CHOOSING AN ADHESIVE FOR EXTERIOR WOODWORK THROUGH MECHANICAL TESTING

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Through mechanical testing, an appropriate adhesive was found for reuniting the fragments of exterior wooden capitals. The adhesive needed to have superior weather resistance, flexibility, gap-filling capability, reversibility, longevity, and workability. From four different adhesive classes, a candidate adhesive was chosen for testing: Butvar B98 with granulated cork, ConServ Flexible Epoxy Patch 200, 3M Marine Adhesive Sealant 5200, DAP Dynaflex 230, and Dow Corning 738 Silicone Rubber with microballoons. The test program included cyclical tensile and compression tests, a uniaxial tensile test, and accelerated weathering. The Epoxy proved to be the most brittle, Butvar showed fatigue until failure, while 3M had excellent properties such as a low elastic modulus (high ductility), (visco)elastic behavior, and longevity. Both DAP and Dow Corning developed fissures in the paint prematurely and were eliminated from extensive cycling tests.

KEYWORDS: *Adhesive, Cyclical tensile and compression test, Uniaxial tensile test, Weathering, Architectural, Exterior woodwork, Conservation, 3M 5200, Butvar B98, Silicone rubber, DAP, Epoxy, Cork, Gap-filling, Flexible*

1. BACKGROUND

Four Ionic capitals, carved out of southern yellow pine, are situated on the exterior of the palladium window of Mount Pleasant, a historic house in Fairmount Park, Philadelphia. The house is open to the public and is administered by the Philadelphia Museum of Art (PMA), in partnership with the City of Philadelphia through the Department of Parks and Recreation.

The house was built for Scottish Ship Captain John Macpherson (1726–1792) and his first wife, Margaret, between 1762 and 1765. Thomas Nevell (1721–1797), an apprentice of Edmund Woolley, the builder of Independence Hall, became the builder and architect of this grand house. The interior has been considered one of the finest surviving examples of Philadelphia architectural carving attributed to the workshop of leading craftsmen Nicholas Bernard and Martin Jugiez.[1] The exterior capitals appeared to be exactly the same as the interior ones and were likely made by the same artisans.

The exterior woodwork was assessed as part of a 2-year preservation project for Mount Pleasant that was started in 2004. Clogging of the highly detailed carving by many coats of paint made it necessary to treat the capitals. After taking them off the building and removing approximately 20 layers of paint, substantial damage to the wood was apparent (Deurenberg 2004) (fig. 1). Each of the capitals was carved out of a solid piece of southern yellow pine with the grain running vertically and the reverse cut out to fit around the pilasters. The wood had split along the grain several times, resulting in five to nine major fragments for each capital. Some of the splits could not be joined together without filling the gaps that were up to 3 mm wide.

2. INTRODUCTION

Adhesives for historic exterior woodwork face more challenging conditions than those used in a museum environment. Flexibility is an important feature for an exterior adhesive, due to the dimensional changes of the woodwork in conditions of widely ranging humidity. In addition, the adhesive often needs to fill gaps because of deterioration of the substrate. Modern polymers, with or without a filler, have many of the desired features of an exterior adhesive.

Tensile testing is frequently employed to investigate stress–strain relationships of polymer adhesives, such as elastic modulus and maximum elongation (Down 1984). For applications with cyclically induced dimensional changes such as exterior woodwork, cyclical tensile and compression testing can give additional insights (Mintrop 1997). One can gather information on the type of deformation (plastic, viscoelastic, or elastic) and the adhesive's strength after repeated loads of compression and tension. The capitals at Mount

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Fig. 1. One of four capitals from Mount Pleasant, shown inverted and in fragments after removal of roughly 20 layers of paint. Attributed to Bernard and Jugiez, Philadelphia, 1762–1765, southern yellow pine, 9.8 × 29.8 × 15.7 cm ($H \times W \times D$), Philadelphia Museum of Art.

Pleasant were a prime example of exterior woodwork that would benefit from cyclical adhesive testing.

The successful adhesive for the capitals needed to have many different properties for a long-term exterior application. The adhesive needed to be resistant to weathering (moisture, cold, and heat) and fit the maintenance schedule for Mount Pleasant, meaning it should be stable for at least 10 years and not inhibit paint adhesion of the mandated Old Village Alkyd paint. To be resistant to weathering, the adhesive should have a T_g of at least 45°C, based on a record maximum temperature in Philadelphia of 41°C,[2] without considering solar heat gain, caused by the eastern location of the capitals on the building.

To comply with current conservation standards, the adhesive further needed to be reversible or retreatable, with or without a barrier layer. The wood-adhesive bond should be weaker than the cohesion of the wood, to ensure failure would take place within the adhesive layer or at the wood-adhesive interface, rather than in the original material.

As the capitals would be affixed to the pilasters with screws, hygroscopic dimensional changes in the wood would largely need to be absorbed within the width of the capitals. Gaps of 1.6 mm or wider between fragments would be (at least partially) filled with adhesive. These adhesive-filled gaps could absorb part of the dimensional changes of the capitals to avoid damage to the original material, such as compression set or splits. For a "worst case scenario," i.e. untreated wood, strictly tangential (direction of the wood with largest dimensional change), a wide range of humidity, and assuming all movement in the capitals would be in the joins and none in the wood itself, the amount of dimensional change that each join would need to be able to absorb was calculated to be ±0.3–0.4 mm, with

$$\Delta D = D_I[C_T(M_F - M_I)] \qquad (1)$$

ΔD was the change in dimension (mm) and D_I the initial dimension (304.8 mm), attained through matching the width of the pilasters on the building to the cutout of the capitals. The dimensional change coefficient C_T was between 0.00263 and 0.00271 for southern yellow pine (longleaf, shortleaf, slash, or loblolly) in the tangential direction. M_F and M_I were the final and initial moisture content (The Forest Products Laboratory 1974). With the average relative humidity (RH) in Philadelphia ranging from 55% (May, P.M.) to 77% (September, A.M.),[3] the equilibrium moisture content would be between 10% (M_I) and 14% (M_F) for unpainted and untreated wood (Hoadley 1980). Using equation 1, the total dimensional change for the capitals would be 3.3 mm, or 0.6–0.8 mm once divided over four to six joins. From its equilibrium dimension, the dimensional change would only be ±0.3–0.4 mm per join, or ±25% for a filled gap of 1.6 mm.

The aim of this research was to find an adhesive with the above-mentioned properties to reunite the ill-fitting fragments of the capitals. To accomplish this aim, cyclical tensile and compression tests were conducted on 10 different adhesive combinations.

3. Adhesives for Exterior Woodwork

Four classes of adhesives appeared suitable for adhering the fragments of the capitals, although they each had different assets: bulked thermoplastic resins, epoxies, silicone rubbers, and certain commercial adhesive formulations.

Thermoplastic resins often are highly reversible, but may be more susceptible to changes in temperature and humidity and are generally inferior gap-fillers (Schniewind and Kronkright 1984; Hatchfield 1986; Stappel 2000). Thermoplastic adhesives form a fairly rigid, solid adhesive line that is not suitable for filling gaps between two ill-fitting fragments of an object. Active fillers improve the mechanical properties of a system (such as elasticity, paintability, chemical resistance, etc.), while inactive fillers or extenders are used mostly to increase volume or lower costs (Mintrop 1997). An active filler is needed to give a thermoplastic system gap-filling capacity as well as the ability to take deformations. The ability to deform is based on the formation of adhesive strands and open spaces between particles of the filler, after evaporation of the solvent (Mintrop 1997). A disadvantage is that these open spaces are prone to hold water (rain), which may cause unnecessary swelling of the surrounding wood and may induce growing of molds.

Epoxies can have excellent gap-filling abilities, generally have good durability of 5–10 years, and good working properties (Fisher and Sheetz 1993; Norman 2002). However, the elastic modulus of most epoxies is too high to exhibit good flexibility in their polymerized state (Grattan and Barclay 1988; Barclay and Matthias 1989).

Silicone rubbers are known for their low elastic modulus, but have poor working properties and are less compatible with paint (Barclay and Grattan 1987; Storch 1994). Silicone rubbers are completely closed systems and hence would not have the problem of trapping moisture in a network of cavities. To improve their working properties and paintability after curing, an active filler should be added. Barclay and Grattan (1987) attribute the following properties to a mixture of silicone rubber and microballoons: low elastic modulus (easily deformed) and very elastic (will regain original form), inert after curing, water repellent but very permeable to moisture. The resultant surface can be carved, sanded, and painted and should last at least 10 years in an exterior environment (Barclay and Grattan 1987). Many types of fillers can improve the properties of silicone rubber in terms of adhesion, cohesion, and shrinkage (Mintrop 1997).

Commercial adhesive formulations appear to have many of the features of an exterior adhesive, but their longevity and reversibility are largely unknown (deMuzio 2003; Lopuszanski 2003).

4. Experimental

4.1 Tested adhesives

The following adhesives were selected for testing: 35% Butvar 98 with granulated cork in two ratios (1:1 and 2:1), ConServ Flexible Epoxy Patch 200, Dow Corning 738 RTV Silicone rubber with phenolic microballoons, 3M Marine Adhesive/Sealant 5200, and DAP Dynaflex 230 Premium Elastomeric Latex Sealant (table 1).

4.1.1 Butvar B98 with granulated cork

Butvar B98, a polyvinyl butyral, was preferred over other thermoplastic resins such as Paraloid B72 and Plexigum PQ610 because of its high T_g of 62°C–68°C, compared to a T_g of 40°C and 32°C, respectively (Stappel 2000). A high T_g would make the adhesive more mechanically stable (less creep) in higher temperatures. Butvar B98 also had good tensile strength (Schniewind and Konkright 1984). Cork was added as a filler, because of its low elastic modulus and resistance to weathering (Mintrop 1997). Microballoons

Table 1. Tested Adhesive Polymers

Adhesive	Composition	Barrier	Symbol
35% (w/v) Butvar B98 with cork 1:1 (w/w)	3.0 g Butvar (approx. 35% w/v) 3.0 g cork (dry and crumbly mixture)	No barrier	1:1
35% (w/v) Butvar B98 with cork 2:1 (w/w)	6.0 g Butvar (approx. 35% w/v) 3.0 g cork (dry and crumbly mixture)	No barrier	2:1
ConServ Flexible Epoxy Patch 200	30.0 ml Component A 10.9 ml Component B 62.5 ml Component C 125.0 ml Component D (very dry and thick paste)	Butvar B98 barrier (approx. 10% w/v)	X
Dow Corning 738 RTV with phenolic microballoons 1:2 (v/v)	97.8 g DC 738 12.7 g microballoons Mixed in a Ziploc bag by kneading. (sticky, thick paste)	Butvar B98 barrier (approx. 10% w/v)	DC
3M Marine Adhesive/Sealant 5200	Used straight from tube (very sticky paste)	Butvar B98 barrier (approx. 10% w/v)	3M
DAP Dynaflex 230 Premium Elastomeric Latex Sealant	Used straight from tube in caulking gun (moderately thick paste, easy to work with)	Butvar B98 barrier (approx. 10% w/v)	DAP

would make the fill brittle, not flexible, and decrease adhesion to the wood significantly (Barclay and Matthias 1989).

Two ratios of Butvar B98 with cork were tested: 1:1 and 2:1. The higher adhesive content of the 2:1 mixture would probably improve adhesion to the surfaces to be glued and have a higher breaking point (F_{max}), but would shrink more. The lower adhesive content of the 1:1 mixture would likely lower the breaking point and result in a more gradual failure at the interface with the substrate, but be less brittle (Mintrop 1997).

Butvar B98 was dissolved in ethanol at a 35% ratio (w/v), and bulked with granulated cork, sifted through wire mesh with openings of 1 × 1 mm.

4.1.2 ConServ Flexible Epoxy Patch 200

ConServ Flexible Epoxy Patch 200 was claimed to be flexible enough to withstand some of the ongoing expansion and contraction of wood. It was intended for filling cavities, rather than structural adhesion, and would be soft enough to be cut after curing. It was applied according to the manufacturer's instructions (table 1).

4.1.3 Dow corning 738 RTV silicone rubber with phenolic microballoons

Dow Corning 738 RTV silicone rubber (DC738), a polydimethyl siloxane), has been a common choice for a flexible adhesive in conservation. It was bulked for paintability with phenolic microballoons (1:2 by volume) (Mintrop 1997).

4.1.4 Commercial adhesive formulations

4.1.4.1 3M Marine Adhesive/Sealant 5200

3M Marine Adhesive/Sealant 5200 was chosen by recommendation (deMuzio 2003). It is a one-part polyurethane adhesive and sealant that chemically reacts with moisture to deliver strong, flexible bonds with excellent adhesion to wood. It forms weather resistant seals on joints. In addition, it will dry in 48 h and completely cure in 5–7 days with no shrinkage (3M 2000). According to the manufacturer's data sheet, it usually fails cohesively when adhered to wood, which is desirable in view of the safety of the object (3M 2000).

4.1.4.2 DAP Dynaflex 230 Premium Elastomeric Latex Sealant

DAP Dynaflex 230 Premium Elastomeric Latex Sealant (DAP) (acrylic latex) is a tight sealant with the durability and low elastic modulus of a silicone and a 50-year durability guarantee. It is mildew resistant, paintable, and permanently flexible without cracking. It has outstanding adhesion to wood (DAP 2003).

It was also chosen by recommendation (Lopuszanski 2003).

4.2 Testing

In conjunction with the Analytical Lab at the PMA and scientists at Rohm & Haas and DuPont, a setup was designed for testing (table 2).

4.2.1 Basic cyclical tensile and compression tests for first elimination

As a first means of elimination, samples of 10 different adhesive combinations underwent 500 cycles in an Applied Test Systems, Incorporated (ATS) Cyclical Tester, Series 904, which was a computer-controlled vertical sealant tester for construction materials. Samples were cycled at a displacement of 0.4 mm and a speed of 3 cycles/min with a controlled temperature of 24°C and 50% RH (fig. 2).

The following combinations were tested: Butvar B98 with granulated cork 1:1, Butvar B98 with granulated cork 2:1, Conserv Flexible Epoxy Patch (with and without a barrier), DC 738 with phenolic microballoons (with and without a barrier), 3M Marine Adhesive Sealant 5200 (with and without a barrier), and DAP Dynaflex 230 (with and without a barrier). Of each combination, there were two painted and two unpainted samples (table 2).

4.2.2 Comprehensive cyclical tensile and compression tests

New samples of adhesives that were not eliminated after the basic cycling tests underwent cyclical compression and tensile tests in a Mechanical Testing and Simulation Systems (MTS) Tensile Tester at a speed of 60 cycles/min. All samples underwent 4000 cycles between ±0.4 mm and an additional 2000 cycles at ±0.6 mm. The number of cycles (4000) was based on 3650 daily cycles for 10 years, the minimum period the adhesive is preferred to last in the capitals. An adhesive that would pass 4000 cycles would probably have more durability than needed for a period of 10 years.

Two batches of samples were cycled, of which one was exposed to accelerated aging in an ATLAS Material Testing Technology LLC Ci5000 Xenon Weather-Ometer (WOM), a large capacity laboratory weathering instrument with a 12,000 W water-cooled xenon arc lamp light source (table 2). Groups of four samples were mounted together on one Weather-Ometer plate that was attached to a rotating base for even exposure to light and spray. Samples were exposed to the ASTM G-155 schedule of 18 min spray, 102 min dry and continuous light (0.32 W/m @ 340 nm) for a period of 1000 h (~6 weeks).

Each batch contained samples of the following adhesives: Butvar B98 with granulated cork 1:1, Butvar B98 with granulated cork 2:1, Conserv Flexible Epoxy Patch (with barrier), and 3M Marine Adhesive

Table 2. Sample Composition and Numbering

Test	Wood	Painted/ unpainted	Weathered	Chosen adhesive	Barrier	Sample number
Basic cyclical tensile and compression tests	19th-century southern yellow pine, tangential	Painted and unpainted	No	35% Butvar B98 with granulated cork, 1:1 (w/w)	No	40 unnumbered samples: two painted and two unpainted of each combination
				35% Butvar B98 with granulated cork, 2:1 (w/w)	No	
				ConServ Flexible Epoxy Patch 200	Yes	
				ConServ Flexible Epoxy Patch 200	No	
				DC 738 with phenolic microballoons, 1:1 (v/v)	Yes	
				DC 738 with phenolic microballoons, 1:1 (v/v)	No	
				3M Marine Adhesive Sealant 5200	Yes	
				3M Marine Adhesive Sealant 5200	No	
				DAP Dynaflex 230	Yes	
				DAP Dynaflex 230	No	
Comprehensive cyclical tensile and compression tests	19th century southern yellow pine, tangential	Painted	No	35% Butvar B98 with granulated cork, 1:1 (w/w)	No	1:1 C7–9
				35% Butvar B98 with granulated cork, 2:1 (w/w)	No	2:1 C7–9
				ConServ Flexible Epoxy Patch 200	Yes	X C7–9
				DC 738 with phenolic microballoons, 1:1 (v/v)	N/A	*Eliminated*
				3M Marine Adhesive Sealant 5200	Yes	3M C7–9
				DAP Dynaflex 230	N/A	*Eliminated*
			Yes	35% Butvar B98 with granulated cork, 1:1 (w/w)	No	1:1 C4–6
				35% Butvar B98 with granulated cork, 2:1 (w/w)	No	2:1 C4–6
				ConServ Flexible Epoxy Patch 200	Yes	X C4–6
				DC 738 with phenolic microballoons, 1:1 (v/v)	N/A	*Eliminated*
				3M Marine Adhesive Sealant 5200	Yes	3M C4–6
				DAP Dynaflex 230	N/A	*Eliminated*
Controls	19th century southern yellow pine, tangential	Painted	No (except for surviving cycled samples, some of which weathered)	35% Butvar B98 with granulated cork, 1:1 (w/w)	No	1:1 C10–12
				35% Butvar B98 with granulated cork, 2:1 (w/w)	No	2:1 C10–12
				ConServ Flexible Epoxy Patch 200	Yes	X C10–12
				DC 738 with phenolic microballoons, 1:1 (v/v)	N/A	*Eliminated*
				3M Marine Adhesive Sealant 5200	Yes	3M C10–12
				Surviving 3M cycled samples	Yes/ No	3M C4–6 (weathered), C7–8 (unweathered)
				DAP Dynaflex 230	N/A	*Eliminated*

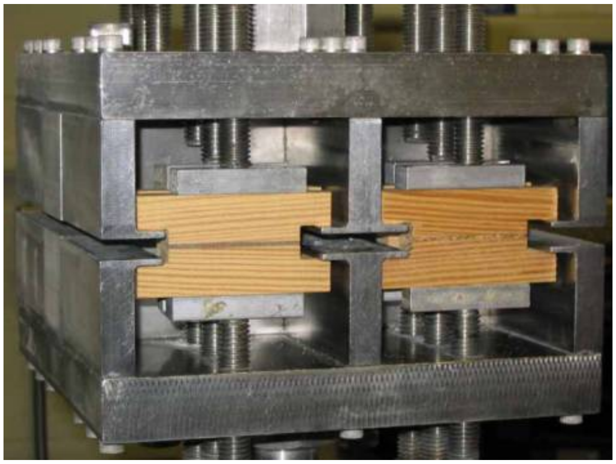


FIG. 2. Epoxy (left) and Butvar (right) samples in the ATS Cyclical Tester. These samples were unpainted and still had square notches. Samples for the comprehensive cycling and uniaxial tensile tests were all painted and had round notches.

Sealant 5200 (with barrier) (table 2). For each of the four adhesives, three samples were made, amounting to a total of 12 samples for each batch. All samples were painted.

4.2.3 UNIAXIAL TENSILE TEST ON CONTROLS

Control samples (no weathering or cycling) were pulled until failure at a speed of ca. 50 mm/min in a MTS 810 servohydraulic tensile tester with a TestStar IIM control and data acquisition package. The same adhesives were tested as in the comprehensive cycling tests: Butvar B98 with granulated cork 1:1, Butvar B98 with granulated cork 2:1, Conserv Flexible Epoxy Patch (with barrier), and 3M Marine Adhesive Sealant 5200 (with barrier) (table 2). For each of the four adhesives, three samples were made. In addition, the residual strength was examined of six 3M samples (weathered and unweathered) that had not failed during cycling, amounting to a total of 18 samples. All samples were painted.

4.2.4 SAMPLE PREPARATION

Samples for the basic cycling tests were made of an unidentified species of softwood (presumably pine, present in the Furniture Conservation lab at the PMA) that had a similar growth rate to that of the capitals. The orientation of the grain in these initial samples was approximately at a 45° angle, so neither tangential nor radial.

Samples for subsequent experiments (comprehensive cycling and uniaxial tensile tests) were prepared with wood from a log of 19th-century southern yellow pine that had a density of growth rings similar to the capitals. Orientation of the grain at the join was tangential. The breaks in the capitals, however, had any orientation between strictly tangential and strictly radial.

The size and shape of the samples were dictated by the ATS cyclical testing machine, used in the basic cycling tests (fig. 3). The samples had an overall dimension of 7.6 × 1.3 × 3.8 cm (3.0 × 0.5 × 1.5 in.) ($W \times D \times H$) and an adhesive surface of 5.1 × 1.3 cm (2.0 × 0.5 in.). The size of the joins in the samples was a factor 3 or 4 smaller than the largest joins in the capitals, which gave the advantage of a stronger and faster influence of the weathering on the complete thickness of the adhesives.

The samples were prepared from larger blocks of pine that were first barrier coated, then adhered, notched, and cut to size. Small strips of wood served as spacers while adhering the two parts of the samples to produce an even adhesive thickness of 1.6 mm. Notches, 1.3 × 1.3 × 1.3 cm (0.5 × 0.5 × 0.5 in.), at each end allowed insertion into the sample grips. Initially, the notches were square, but for the later comprehensive cycling and uniaxial tensile tests, the notches were rounded to help distribute stresses.

Before application of an adhesive that was not readily reversible (Epoxy, Silicone Rubber, 3M, and DAP Dynaflex 230), the wood was coated with a barrier layer of Butvar B98 (table 1). This layer was applied in two coats of a 10% solution in ethanol (w/v) to produce a coherent film that was allowed to dry for respectively 3 and 5 days (Ellis and Heginbotham, 2004). A barrier layer would also prevent staining of the wood by silicone rubbers.

Both painted and unpainted samples were tested (table 1). Painted samples received two coats of Old Village oil paint on one coat of white Old Village exterior oil base primer 1236 with a drying time of 3 days after each coat and light sanding with 220 sandpaper in between coats.

4.3 DATA ANALYSIS

The cyclical tensile and compression tests and uniaxial tensile test were designed to obtain information on the behavior of the adhesive, including paint and

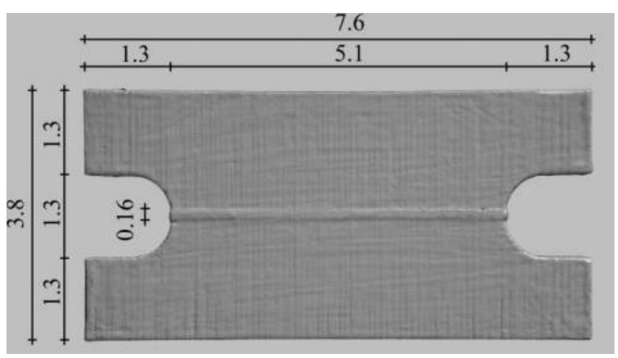


FIG. 3. One of the samples (3M C6) for cycling and uniaxial tensile tests with dimensions in centimeters

barrier layer compatibility, gap-filling ability, workability, failure type and location, maximum load and displacement, type of deformation, and compression behavior. The following sections clarify how the test data were interpreted to obtain this information.

4.3.1 Data collection

Samples were visually monitored for failure after 25, 50, 100, 250, and 500 cycles during the basic cycling tests in the ATS Tester.

During the comprehensive cyclical tensile and compression tests, load and displacement data were gathered throughout cycle numbers 1, 5, 10, 20, 50, 100, 200, 500, 1000, 2000, and 4000 for the first 4000 cycles; and cycle numbers 4001, 4005, 4010, 4020, 4050, 4100, 4200, 4500, 5000, and 6000 for the additional 2000 cycles.

For the uniaxial tensile tests in the MTS tester, similar data were gathered continuously until failure.

4.3.2 Initial evaluation and elimination

During the basic cycling tests, the samples were visually monitored for fissures in the paint or sample failure as a means of first elimination. The unpainted samples focused mostly on the behavior of the adhesive with or without a barrier layer, while the painted samples provided information on the compatibility of all materials (wood, barrier, adhesive, and paint). Workability of the adhesives was noted, as well as their ability to fill gaps, and the influence of the barrier layer.

4.3.3 Failure type and location

Failures were evaluated by type (fatigue or sudden failure) and location (adhesive, cohesive, or substrate). Graphs helped visualize how the adhesives failed.

Figures 4 and 5 depict graphs of the development of positive load over the first 4000 cycles in the comprehensive cycling tests, created from data gathered during tension of the cyclical compression and tension tests. These graphs showed the type of failure, which was either fatigue or sudden failure. Fatigue was indicated by a gradual decrease of the load during cycling. If failure followed fatigue, the curve would drop below 25 kg load. A steep drop in load to zero was due to sudden failure of the sample.

Sudden failures during the first application of tension could be confirmed by a sudden drop in load in the load vs. displacement graph (cycling graph) of the first cycle (fig. 6), and a flattened curve of the last cycle (fig. 7).

Failure type of the control samples was visualized through the load, displacement graph of figure 8, created from data gathered during the uniaxial tensile test.

The location of failure was noted as either adhesive (bond of adhesive to substrate), cohesive (within the adhesive layer), or substrate (in the wood).

4.3.4 Maximum load and displacement at failure

Failures were further examined by comparison of the maximum values of load and displacement at the point

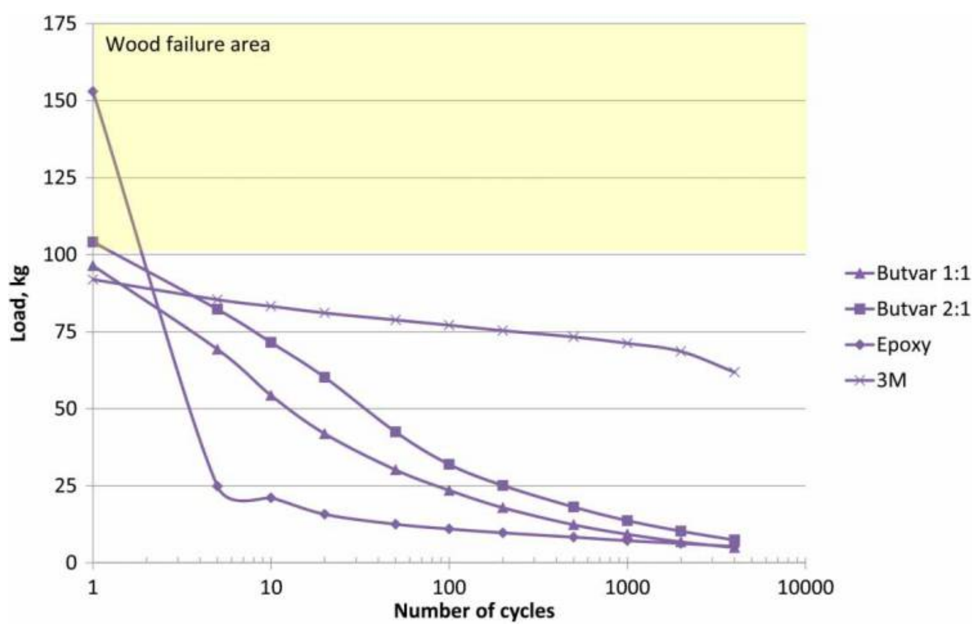


Fig. 4. Graph of the development of average positive load (tension) for unweathered samples for cycles 1–4000, showing fatigue followed by failure (Butvar), sudden failure (Epoxy), and fatigue (3M); plotted samples: samples C7–9 for Butvar 1:1 and Butvar 2:1; samples C7–8 for 3M; samples C8–9 for Epoxy

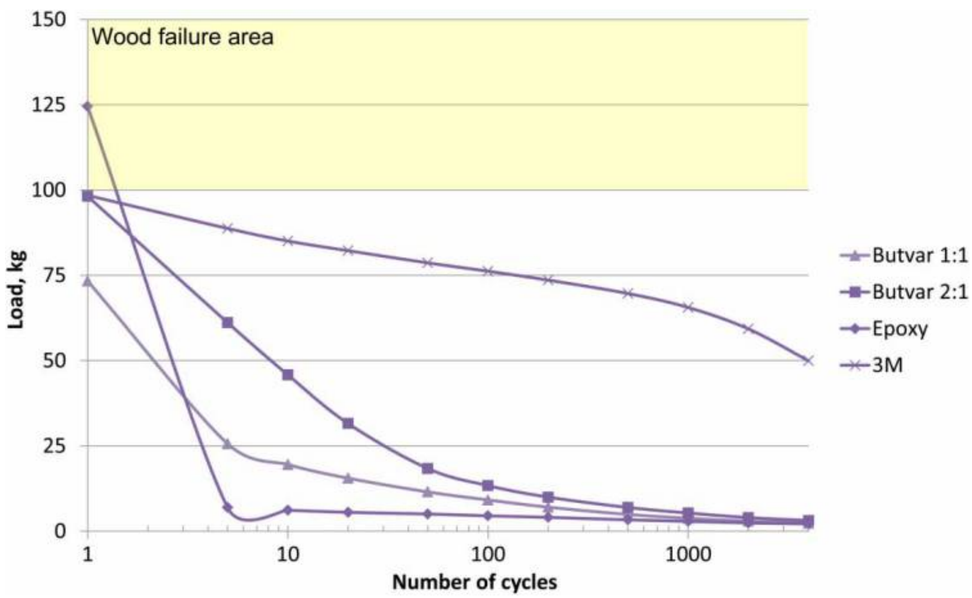


FIG. 5. Graph of the development of average positive load (tension) for weathered samples for cycles 1–4000, showing fatigue followed by failure (Butvar), sudden failure (Epoxy), and fatigue (3M); plotted samples: samples C4–6 for Butvar 1:1, Butvar 2:1, and 3M; samples C5–6 for Epoxy

of failure, and the ratio between them (fig. 9). The uniaxial tensile test provided data for the control samples and surviving cycled 3M samples. For the cycled Butvar and Epoxy samples, data were used from the first cycle of the cycling tests, during which these adhesives either started to fail or had failed completely. Since data were gathered continuously during the first cycle, they could be used in a manner similar to the failure data from the uniaxial tensile test.

The load–displacement ratio plotted in the graph of figure 9 (line with triangular markers) was not a true tensile modulus, since it did not reflect the purely elastic linear section of deformation only. Rather, it was the ratio between the maximum load at the point of failure and its corresponding displacement. As such, the ratios were examined comparatively. A lower ratio meant a more ductile adhesive (i.e., highly able to deform under tensile stress), a higher ratio a

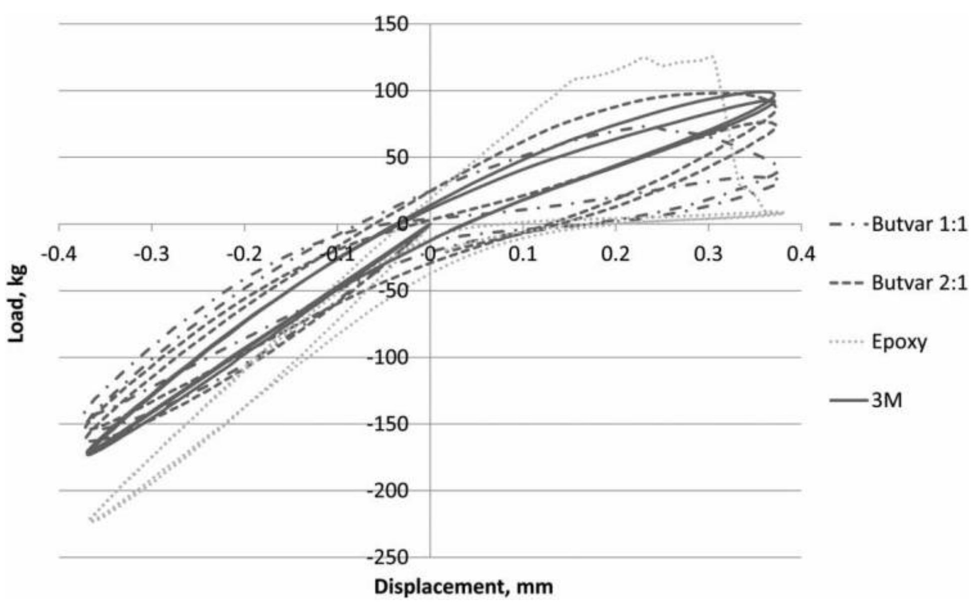


FIG. 6. Average load vs. displacement graph of weathered samples, first cycle. The Epoxy samples show sudden failure, while the Butvar® 1:1 and 2:1 show fatigue followed by failure; plotted samples: C4–6 for Butvar 1:1, Butvar 2:1, 3M, and C5–6 for Epoxy

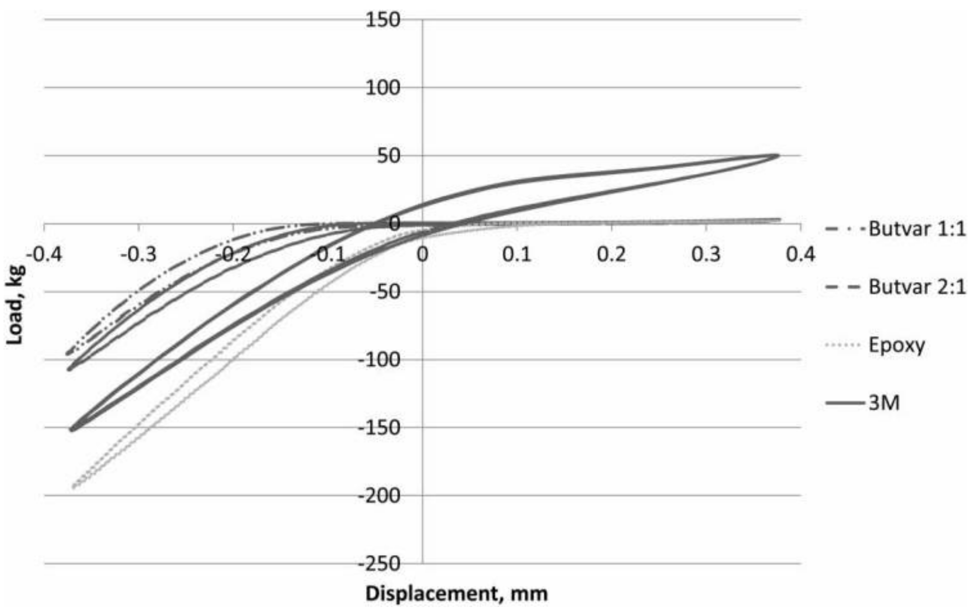


FIG. 7. Average load vs. displacement graph of weathered samples, cycle 4000. All samples have failed, except for the 3M samples; plotted samples: C4–6 for Butvar 1:1, Butvar 2:1, 3M, and C5–6 for Epoxy.

more brittle adhesive. It was important to have a ductile adhesive that could absorb the necessary deformations in the capitals without putting high stress on the wood and risk new failures in the original material.

4.3.5 TYPE OF DEFORMATION

Amorphous polymers, such as the tested adhesives, behave like glass at low temperatures. They only exhibit elastic behavior at small deformations, meaning that they immediately deform upon application of a load and completely regain their original shape after removal of the load. At intermediate temperatures (in the T_g region), they behave like rubbery solids (viscoelastically), having delayed elastic behavior or creep. At high temperatures, they behave like viscous liquids with plastic (permanent) deformation (Mintrop 1997; Callister 2001).

Since testing of the polymers took place well under their T_g, true plastic deformation was unlikely at the relatively small displacements of 0.4–0.6 mm. Elastic or viscoelastic behavior was more likely.

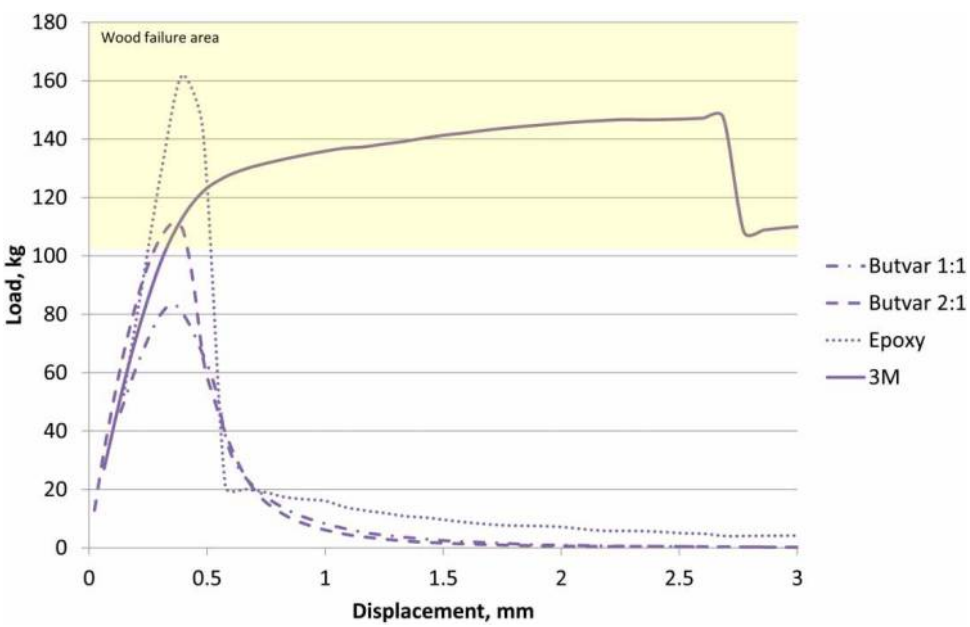
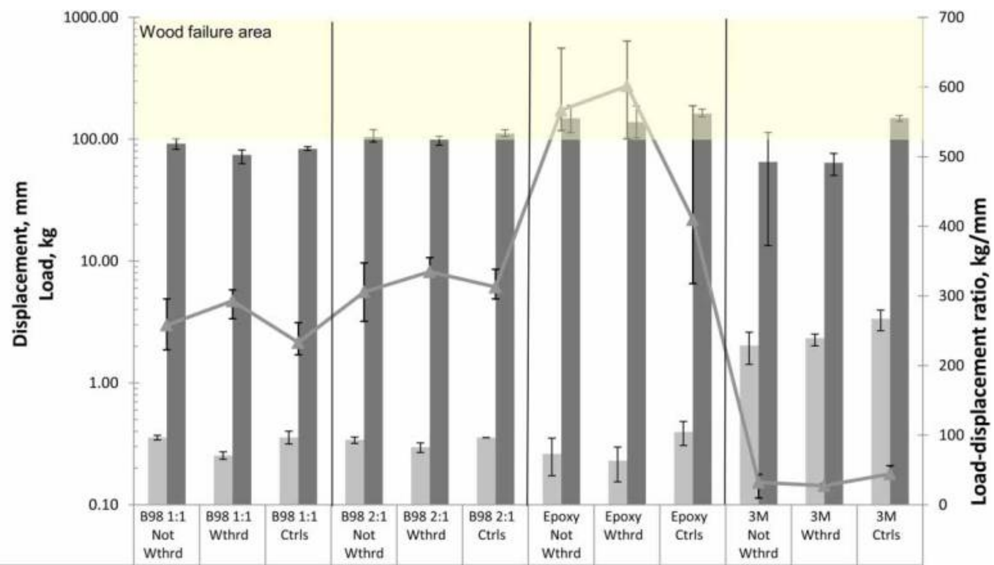


FIG. 8. Load vs. displacement average curves of controls during uniaxial tensile test; plotted samples: C10–12 for Butvar 1:1, Butvar 2:1, and Epoxy; sample C12 for 3M



	B98 1:1 Not Wthrd	B98 1:1 Wthrd	B98 1:1 Ctrls	B98 2:1 Not Wthrd	B98 2:1 Wthrd	B98 2:1 Ctrls	Epoxy Not Wthrd	Epoxy Wthrd	Epoxy Ctrls	3M Not Wthrd	3M Wthrd	3M Ctrls
Displacement, mm	0.36	0.25	0.36	0.34	0.30	0.36	0.26	0.23	0.40	2.02	2.32	3.36
Load, kg	92	74	83	104	99	111	148	138	162	65	64	149
Load-displacement ratio, kg/mm	258	293	233	306	335	313	567	602	410	32	28	44

FIG. 9. Maximum values of displacement and load for all samples at the point of failure, including the load–displacement ratio at these maximum values

The cycling graphs of figures 6 and 7 indicated whether the deformation was viscoelastic or elastic. During cycling, the samples were first compressed (negative displacement) and then stretched (positive displacement). The original dimension was at zero. For a perfectly elastic material, the curves for application and release of load would overlap and go through zero. For materials that underwent viscoelastic or plastic (permanent) deformation, the curve for release of a load would not overlap the curve of applying the load, creating a hysteresis loop. Hysteresis height, in this case, was the difference in load values (*y*-axis) between the curves. The hysteresis height dictated the area inside the loop that was equal to the energy lost during the cycle; if the hysteresis height was very small, the material was mostly elastic, as there was little tension or compression needed to return to the sample's original dimension (Mintrop 1997).

4.3.6 Compression behavior

Finally, the adhesives' compression behavior was examined by comparing the negative loads under compression (negative displacement) (fig. 10).

5. Results

5.1 initial evaluation and elimination

Both the DC738 and DAP adhesives developed fissures in the paint at the interface of adhesive and substrate and over the adhesive between 25 and 50 cycles. In addition, DAP had partial adhesive failure. The Butvar and 3M adhesives developed fissures in the paint at a later point, after 100–250 cycles, while the Epoxy did not have any fissures in the paint that were visible by eye.

The basic tests indicated that all adhesives could unite two pieces of wood and fill a 1.6 mm wide gap. Butvar with cork was a rather dry and crumbly mixture, while the Epoxy, Silicone Rubber, and 3M were sticky pastes. DAP was a moderately thick paste.

The barrier layer of the Epoxy, DC738, 3M, and DAP samples appeared to have no negative effect on strength.

5.2 failure type and location

All Butvar and Epoxy samples failed during the first 4000 cycles, with Butvar 1:1 at 5 cycles (weathered) or around 100 cycles (unweathered), Butvar 2:1 at 30 cycles (weathered) or around 200 cycles (unweathered), and Epoxy (weathered and unweathered) within the first cycle. Only the 3M samples survived both the 4000 cycles at ±0.4 mm and the additional 2000 cycles at ±0.6 mm.

The Butvar samples showed a gradual decrease of the load during the comprehensive cycling tests, ending at less than 15 kg at the end of the first 4000 cycles and even lower values at the end of the total 6000 cycles (figs. 4, 5). The Butvar samples all failed cohesively (table 3). The control samples failed abruptly for both the 1:1 and 2:1 mixtures (fig. 8).

All Epoxy samples failed suddenly during the first application of tension for the cycled samples, before

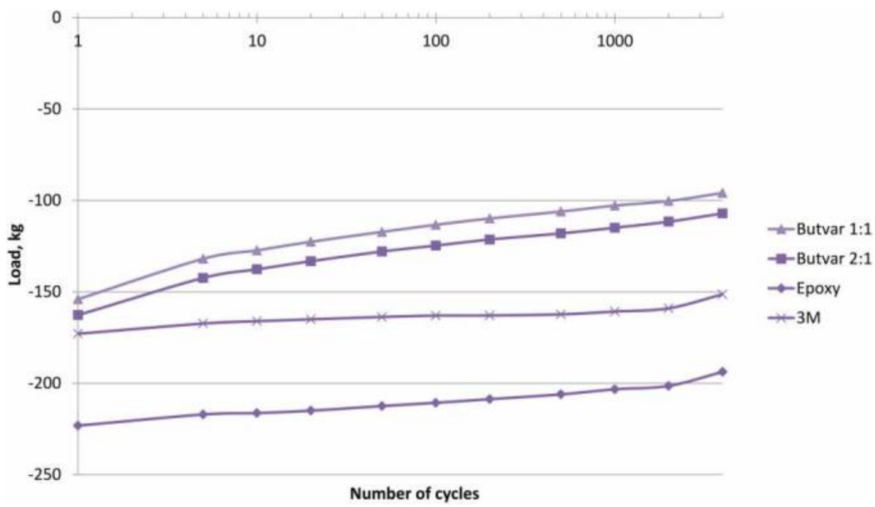


FIG. 10. Graph of the development of average negative load (compression) for weathered samples; plotted samples: samples C4–6 for Butvar 1:1, Butvar 2:1, and 3M; samples C5–6 for Epoxy

reaching the maximum displacement of 0.4 mm (fig. 6). The control samples failed suddenly around 0.4 mm at relatively high loads, (fig. 8). In all cases, failure was in the substrate. The weathered samples failed at lower load values.

The 3M samples also showed a gradual decrease in load, but they ended on average around 62 kg after 4000 cycles and at 51 kg after 6000 cycles for the unweathered samples (fig. 4) and around 50 and 32 kg after 4000 and 6000 cycles for the weathered samples, respectively (fig. 5). When pulled to failure in the uniaxial tensile test, 3M samples (both cycled and control) generally failed adhesively, except for C9 and C12, which failed in the substrate (table 3). The 3M control samples had an elongated average curve that had a sudden drop in load around 2.77 mm due to wood failure in sample C-12 (fig. 8). Curves of the other two 3M control samples gradually sloped down to zero load due to adhesive failure (not depicted).

5.3 MAXIMUM LOAD AND DISPLACEMENT AT FAILURE

In general, weathered samples failed at lower load and displacement values than the unweathered samples at the equivalent number of cycles (fig. 9). However, the weathered 3M samples failed at slightly larger displacements.

The unweathered and control Butvar samples reached 0.35 mm displacement on average, while the weathered samples started to fail around 0.31 mm for the 2:1 mixture and 0.23 mm for the 1:1 mixture (fig. 6). There was no notable difference in maximum displacement between the two different ratios for the unweathered or control samples. At failure, Butvar samples had a maximum load of 74–92 kg for the 1:1 mixture and 99–111 kg for the 2:1 mixture, resulting in a load–displacement ratio between 233 and 293 kg/mm for Butvar 1:1, and between 306 and 335 kg/mm for the 2:1 mixture (fig. 9).

Epoxy samples failed at an average maximum displacement of 0.23 mm for the weathered and 0.26 mm for the unweathered samples. During the uniaxial tensile test, the Epoxy control samples failed on average at 0.40 mm. The corresponding maximum load values were 138, 148, and 162 kg, with a load–displacement ratio between 410 and 602 kg/mm (fig. 9).

In the uniaxial tensile test, the 3M control samples failed at an average of 3.36 mm maximum displacement, while weathered and cycled 3M samples failed at 2.32 mm, and unweathered cycled samples at 2.02 mm on average. Their maximum loads at failure were 64–65 kg for the cycled samples (weathered and unweathered) and 149 kg for the control samples. The load–displacement ratio was 28 kg/mm (weathered and cycled), 38 kg/mm (unweathered and cycled), and 44 kg/mm (controls) (fig. 9). It should be noted that due to the gradual failure of the 3M control samples, the average curve of figure 9 (average load at a given displacement) has different maximum values for load and displacement (147 kg and 2.77 mm) than the averages calculated from the peak loads and corresponding displacements of the individual samples (149 kg and 3.36 mm).

5.4 TYPE OF DEFORMATION

All adhesives showed a hysteresis curve during cycling. The hysteresis height of the first cycle was largest for the Epoxy samples (51 for unweathered

Table 3. Type of Failure after Weathering, Cycling, and Uniaxial Tensile Tests

		After weathering	After 4000 cycles	After 6000 cycles	After uniaxial tensile test
Butvar 1:1	Controls	N/A	N/A	N/A	Cohesive failure (C10–12)
	Unweathered	N/A	Partial cohesive failure* (C7–9)	Cohesive failure (C7–9)	Not tested/already failed
	Weathered	Thin fissures in paint at interface and over adhesive on both sides, some brown leaching (C4,6)	Partial cohesive failure (C4–6)	Cohesive failure (C4–6)	Not tested/already failed
Butvar 2:1	Controls	N/A	N/A	N/A	Cohesive failure (C10–12)
	Unweathered	N/A	Partial cohesive failure (C7–9)	Cohesive failure (C7–9)	Not tested/already failed
	Weathered	No fissures	Partial cohesive failure (C4–6)	Cohesive failure (C4–6)	Not tested/already failed
Epoxy	Controls	N/A	N/A	N/A	Substrate failure (C10–12)
	Unweathered	N/A	Substrate failure (C8 and 9) (C7 improperly mounted)	Substrate failure (C7)	Not tested/already failed
	Weathered	No fissures	Substrate failure (C4) Adhesive failure (C5) Partial substrate failure (C6)	Substrate failure (C6) Substrate/adhesive failure (C5)	Not tested/already failed
DC 738	Unweathered	Eliminated	Eliminated	Eliminated	Eliminated
3M	Controls	N/A	N/A	N/A	Adhesive failure (C10–11) Substrate failure (C12)
	Unweathered	N/A	No failure (C7–8) Substrate failure (C9)	No failure (C7–8)	Adhesive failure (C7–8)
	Weathered	Very thin fissures in paint over adhesive on exposed (C6) or both sides (C5)	No failure (C4–6)	No failure (C4–6)	Adhesive failure (C4–6)
DAP 230	Unweathered	Eliminated	Eliminated	Eliminated	Eliminated

*Cohesive failure: failure within the adhesive layer; adhesive failure: failure at interface of adhesive and wood; substrate failure: failure within wood.

and 54 kg for weathered), closely followed by the Butvar 1:1 samples (34 for unweathered and 45 kg for weathered) and Butvar 2:1 samples (40 for unweathered and 52 kg for weathered), with the 3M samples having the lowest values (17 for unweathered and 21 kg for weathered) (fig. 6). Hysteresis height decreased during cycling, as evidenced by the 3M curve in figure 7.

5.5 COMPRESSION BEHAVIOR

In all cases, the negative load evened out before 1000 cycles and ended at smaller load values. The results for the weathered samples are plotted in figure 10. The Butvar samples showed the largest drop in negative load with Butvar 1:1 starting at −154 kg and ending at −96 kg at the end of 4000 cycles, a drop of 58 kg. Butvar 2:2 started at −163 kg and ended at −107 kg. The Epoxy samples changed from 223 to 194 kg, while the 3M samples went from 173 to 151 kg. Unweathered samples had slightly smaller load values, but similarly shaped curves.

6. DISCUSSION

6.1 INITIAL EVALUATION AND ELIMINATION

The early development of fissures in the paint over the adhesive in the samples of DC 738 and DAP appeared to be due to the adhesives' low modulus of elasticity, and subsequent bulging during compression. Earlier studies did not address the longevity of paint over a bulked silicone rubber, although Barclay and Grattan (1987) did mention various options for inpainting. DAP was claimed to be paintable with oil-based paints (DAP 2003), but had paint adhesion failure comparatively early during the cycling tests. In addition, DAP had partial adhesive failure. Fissures in the paint would expose the adhesive to the weather and lead to premature loss of the paint. Frequent repainting was undesirable, and these adhesives were therefore eliminated from further testing.

Since the barrier layer did not appear to have a negative effect on adhesion, all 3M and Epoxy samples were tested with a barrier layer in subsequent stages. Podany (2001) and Ellis and Heginbotham (2004) showed that barrier layers did not impair strength of a join in their experiments. In fact, barrier layers may have made the bond stronger in Young's tests (Young 2011), which may increase the risk of failure in the original substrate rather than in the adhesive layer or at the interface.

The compatibility of barrier layer and adhesive ensured that the Epoxy and 3M adhesives would not only make a satisfactory bond but also be reversible.

6.2 FAILURE TYPE AND LOCATION

The Butvar and 3M adhesives both showed fatigue in the development of positive load graphs by a gradual decrease of the load during the comprehensive cycling tests. However, for the Butvar samples, fatigue was followed by failure, given the gradual drop in load to less than 15 kg at the end of the first 4000 cycles and even lower values at the end of the total 6000 cycles. For the 3M samples, fatigue was not followed by failure, since the loads remained well over 25 kg.

The weathered Butvar samples failed more rapidly than the unweathered samples with loads below 25 kg around 5–30 cycles for the weathered samples and 100–200 cycles for the unweathered samples, with the 1:1 mixture having the lower values

The cycling graphs (fig. 6) plainly showed how the Epoxy samples generally failed abruptly during the first application of tension, with a sudden drop in load to below 10 kg. Only Epoxy sample C7 (unweathered) did not fail immediately. It appeared not to have been mounted securely during the first cycling period, judging by its erroneous behavior throughout cycling. It readily failed during the second period of cycling at a displacement of 0.2 mm, well below the initial displacement of 0.4 mm. Therefore, it can be assumed that it would have failed in the first 4000 cycles, like the others.

During the uniaxial tensile test, seven out of nine of the 3M samples failed at the interface, which was desirable for conservation purposes; the other two failed in the substrate. All Butvar samples (cycled and controls) failed cohesively, which is also a safe failure location. All Epoxy samples, however, failed in the substrate, which is not acceptable in light of protecting original material.

6.3 MAXIMUM LOAD AND DISPLACEMENT AT FAILURE

3M cycled samples failed at displacements that were higher by a factor of 5–10 (2.02–2.32 mm) relative to Butvar and Epoxy samples (0.23–0.40 mm) and by a factor of 5 higher than what was calculated to be the expected dimensional change in the capitals (0.3–0.4 mm). The 3M control samples failed at displacements that were more than 200% (3.36 mm) of the original adhesive thickness (1.6 mm), and more than eight times the calculated dimensional change, indicating outstanding toughness. Both Butvar and Epoxy samples failed below the desired displacement of 0.4 mm during cycling, with the Epoxy control samples only barely reaching this value.

When compared to the other tested adhesives, the load–displacement ratio of the Butvar samples was medium high (high load at low displacement), indicating that the adhesive mixtures had a medium high tensile modulus when stretched, i.e. having medium ductility or being somewhat brittle.

The Epoxy samples had the highest load–displacement ratio of all adhesives, making it the adhesive with the highest tensile modulus. The ratio was lower in the control samples, due to the higher displacement of these samples before failure.

The low load–displacement ratio indicated that 3M was a very ductile adhesive: it could take large displacements without subsequent high loads. The control samples showed a much higher load and a somewhat higher maximum displacement than the cycled 3M samples (with or without weathering).

Weathering did not clearly differentiate between the adhesives, but did indicate that the paint was sufficiently compatible with Butvar, Epoxy, and 3M adhesives and that the adhesives generally failed at lower loads and displacements.

6.3.1 Maximum load and avoidance of wood failure

The adhesive's maximum load at the point of failure should be lower than that of the substrate to reduce the risk of failure in the original material.

Of all 36 samples (unweathered, weathered, and controls), 11 samples had wood failure. Wood failure occurred at loads between 103 kg (Epoxy sample C5) and 189 kg (Epoxy sample C9), with an average of 150 kg (table 4). The maximum load that any of the adhesive samples should have during cycling between the calculated displacements, should not exceed 103 kg, the smallest load at which wood failure occurred, and preferably be well below this value.

The range of values at which wood failure occurred is indicated as a lightly shaded area in the development of positive load graphs (figs. 4, 5), the load, displacement graph of the controls (fig. 8), and the graph of maximum displacement and load at failure (fig. 9).

The unweathered Butvar samples reached the lower end of wood failure loads during cycling and the uniaxial tensile test with 104–111 kg on average.

All Epoxy samples were in the load range of 103 and 189 kg, and had wood failure.

The 3M samples did not reach unsafe load values during the first cycling between ±0.4 mm, but came in the lower range of wood failure when cycled between ±0.6 mm, but did not fail. Since this cycling was done to explore a more extreme situation that will probably not occur in the situation of the capitals at Mount Pleasant, the chance of wood failure can be considered very low for the 3M samples.

3M C9 (unweathered) was the only sample that failed in the wood during cycling. It seemed to have had a partial failure in the wood between 200 and 500 cycles. During the additional 3500 cycles, the load values were significant enough (around 45 kg) to not have had complete wood failure, but the curves of sample 3M C9 were very irregular in comparison with the other 3M samples. Therefore, it was omitted in the average graphs. It may have had a defect in the wood or the tester may have overrun its settings.

The only sample that had wood failure during the uniaxial tensile test was sample 3M C12. It broke at an extreme displacement of 2.3 mm during the uniaxial tensile test, far outside the expected displacement in the capitals.

Although the tangential orientation has the most hygroscopic dimensional change and is therefore good for calculating the largest possible dimensional change

Table 4. Wood Failure Evaluation

Adhesive	Sample #	Displacement (mm)	Load (kg)	Min./max.	Timing
Epoxy (unweathered)	C7	0.17	114		First cycle (second period)
	C8	0.26	142		First cycle
	C9	0.35	189	Max.	First cycle
	Average of C7–9	0.26	148		First cycle
Epoxy (weathered)	C4	0.24	124		First cycle
	C5	0.15	103	Min.	First cycle
	C6	0.30	188		First cycle
	Average of C4–6	0.23	138		First cycle
Epoxy (controls)	C10	0.31	176		Uniaxial tensile test
	C11	0.40	157		Uniaxial tensile test
	C12	0.48	153		Uniaxial tensile test
	Average of C10–12	0.40	162		Uniaxial tensile test
3M (unweathered)	C9	?	68		Between cycle 200 and 500 (defect?)
3M (controls)	C12	2.26	150		Uniaxial tensile test (extreme displacement)

in the capitals (eq. 1), the weakest orientation of the wood is the radial orientation. Test samples with a radial orientation at the join would likely have lowered the maximum acceptable load values for avoiding wood failure.

6.4 TYPE OF DEFORMATION

None of the adhesives behaved perfectly elastically, indicated by their hysteresis curves during cycling. 3M could be classified as the most elastic adhesive, as it had the lowest hysteresis height, compared to Butvar and Epoxy, which exhibited more viscoelastic behavior.

6.5 COMPRESSION BEHAVIOR

All adhesives became more malleable during cycling, probably because of the (partial) failures during tension.

The Epoxy adhesive was rigid under compression, as shown by the negative load values during compression, which were the largest of all adhesives. The Butvar samples were the most malleable according to their least negative load values.

Weathering appeared to make all adhesives slightly more rigid.

6.6 TESTING SPEED

The testing speed during the comprehensive cycling may have had an inordinate influence on the performance of the adhesives. The Butvar and Epoxy adhesives visually appeared to fare much better in the basic cycling tests, which had parameters analogous to the comprehensive cycling tests except for cycling speed. Cycling speed in the ATS tester (3 cycles/min) was roughly a factor 20 lower than in the MTS tester (60 cycles/min). Lower speed had the same influence as higher temperature would in decreasing the tensile modulus and yield point. In other words, the extra time given by a lower speed allowed the polymers to deform and behave more elastically, rather than break (Callister 2001). This viscoelastic property may explain why the Butvar and Epoxy samples failed during cycling in the faster MTS tester, but not in the slower ATS tester. The uniaxial tensile test speed was also lower than the MTS cycling speed: the cycled samples had a top speed of 14.0 cm/min around displacement zero and 7.9–12.5 cm/min at the point of failure, while the control samples were pulled at a constant speed of 5.1 cm/min.

In Mintrop's 1997 study, he deliberately chose to test adhesives at only 80% of the expected dimensional change for this reason. The tensile tester he used had a speed of 5 mm/min, roughly 60 times slower than the uniaxial tensile test speed on the controls in this study. The Butvar and Epoxy control samples failed between 0.31 and 0.35 mm, which is right around 80% of the calculated dimensional change of 0.4 mm in the situation of the capitals. Considering this percentage and keeping in mind that the deformation speed to be expected in the situation of the capitals would be substantially lower than the above tests, we may find that the Butvar and Epoxy behave better in an outdoor application than suggested by the cycling tests. Future research could focus on low-speed testing of the adhesives.

7. TREATMENT

Treatment of the capitals was completed in 2006. The 3M adhesive was used to adhere the multiple pieces of the capitals over a barrier layer of 35% Butvar in ethanol, in a fashion similar to the samples. The capitals were reinstalled on the building, each attached with three stainless steel screws through existing nail holes. When examined in December 2014, they appeared to have held up well with no failures in the paint, adhesive, or wood substrate.

8. CONCLUSIONS

The cyclical tensile and compression testing experiment led to using 3M 5200 Adhesive/Sealant as a gap-filling, flexible adhesive for reuniting the fragments of four exterior wooden capitals. According to weathering, cycling, and uniaxial tensile tests, the adhesive proved to have good weather resistance, flexibility, gap-filling capability, longevity, workability, and an appropriate failure type (adhesive). Applying it on a barrier layer of Butvar B98 ensured reversibility as well. Conserv Flexible Epoxy Patch 200 failed abruptly in the substrate during the first cycle, while Butvar B98 with cork 1:1 and 2:1 failed cohesively and more slowly, but still at an early stage. Dow Corning 738 RV and DAP Dynaflex 230 developed fissures in the paint prematurely and were eliminated from more extensive cycling. The executed treatment with the 3M adhesive proved to be successful when examined after 8 years of outdoor exposure.

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(Technical Manager, Adhesives and Sealants, R&H), Mary Sieffert (Weather-O-Meter Expert in Elastomeric Roof Coatings, Technical Service, R&H), and Peter Eastman (Senior Scientist Microscopy Group Central, R&H). Weathering of the samples was conducted by Mary Sieffert at R&H Company, while cyclical tensile and compression testing was executed by Don Huang at the DuPont Experimental Station. Preliminary tests were done by John Lopuszanski, Senior Applications Technician, Adhesives and Sealants at R&H Company. Behrooz Salimnejad, then Associate Conservator at the PMA, now the Elaine S. Harrington Senior Conservator of Furniture and Woodwork, adhered the parts of the capitals, based on this research, reinstalled them on the building, and examined them in 2014.

Notes

1 Philadelphia Museum of Art, *Historic Houses*, http://www.philamuseum.org/historichouses (accessed October 13, 2014)
2 According to the BBC Weather Centre, http://www.bbc.com/weather/4560349 (accessed April 10, 2014)
3 According to the BBC Weather Centre, http://www.bbc.com/weather/4560349 (accessed April 10, 2014)

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Sources of Materials

3M Marine Adhesive Sealant 5200
3M Center
Building 223-6S-06
St. Paul, MN 55144-1000

Butvar B98
Conservation Materials Ltd.
1275 Kleppe Lane, #10
PO Box 2884
Sparks, NV 89431

Conserv Flexible Epoxy Patch 200
Housecraft Associates
7 Goodale Rd
Newton, NJ 07860

DAP Dynaflex 230
DAP Inc.
2400 Boston Street
Baltimore, MD 21224

Dow Corning 738 RTV
Dow Corning Corporation
South Saginaw Road
Midland, MI 48686

Granulated cork
Jelinek Cork Group
4500 Wier Industrial Estates
PMB 167
Niagara Falls, NY 14305

Old Village Exterior Primer 1236 white
Old Village Exterior Paint M26727 (Alkyd)
Old Village Paint
400 Stenton Ave
Plymouth Meeting, PA 19462

Phenolic Microballoons
Asia Pacific Microspheres SDN BHD NO 9
Jalan Utas 15/7
40200 Shah Alam, Selangor Darul Ehsan, Malaysia

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