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AN EVALUATION OF POTENTIAL ADHESIVES
FOR MARBLE REPAIR

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WINSTON O. SOBOYEJO, AND NIMA RAHBAR

ABSTRACT

A collaboration among conservators, conservation scientists, and material scientists has yielded interesting results in evaluating adhesives for the reassembly of marble fragments. Understanding the properties of adhesives in this context is essential for their optimal use. Two such properties – interfacial fracture toughness and bond line width – were examined. This project aims to aid conservators in making informed decisions in choosing adhesives by comparing the performance of thermoplastic and thermosetting resins commonly used in marble repair. The interfacial fracture toughness of Brazil-nut specimens is determined using tensile splitting tests. The following eight adhesive systems were used: Paraloid B-72, Paraloid B-48N, a 3:1 mix of Paraloid B-72 and B-48N, Hxtal NYL-1, Epotek 301-2, Akepox 2000, Marmorkitt 1000, and a Paraloid B-72/Epotek 301-2 sandwich.

One hypothesis tested in this study is that thermoplastic resins could be used as structural adhesives for marble repair. The goal of using such adhesives is to provide reversibility while maintaining adequate strength of the joint. Results indicate that from the perspective of interfacial toughness, several thermoplastic systems are viable for marble repair.

Another important property, adhesive bond width, is also determined. For each adhesive, the bond width is compared to values previously published in conservation literature. These data are then used to assess the correlation between bond widths and interfacial toughness of the various adhesives.

1. INTRODUCTION

Adhesives are widely used in repairing marble objects and architectural ornaments. Few scholarly articles (Koob 1986; Down 1996; Podany et al. 2001) in the conservation literature address the difficulties faced by conservators when choosing an adhesive for marble repair. Understanding the properties of adhesives used in these contexts is essential for their optimal use. This paper aims to add to the information on this subject and aid conservators in making more informed decisions when selecting adhesives.

The samples for this study, generally referred to as Brazil-nut specimens, consist of Carrara marble disks created from two semicircular specimens bonded together with one of the eight thermoplastic or thermosetting adhesives chosen for the study. Using these samples, interfacial fracture toughness was determined using tensile splitting tests. Interfacial fracture toughness is defined as the ability of a material containing a crack to resist fracture. In this study, it specifically refers to the resistance of the interface between marble and adhesive to decohesion. Interfacial fracture toughness is generally accepted in the fracture mechanics community as a more accurate, quantitative, and reliable than interfacial strength as it takes into consideration additional parameters including stress state, flaw size, and specimen geometry. (Kuhl and Qu 2000). Fracture mechanics "relates the crack length, the material's inherent resistance to crack growth, and the stress at which the crack propagates. Hence, fracture mechanics can be used to address the prescribed parameters in this work by estimating the interfacial fracture toughness to analyze the interfaces between a range of restoration adhesives and marbles used to restore sculptures and historical objects" (Rahbar et al 2010, 4939). Adhesive bond widths were also determined for each adhesive and are compared to values previously published in conservation literature.

One hypothesis tested in this study is that thermoplastic resins can be used as structural adhesives for marble repair. The goal of using thermoplastic adhesives is to provide reversibility while maintaining adequate strength of the joint. Of equal importance, but beyond the scope of this paper, are the creep and fatigue characteristics of joints created with thermoplastic resins.

2. ADHESIVES USED IN CURRENT STUDY

Four classes of adhesives were used in the current study: acrylics, polyvinyl acetals, epoxies, and polyesters. The majority of the adhesives tested in this study are for indoor use only.

2.1 ACRYLIC RESINS

The use of acrylic resins in conservation has been common since the 1950s, when Lucite 44, a polybutyl methacrylate, was used as a varnish for oil paintings (Horie 1987). Their popularity in the past 50 years has not waned, and it is likely that they will remain in use provided they "fulfil all criteria for present day conservation" (Robson 1992). Acrylic resins used in conservation normally fall into two families–acrylates and methacrylates. The glass transition temperature (T_g) of methacrylates is higher than that of the acrylates, and copolymers with the desired T_g can be made by varying the percentage of each monomer in the final mix (Horie 1987). Two such acrylic copolymers, Paraloid B-72 and Paraloid B-48N manufactured by Rohm & Haas, as well as a 3:1 mix (by volume) of Paraloid B-72/Paraloid B-48N, were tested in this study. Paraloid B-72 is a copolymer of ethyl methacrylate/methyl acrylate with a T_g of 40˚C (Horie 1987). The popularity of this resin in conservation has led to its use as a coating, consolidant, and adhesive. For this study, a 40 wt% solution of Paraloid B-72 was prepared in an acetone/ethanol (10:1 by weight) solvent solution.

Paraloid B-48N is a copolymer of methyl methacrylate and butyl acrylate with a T_g 50˚C (Horie 1987). It is often used as a protective film for canvas paintings and metal. As with the Paraloid B-72, a 40 wt% solution of Paraloid B-48N was prepared in an acetone/ethanol (10:1 by weight) solvent solution.

The 3:1 ratio (by volume) of B-72/B-48N was based on the recommendation of Conservator Donna Strahan at the Metropolitan Museum of Art, who had used it as an adhesive for marble in archaeological contexts. Indeed, the mix should give an adhesive with a higher T_g than using Paraloid B-72 on its own, as Paraloid B-72's relatively low T_g is often of concern to conservators due to possible cold-flow issues.

2.2 EPOXY RESINS

Epoxy resin systems consist of two parts: an epoxide component that reacts with a hardener. Diglycidyl ether of bisphenol A (DGEBA) is normally the epoxide component, while the hardeners are often aliphatic amines and amides. Epoxy resins normally shrink about 5% during hardening. They have been used widely for glass, stone and wood (Horie 1987). Epotek 301-2, Hxtal NYL-1, and Akepox 2000 were used in this study in accordance with manufacturer's instructions.

2.3 POLYESTER RESINS

There are many polyester resin products on the market for stone repair with a wide range of viscosities. Polyesters are also two-part systems, in which an initiator is mixed with a resin pre-polymer containing a reactive monomer, normally styrene. The polymerization first results in

a gel, and then a hard solid. The working time (12–20 minutes at room temperature) can be regulated by adjusting the amount of initiator used. Some amount of shrinkage is involved in the polymerization process that likely continues for months (Hoarie 1987).

The use of polyesters in conservation began shortly after their introduction to the market in the 1940s, and they have been used as consolidants and fillers for wood, as well as adhesives and consolidants for stone. While polyesters cannot be dissolved in organic solvents, they can be removed after swelling (Horie 1987). The polyester resin tested in this study was Marmorkitt 1000.

3. EXPERIMENTAL

3.1 INTRODUCTION

This project involved the use of the adhesives listed in the previous section to adhere samples of Carrara marble, making the so-called Brazil-nut sandwich fracture specimens (figs. 1, 2). The glued samples were then subjected to tensile splitting to determine the interfacial toughness based on a modified standard ASTM D3967-95a (ASTM 1995). Experiments using Brazil-nut specimens for the testing of interfacial toughness were first designed and employed in 1990, and the use of this specific type of sample is essential to the testing method (Wang and Suo 1990). Since then, this type of testing has been used in assessing adhesive performance in microelectronic devices and electronic packages (Kuhl and Qu 2000). No reference was found for using this type of testing for adhesives in conservation. The interfacial fracture toughness is measured over a range of mode mixities, from pure tension to pure shear, by varying the angle between the direction of the applied load, P, and the long axis of the flaw. This angle is referred to as θ. The load at which fracture occurred was recorded, as was the type of failure, i.e. its occurrence in the marble or in the adhesive line.

In addition, the width of each adhesive bond line was measured and compared to previously published standards in conservation literature (Bradley 1984; Podany et al. 2001) for adhesive bond widths. These data were used to determine a possible correlation between bond widths and interfacial toughness.
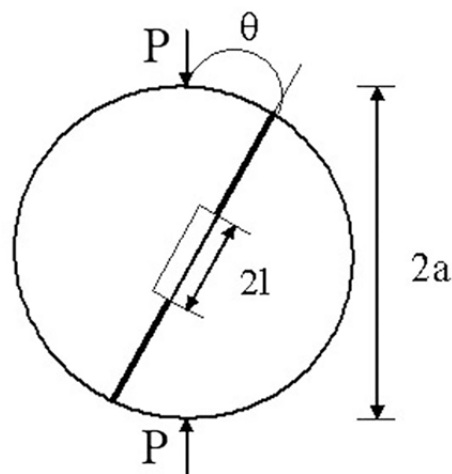

Fig. 1. Brazil-nut specimen with adhesive layer, where P is the applied tensile load and θ is the loading angle. Both measurements are used to calculate the crack tip phase angle, ψ. (Wang and Suo 1990)
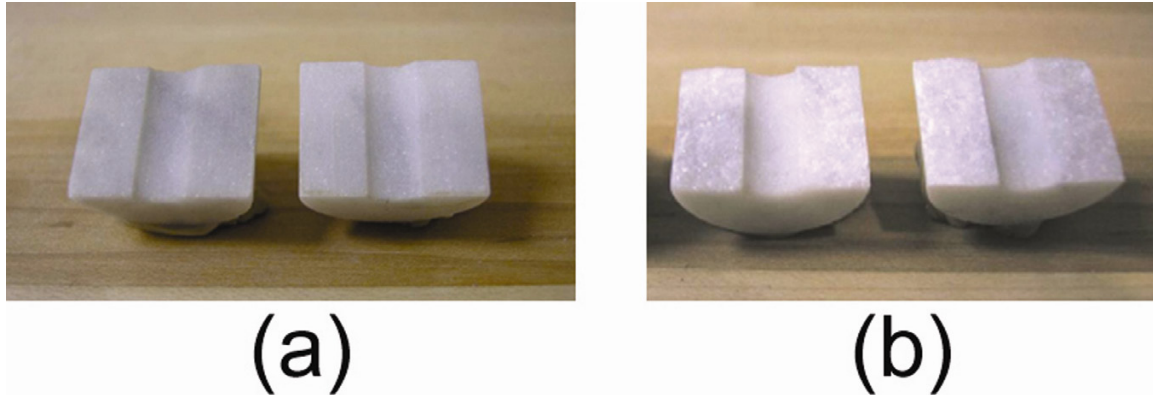
Fig. 2. a) Smooth surface half cylinders cut using abrasive water-jet machining
b) Whole marble cylinder split for testing of adhesive on fractured surfaces (Photographs by authors)

3.2 SAMPLE PREPARATION

For every adhesive, 50 samples were prepared: 25 with smooth bonding surfaces and 25 with fractured bonding surfaces (fig. 2). The final samples were cylinders with a diameter of 24 mm and a height of 8 mm, with an elliptical hole in the center measuring 6.4 mm x 3 mm. This elliptical hole provides each sample with a uniform flaw of defined dimensions, orientation, and location. In this way, subsequent tensile testing of the samples would not measure the inevitable flaws that would be present in a material such as marble, but would in fact measure the performance of the adhesive. In these terms, the flaw acts as an equalizing factor for a heterogeneous material like marble.

An important step in the sample preparation was the use of abrasive water-jet machining (AWJM) technology for the shaping of the initial cylinders that were used to make the final samples. This technology uses water at high pressure (400–1400 MPa), mixed with an abrasive to cut a variety of materials. The desired shape is programmed into a computer that controls the cutting machine. This type of machining is considerably cleaner and more efficient than other cutting processes. Among the many advantages of AWJM are the minimum wetting of the working surface, the ability to start cutting from any point, minimal heat production and minimal burring (Kalpakjian and Schmid 2006).

3.2.1 Fractured Surface Samples

For the fractured surface samples, cores incorporating the elliptical hole were cut from the original Carrara slab using AWJM. The dimensions of these initial cores were 24 mm in diameter and 19 mm in height.

In order to have consistent fractured surfaces for gluing, each core was then subjected to incrementally increasing pressure using an Instron 4201 Table Top Electromechanical Test System operated by the author, and located at the Metropolitan Museum of Art. Enough pressure was applied to make a clean break across the diameter, with the break bisecting the elliptical hole's length. The pressure was applied parallel to the long axis of the elliptical hole, and the presence of the flaw aided with controlling the direction of the fracture. The machine's metal plate surfaces were each fitted with a plastic lid, which decreased the damage and pulverization at the meeting point between stone and metal. Using this system, fractures from one sample to the next were uniform, a requisite for having consistent results in the testing phase (fig. 2b). In

preparation for the adhesive, each surface was lightly cleaned using a synthetic sable10 mm brush in order to rid the face of residual marble dust and debris, and blotted with acetone.

3.2.2 Smooth Surface Samples

In order to make the smooth surface samples, the abrasive water jet technology was used to cut half-cores from the original piece of marble, each with half of the elliptical flaw (fig. 2a); the assembly of two of these half cylinders then resulted in a full cylinder. In preparation for the adhesive layer, each surface was lightly cleaned using a sable/synthetic 10 mm brush in order to rid the face of marble dust and debris, and wiped clean with acetone.

3.3 APPLICATION OF ADHESIVE

After preparing the smooth and fractured surfaces, the adhesive was applied to the samples. For each adhesive, a new synthetic sable 10 mm brush was dipped into the container holding the solution. Excess adhesive was brushed away against the rim of the container. The adhesive was then brushed on the surface of the half cylinder along its length with two passes, covering the surface of the marble. The adhesive-saturated half cylinder was then attached to its respective dry half. Excess adhesive was removed mechanically or with acetone.

For application of the layers comprising the Paraloid B-72/Epotek 301-2 sandwich, a layer of Paraloid B-72 (10 wt% in acetone) solution was applied with a brush to each half cylinder along its length, covering the surface of the marble. This barrier coat was then cured at room temperature for fourteen days. After the curing period, the Epotek 301-2 layer was applied to a half cylinder. The adhesive-saturated half cylinder was then attached to its respective dry half.

3.4 CLAMPING AND CURING OF ADHESIVE

One of the first steps in developing the sample preparation protocol was the identification of the optimal pressure needed for maximum adhesion. This was done empirically: an even layer of a Paraloid B-72 solution (40 wt% in 10:1 acetone and ethanol) was applied to two 1 in. cubes of marble. Using an Instron 4201, pressures of 300, 700, and 1400 kPa were then applied in order to observe the effect on adhesion. At 300 kPa, the adhesive layer was thick, making the two pieces of marble slide off one another and impeding adhesion. At 1400 kPa, almost all the adhesive was squeezed out of the joint, resulting in a dry joint with no adhesion. At 700 kPa, there was enough adhesive, but not a thick layer that would obstruct adhesion; 700 kPa was subsequently identified as the optimal pressure at which to cure the samples.

To maintain even pressure across the sample sets, a clamping system was designed, consisting of two pieces of plywood measuring 41 cm x 2.54 cm x 2 cm, nuts, and bolts (fig. 3). The holes for the bolts were drilled 7 cm from one another, and each clamp system held a total of ten cylinders. Once the glued samples were placed in the clamping system, the nuts and bolts were tightened to approximately 700 kPa using a torque wrench. This pressure value was converted into torque units, Newton meter (N · m), by considering the length of the torque wrench handle, the pitch of the screws used in the system, the clamping area dimensions, and the force necessary per screw to exert 700 kPa (Semat 1958; Hurd 2005). Each bolt was tightened to 6 N · m.

After tightening the six bolts to 6 N · m, the glued cylinders were allowed to cure under pressure for 7–30 days depending on the adhesive (thermosetting versus thermoplastic). The cores were then cut to their final height of 8 mm using a Struers Accutom-50 precision saw. Each

cylinder yielded two samples to be used in interfacial fracture testing. It should be noted that the samples continued curing after they were unclamped until they were subjected to tensile testing. The adhesives cured for an average of three to four months before testing.
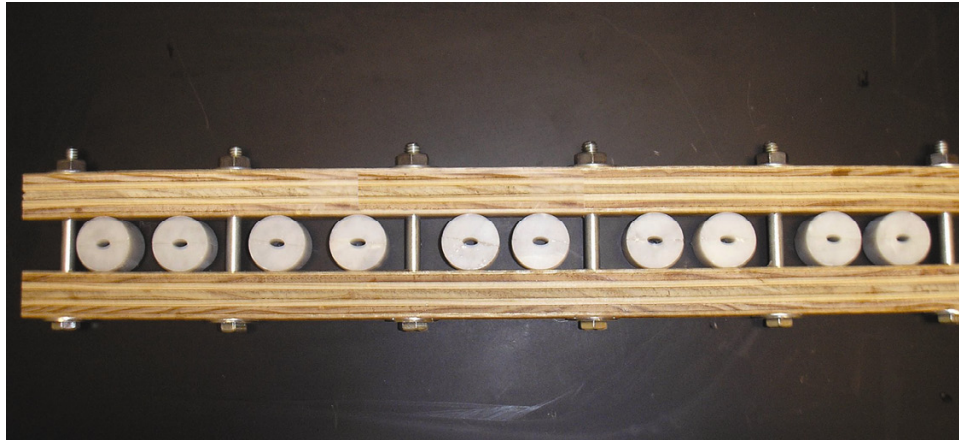

Fig. 3. Clamping system devised for the curing of glued cylinders using plywood, nuts and bolts
(Photograph by authors)

3.5 SPECIMEN TESTING

After sample preparation was complete, tensile tests were conducted by the authors using an Instron 8281 dual-column mechanical analyzer controlled with a proprietary data acquisition software application. The instrument is located in the Department of Mechanical and Aerospace Engineering at Princeton University. The applied tensile load, P, was increased at a rate of 0.005 mm/sec for each loading angle, θ (fig. 1), until complete fracture occurred. The critical load at which fracture occurred for each loading angle was recorded. There were 10 specimens in each sample set; each specimen was placed in the machine at one of 5 different loading angles. The first two specimens were placed into the machine with the elliptical hole at 3° from vertical (the loading angle θ). The remaining specimen pairs in each sample set were tested at 8°, 13°, 18°, and 27°.

After the experimental data were recorded, the loading angle θ and the corresponding critical load were converted to crack-tip phase angle, ψ (see Atkinson et al. 1982). Based on these, the corresponding fracture energy, G, was then established. The interfacial fracture toughness curve was generated by plotting the fracture energy, G, versus the crack-tip phase angle, ψ (fig. 4). These curves were then used to characterise the interfacial fracture toughness of adhesive/marble interfaces (Wang and Suo 1990).

Bond line widths for each specimen type were also recorded using 2.5 mm samples that were produced as a by-product of slicing during sample preparation. These samples were acid-etched and stained using an alizarin-HCl solution (1 g alizarin in 100 mL of 10% HCl solution) in order to have a better contrast between the marble and the adhesive layer. The samples were then examined at a magnification of 175x using a Keyence VHX-500 series digital microscope (fig. 5).

Using the microscope's measuring feature, 50 measurements were taken with an approximate distance of 0.02 mm between each measurement. Using the data, the range of widths, the average width and the average deviation were determined (table 1).
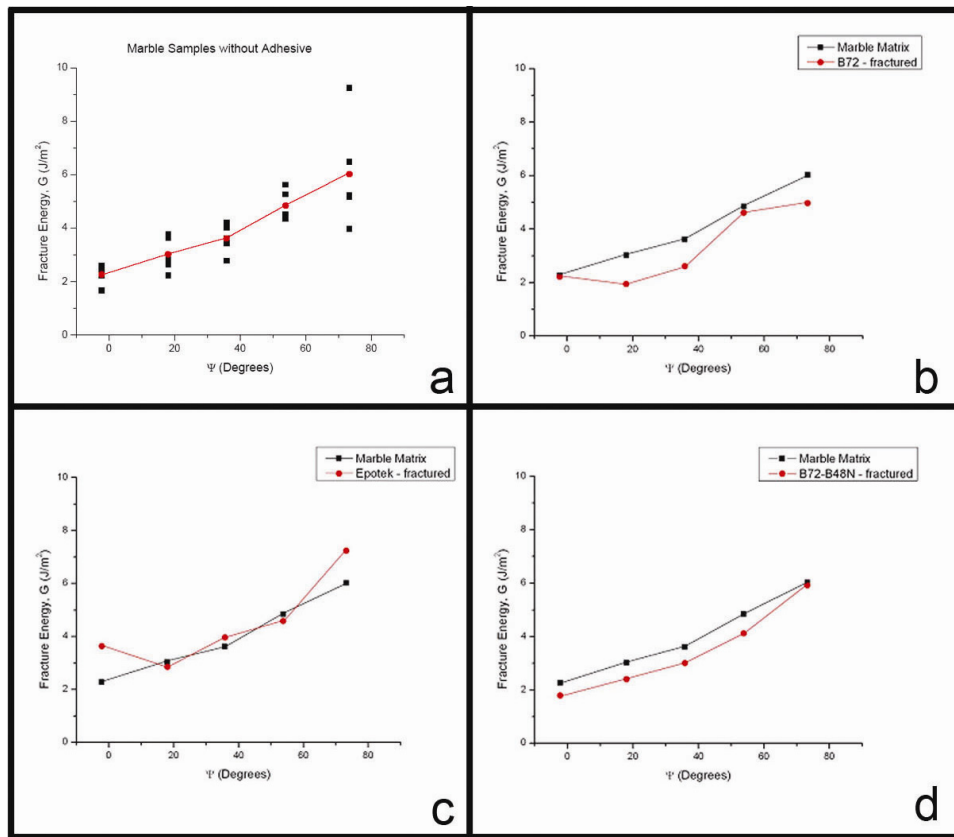
Fig. 4. Interfacial fracture toughness curves: a) marble matrix, b) marble matrix curve overlaid on Paraloid B-72 curve (prepared as described in Section 2.1), c) marble matrix curve with Epotek 301-2 curve, d) marble matrix curve with 3:1 B-72:B-48N blend curve (prepared as described in Section 2.1)
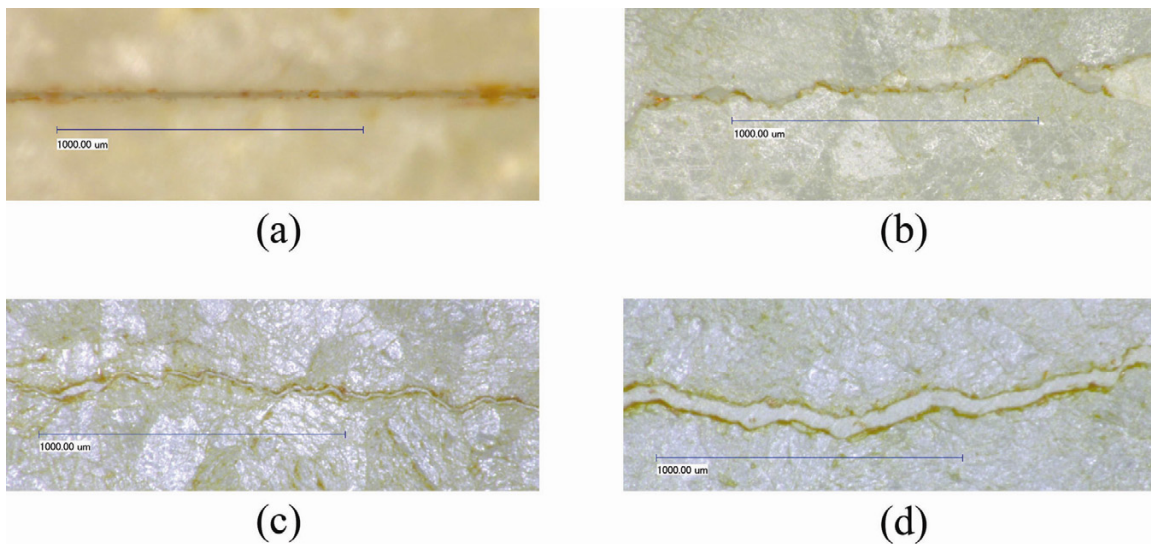

Fig. 5. Bond dimensions at 175x magnification. a) two smooth surfaces without an adhesive, b) two fractured surfaces without an adhesive, c) Paraloid B-48N in a fractured joint, d) Paraloid B-72/Epotek 301-2 sandwich in a fractured joint. (Photographs by authors)

Table 1. Average bond line measurements in μm (average +/– standard deviation)

Adhesive	Smooth	Fractured
Dry join (no adhesive)	20.89 +/– 1.79	22.87 +/– 3.64
40 g B-48N, 54 g acetone, 6 g EtOH	31.83 +/– 5.79	21.94 +/– 4.86
40 g B-72, 54 g acetone, 6 g EtOH	28.07 +/– 4.13	39.20 +/– 4.96
3:1 (by volume) mix B-72/B-48N	23.00 +/– 2.77	41.26 +/– 13.41
10g Mowital B60HH, 40 g EtOH	26.20 +/– 3.49	30.13 +/– 5.34
Hxtal NYL-1	30.81 +/– 3.42	44.29 +/– 10.29
Epotek 301-2	28.89 +/– 4.41	48.44 +/– 5.99
Akemi Akepox 2000	46.16 +/– 5.72	37.91 +/– 6.08
Marmorkitt 1000	38.40 +/– 2.65	56.47 +/– 7.39
B-72/Epotek sandwich	29.01 +/– 3.98	58.06 +/– 5.97

4. RESULTS AND DISCUSSION

4.1 INTERFACIAL TOUGHNESS

Several interfacial fracture toughness curves will be used in this section to illustrate points of discussion. In general, interfacial toughness is higher in the fractured samples compared to the smooth samples. The higher fracture energies in the fractured-surface specimens are not surprising and are probably a result of the overall increased surface area of contact in the fractured samples. Over the range of the phase angles measured, Paraloid B-48N exhibits the lowest interfacial toughness for both the smooth and fractured surfaces. Akepox 2000 showed the highest fracture energy on fractured surfaces while Epotek 301-2 was the toughest on smooth surfaces. For all adhesives on both surfaces, the interfacial fracture toughness increases with increased crack-tip phase angle, ψ. Marble on its own, tested as the control, was a stronger material than most of the adhesives tested (fig. 4a). It is also important to note the large scatter observed in the data. This scatter increased with increasing phase load angle, a phenomenon typical of interfacial fracture toughness data (Thurston and Zehnder 1993).

On the whole, the thermosetting adhesives were higher in fracture toughness than the thermoplastic adhesives. However, the performance range of the two types was similar, with both types falling between 2–10 J/m^2. Interesting results were observed for Epotek 301-2 (fig. 4c) and Hxtal NYL-1, both of which are low-viscosity epoxies. They each exhibited high fracture energies at the lowest phase angle in the fractured specimens, while Hxtal NYL-1 also showed the same trend in the smooth samples. This behavior might be caused by the absorption of the two epoxies into the stone (a halo of epoxy adjacent to the adhesive line was noted when making the samples) and in essence consolidating the stone near the adhesive line. Interesting results were also seen with the Paraloid B-72/Paraloid B-48N mix (fig. 4d). The fracture toughness curve closely followed that of marble matrix.

The fracture patterns were also observed and recorded. The three patterns are illustrated schematically in figure 6. Type 1 failure was in the adhesive line while Type 2 failure was a crack in the marble parallel to the adhesive line. Type 1 was rare and found only in thermoplastic resins. Type 2 failure was observed mainly at low compression angles, notably in Hxtal NYL-1 and Akepox 2000–both thermosetting adhesives.

By far the most prevalent failure type observed was Type 3, with the failure initiating at the upper left part of the sample, continuing through the elliptical hole and terminating at lower right part of the sample. This type of failure was seen in fractured and smooth samples with all types of adhesives and at various compression angles, and indicates that as a group these adhesives are strong enough to join marble.

Type 1: Failure in adhesive.   Type 2: Failure parallel to adhesive line.   Type 3: Fracture starting at upper left and terminating lower right.
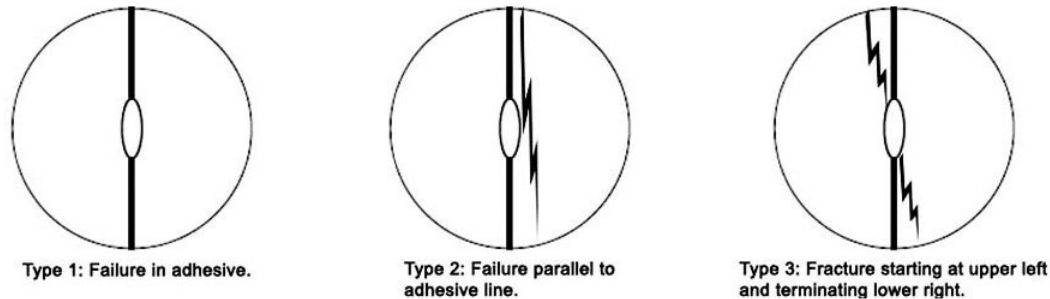
Fig. 6. The three failure types observed in the tested samples

4.2 BOND LINE WIDTH MEASUREMENTS

The average bond thicknesses for the various adhesives are presented in table 1. Bradley (1984, 24) gave the figure of 30 μm as the lower limit of a bond width. Bradley observes that a joint smaller than 30 μm can result in "adhesive starvation," but no mention is made of bond widths thicker than 30 μm. Podany et al. (2001, 24) mentioned the bond thickness of 100–300 μm as being commonly accepted. Bond lines thinner or thicker than this indicated range were said to have too many voids, causing weaknesses in the adhesive layer. However, considering an object with numerous joints being reassembled, a bond width of 200 μm seems thick enough to cause a perceptible displacement at the end of assembly. The smooth surface adhesive bonds studied here measured on average between 26 μm and 46 μm. All are invariably smaller than the range stated by Podany et al.; the largest bond width for the smooth surface, at 46 μm, was seen in the Akemi Akepox 2000. The largest bond width overall was observed in the fractured Paraloid B-72/Epotek 301-2 sandwich, at 58 μm.

Seven of nine adhesive layers were found to be thicker in the fractured samples than in the smooth samples. In order to rule out the nature of the break as a deciding factor in this inconsistency, two dry joints (one smooth and one fractured) were placed in the clamping system under 700 kPa and examined under the microscope in order to measure the width of the breaks before the gluing process. The smooth surfaces were separated by an average gap of 21 μm, while the fractured surfaces were separated by an average gap of 23 μm. The difference of 2 μm between the two types of samples therefore does not explain the discrepancy between the widths of the two surfaces once the adhesive is applied (reaching a maximum average difference of almost 30 μm in the Paraloid B-72/Epotek 301-2 composite system).

Paraloid B-72 (smooth) and Epotek 301-2 (smooth) had very similar bond thicknesses at 28.07 μm and 28.89 μm, respectively. If the two were compared using bond thicknesses as the only variable, one would think their fracture energies would be similar. However, the fracture energy of Epotek 301-2 (average fracture energy 7 J/m^2 at the highest ψ) is higher than that of Paraloid B-72 (average fracture energy 4 J/m^2 at the highest ψ). Evidently, the type of adhesive in this case was more relevant to fracture toughness than to bond thickness. To fully comprehend the relation between bond thickness and fracture toughness, future research should consider the application of different adhesive thicknesses before tensile splitting.

5. CONCLUSION

A primary aim of this project was to establish a step-by-step method for adhesive testing for stone. The ability to make uniform samples and thus have consistent data among all sample sets was the most important aspect of the protocol. Key aspects in having consistent samples involved the use of AWJM, a uniform clamping system for all samples, and the determination of optimal pressure for curing the samples. This sample preparation protocol proved extremely functional when making uniform Carrara marble samples and should be tested with other types of stone to assess its effectiveness.

When considering at the performance of the eight adhesive systems, it can be concluded that all are strong enough for use on Carrara marble, as the majority of the failures were observed in the marble rather than the adhesive line. The similar performance of thermosetting and thermoplastic adhesives was a surprising result, as the initial assumption was that the thermosetting would be much tougher than the thermoplastic adhesives. Therefore, other properties such as reversibility can be used as a determinant when choosing an adhesive.

The fracture patterns observed for the adhesives were all similar, with the majority of the breaks in the marble and not at the adhesive line. The conventional wisdom in conservation is to use an adhesive that is not as strong as the matrix. That convention needs to be re-examined, since even the thermoplastic adhesives could be considered by these standards to be too strong for marble repair.

This paper is a first step in compiling practical data on marble adhesives for use in conservation. Ideally, the same protocol can be used on other types of stone in order to expand knowledge of adhesive performance on a range of matrices. This research involved a limited range of adhesives; more should be tested to have a more complete picture and a deeper understanding of adhesives used in marble repair.

ACKNOWLEDGEMENTS

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SOURCES OF MATERIALS

Abrasive Water Jet Machining
    Hydro-Cutter, Inc.
    1177 Stafford Street
    N. Oxford, MA 01537
    (508) 892-7481
    www.hydro-cutter.com

Paraloid B-72, Paraloid B-48N, Hxtal NYL-1
    Talas
    330 Morgan Avenue
    Brooklyn, NY 11211

Akemi Akepox 2000, Akemi Marmorkitt 1000
    Stone Boss
    26-04 Borough Place
    Woodside, NY 11377

Epo-Tek 301-2
    Epoxy Technology, Inc.
    14 Fortune Drive
    Billerica, MA 01821
    (978) 667-3805

Carrara marble
    ABC Stone
    234 Banker Street
    Brooklyn, NY 11222

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