy resin, which is a highly polar material (Down, 2001), is unable to bond satisfactorily to such a low polarity material. This hypothesis is supported by the fact that 100% of failure occurred between the B-67 and epoxy layers (see next section). If this is in fact the case, then the search for a strong barrier material soluble in low or non-aromatic solvents may be inherently unlikely to succeed.



	Mean Pressure at Failure	Standard Deviation	Samples Tested	Results Excluded
No barrier	1301 psi	±233	10	1
Butvar B-98	1403.1 psi	±134.6	9	1
Paraloid B-72	1350.4 psi	±327	10	—
Liquid Hide Glue	1153.8 psi	±366.9	9	—
Acryloid B-67	251.8 psi	±78	9	—

TABLE 2 Results of shear strength testing using epoxy adhesive alone and with four different barrier layers.

### Failure Analysis—Estimated Percentage Wood Failure

ASTM Designation D 905-98 specifies that an estimated percentage wood failure be calculated. In a simple adhesive testing scenario, this serves to distinguish between areas in which the adhesive has failed and areas where the wood has failed. This study presented a more complex situation than anticipated by ASTM standards because of the use of the barrier coatings. The control samples prepared with direct epoxy bonds were analyzed according to the ASTM standard, which distinguishes only between wood failure and adhesive failure. For samples prepared with barrier adhesives, failure was divided into the following four groups:

1. Wood failure, wherein wood was removed from one of the faces of the wooden sample.
2. Barrier coating/wood failure, meaning that the barrier coating was pulled from the wooden face, sometimes, but not always taking tiny wood fibers along with it.
3. Barrier coating/epoxy failure, where the joint failed in the interface between the two.
4. Epoxy failure, where the epoxy adhesive was pulled apart and remains were found on both faces of the sample.

For all samples, the percentage failure of each type per sample was defined using a gridded, transparent plastic ruler (fig. 9). The grid, which divides square inches into 254 units, was placed on top of each bond surface after failure and examined under a stereo microscope. The number of square

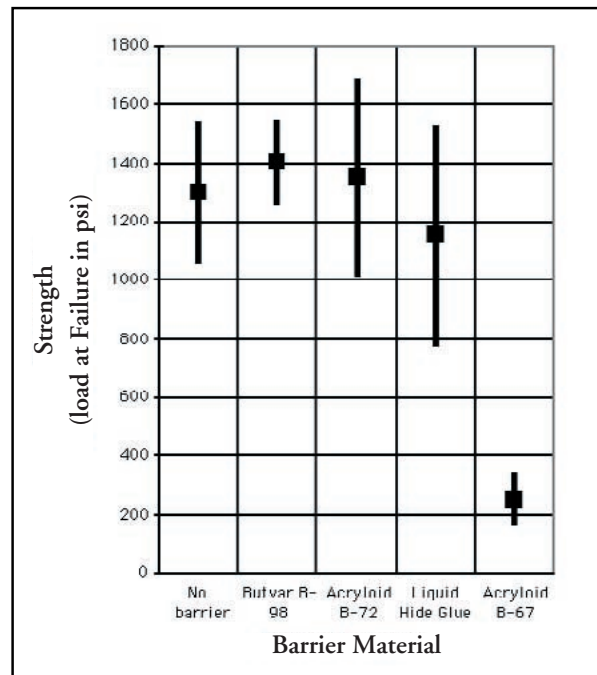


FIG. 8 Results of Instron shear strength testing of epoxy bonds with different barrier materials. The graph shows the mean strength of the ten samples tested and indicating the range of one standard deviation.

units in which each type of failure occurred were counted, and the percentage failure of each type was calculated.

In the event that an epoxy bond on a wooden artifact is stressed to the point of failure, it is preferable that the adhesive break away cleanly from the substrate without causing additional damage to the wood. The results of the failure analysis, shown in Table 3, indicate that all four barrier materials tested offer some protection to the underlying

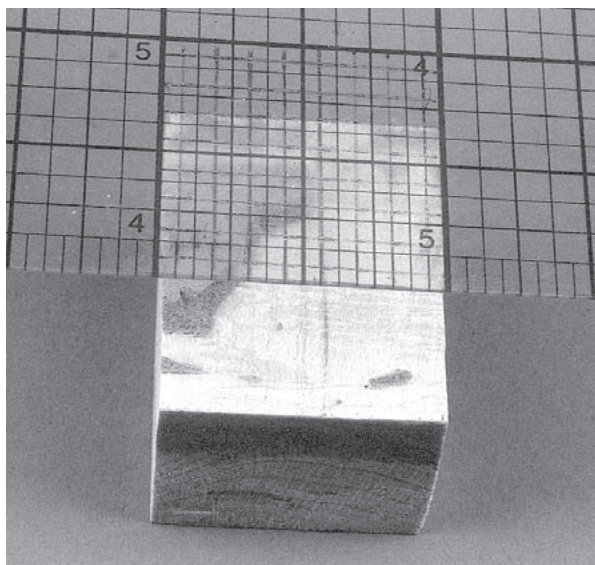


FIGURE 9

wood by reducing wood failure when the bond is broken. Wood failure, which was 6.8% for wood bonded directly with epoxy, was reduced to nil or virtually nil when any of the four barriers was used. B-98 and liquid hide glue samples tended to fail at the interface between the epoxy and the barrier layer, while B-72 tended to fail at the interface between the wood and the barrier. B-67, which failed the overall strength testing, always failed at the interface between the epoxy and the barrier, indicating that the cause of failure was poor adhesion between the B-67 and the epoxy.

### Reversibility of Barrier Coatings in Solvent Vapors

In order to test the reversibility of the barrier coatings, spare sample blocks of maple, coated with the

barriers, were bonded to one another using Araldite 1253. The three barrier materials reversible in organic solvents (B-72, B-67, and B-98) were tested for reversibility in solvent vapor chambers with no direct application of liquid solvent. After the epoxy had cured, screws were inserted into the upper and lower pieces of wood. Four large steel washers were suspended from the bottom screws. The total weight of the four washers was approximately 88 grams. The samples were suspended by the upper screws on bamboo skewers laying on the rim of glass beakers, which were enclosed in a sealed, clear polyethylene bags (fig. 10). Approximately 2 ml of the appropriate solvent was placed in the bottom of the beakers. Xylene was used for the sample bonded with Paraloid® B-72; ethanol for the Butvar® B-98 sample; and Shell Mineral Spirits 135 for the Acryloid® B-67 sample.

The Butvar B-98 sample fell apart on its own in three to four days. The Paraloid® B-72 sample came apart with gentle pressure after five days in the solvent rich environment. The Acryloid® B-67 coating did not fall apart, even with gentle pressure, after five days, at which point the test was suspended. These result suggest that B-98 and B-72 barrier layers are practically reversible in ethanol and xylenes respectively, even without direct application of liquid solvent. The disadvantages of using ethanol for reversal on objects with painted or varnished surfaces have been discussed above. While xylenes should be safe to use with many painted or varnished surfaces, it will not be safe with all. Furthermore, the health hazards asso-

	Wood Failure	Barrier coating/ wood failure	Barrier coating/ epoxy failure	Epoxy failure
No barrier	6.8%	n/a	n/a	93.2%*
Butvar B-98	0.3%	16.6%	71.8%	11.3%
Paraloid B-72	nil	62.1%	37.9 %	nil
Liquid Hide Glue	nil	27.3%	64.7%	8
Acryloid B-67	nil	nil	100%	nil

\* indicates non-wood failure according to ASTM D 905-98

TABLE 3 Failure analysis

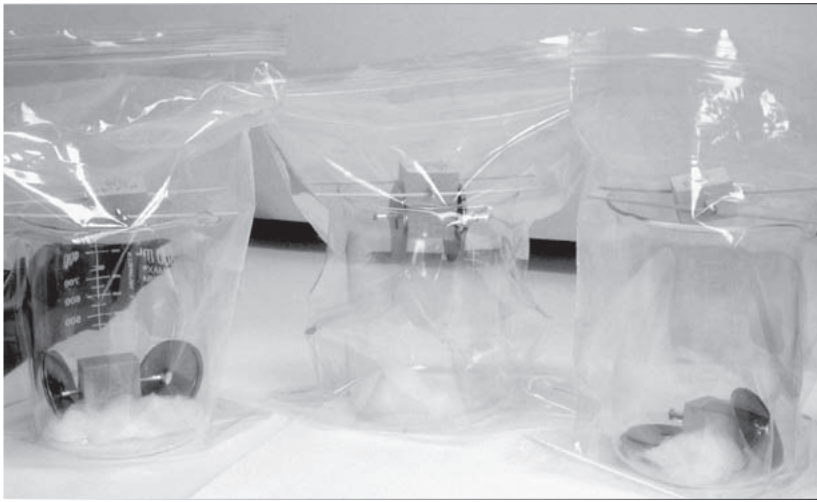


FIGURE 10

ciated with xylenes makes them a less-than-ideal choice for barrier reversal. The failure of the B-67 barrier layer to be reversed by the vapor of mineral spirits (in which it had previously been dissolved) is somewhat mysterious, though it may be related to the low vapor pressure of the solvent. Had it not been for the failure of B-67 to perform adequately in strength testing, further testing of more volatile and/or more polar solvents might have been warranted.

#### Microwave Reversibility of Hide Glue

The possible utility of microwave technology for reversing hide glue joints has not gone unnoticed, especially by furniture conservators (Neher, 1996 and Anderson & Podmaniczky, 1990). Theoretically, microwave radiation can be used to excite the hide glue's water molecules, heating and weakening the glue line to the point that it either falls apart or comes apart with gentle pressure. In practice, consumer microwave ovens can be used to deliver the microwaves if an object is small enough; otherwise there are hand-held devices (available at considerable expense) such as the WorkRite Wood Welder, which generate radio frequencies for use in industrial applications.

A simple experiment was undertaken to separate sample blocks that had been bonded with epoxy using Titebond Liquid Hide Glue as a barrier coating. The samples were heated in a microwave oven: some had water injected into the bond line with a very fine syringe. All samples came apart

easily after 20-30 seconds. Intentional over exposure in the microwave oven resulted in scorching of the wood blocks, demonstrating that this method of reversal has some potential dangers. Microwave reversal is also not suitable for joints in close proximity to metal fasteners or ornaments. While the reversal of hide glue barrier layers with microwave radiation cannot be universally recommended, further study is called for in this area as the tech-

nology might prove to be useful.

#### CONCLUSION

This study demonstrates that Paraloid® B-72 is a suitable material for use as a reversible barrier layer for epoxy joints in wood. It offers strength comparable to epoxy used alone as well as to other proven and widely used barrier materials (Butvar® B-98 and hide glue). Like these other barrier materials, B-72, appears to offer some protection to underlying wood in the event that the epoxy bond is broken. Additionally, B-72 was shown in practice to be a reversible barrier material in xylene vapor. This offers significant advantages over B-98 and hide glue when making repairs near finished, painted or otherwise sensitive surfaces. This study also demonstrates that Acryloid® B-67 is not a suitable material for use as a reversible barrier layer for epoxy joints in wood. B-67 failed both strength and reversibility tests.

#### ACKNOWLEDGEMENTS

The authors would like to thank especially the J. Paul Getty Museum's Brian Considine, Jane Bassett, Julie Wolfe, George Johnson and Mark Mitton of Decorative Arts and Sculpture Conservation, along with Jerry Podany of Antiquities Conservation, for their support and help throughout the project; Stefan Simon of the Getty Conservation Institute for permission to use the Institute's equipment; Urs Mueller, for providing cheerful advice, Instron training and set-up; and

Jane Down of the Canadian Conservation Institute for her helpful guidance.

#### MATERIALS SOURCES

Available from Conservation Support Systems, Santa Barbara, CA 93101:

- Acryloid® B-67, poly (iso-butyl methacrylate)
- Butvar® B-98, Polyvinyl Butyral Resin
- CIBA Araldite AV 1253®, Vantico Inc.
- Paraloid® B-72, copolymer of ethyl methacrylate and methyl acrylate
- Shell Mineral Spirits 135, slow evaporating, 15% aromatic content

Available from Franklin International, Columbus Ohio, 43207

- Titebond® Liquid Hide Glue

#### BIBLIOGRAPHY

American Society for Testing and Materials. 2001. Designation D 905-98: Standard Test Method for Strength Properties of Adhesive Bonds in Shear by Compressive Loading. *Annual Book of ASTM Standards* 15.06: 22–25.

Anderson, Mark, J. & Michael S. Podmaniczky. 1990. Preserving the Artifact: Minimally Invasive Conservation Treatment at the Winterthur Museum. *Papers from the Wooden Artifacts Group*. Richmond: Wooden Artifacts Speciality Group, AIC.

Blackshaw, S. M. & S. E. Ward. Eds. J. O. Tate, N. H. Tennent & J. H. Townsend. 1983. Simple Tests for Assessing Materials for Use in Conservation. *The Proceedings of the Symposium Resins in Conservation*. Edinburgh: Scottish Society for Conservation and Restoration. 2.1–2.15.

Bradley, S. 1984. Strength Testing of Adhesives and Consolidants for Conservation Purposes. *Adhesives and Consolidants*. London: IIC. 22–24.

Buck, Susan L. 1990. A Study of the Properties of Commercial Hide Glue and Traditional Hot Hide Glue in Response to Changes in Relative Humidity and Temperature. *Papers from the Wooden*

*Artifacts Group*. Richmond: Wooden Artifacts Speciality Group, AIC.

Ciba Specialty Chemicals Corporation, North America. 2001. Araldite 1253 packaging materials. Down, J. L., J. MacDonald, J. Tetreault and R. S. Williams. 1996. Adhesive Testing at the Canadian Conservation Institute, Past and Future. *Studies in Conservation* 41:19–44.

Down. 2001. Review of CCI Research on Epoxy Resin Adhesives for Glass Conservation. *Reviews in Conservation*. 2:39–46

Horie, C. V. 1987. *Materials for Conservation*. London: Butterworths.

Koob, Stephen P. 1986. The Use of Paraloid B-72 as an Adhesive: Its Application for Archaeological Ceramics and Other Materials. *Studies in Conservation* 31: 7–14.

Monsanto Co. 1994. *Butvar Polyvinyl Butyral Resin—Properties and Uses*, Pub. No. 2008084B.

Neher, Albert. 1996. Radio Frequency Heating and the Reversibility of Animal Glue in Furniture Joinery. *UKIC Conservation News* 59: 35–37.

Podany, J., Garland, K. M., Freeman, W. R. & J. Rogers. 2001. Paraloid B-72 as a Structural Adhesive and as a Barrier within Structural Adhesive Bonds: Evaluations of Strength and Reversibility. *JAIC* 40: 15–33.

Spirydowicz, K., E. Simpson, R. A. Blanchette, A. Schniewind, M. K. Toutloff & A. Murray. 2001. Alvar and Butvar: The Use of Polyvinyl Acetal Resins for the Treatment of the Wooden Artifacts from Gordion, Turkey. *JAIC* 40: 43–57.