Interfacial Interactions of Modern Paint Layers

Christina Young

Abstract: *This paper reports on experimental work to measure the adhesion, flexibility, and the onset of cracking of modern paint layers. The objective was to investigate systematically and in greater detail the most significant findings of previous research into the mechanical characterization of composite paint layers with different media on canvas substrates. First, testing of layered alkyds and acrylics indicates that at low strains the mechanical properties of the composites can be predicted by combining the properties of the individual layers in proportion to their thickness. However, at higher strains this is not the case. After the failure of an alkyd layer the stiffness is higher than that what would be predicted assuming the load was taken by the acrylic layer only. Second, acrylic primings reduce the amount of cracking in the alkyd top layers, appearing to retard their normal brittle behavior. Conversely, failure of alkyd primings occurs but the acrylic top layer remains visually intact. Samples consist of commercially available oil, alkyd, and acrylic primings with subsequent paint layers of different pigments in oil, acrylic, and alkyd media. The priming and paint layers were prepared as composites on canvas and as free films. Mandrel and fold tests were performed to measure flexibility; the tensile force needed to detach the upper paint layer from a composite was used to measure adhesion. Crack initiation/propagation were captured using a CCD. The acquired data will result in a better understanding of interfacial interactions and potential failure modes of modern paint-layer combinations.*

Introduction

The combinations of paints that may be chosen for the execution of a painting has expanded dramatically as new paint media have been developed by the coatings industry and then adopted by artists. However, the majority of artists working in a two-dimensional format still use canvas as their main support. Much of the research into the potential problems with modern paints has focused on the use of industrial paints as part of the design layer, whether it has been applied directly onto the canvas or on top of a priming layer. In modern and contemporary works, there has been very little discussion about the role of the canvas or the priming and the possible influences these might have on the longevity of the work and observed mechanical cracks or delamination.

This paper focuses on the properties of artist's alkyd and acrylic primers. It describes how their physical behavior is altered when used in combination with oil, alkyd, and acrylic paints. The research has two aims: first, to investigate layered structures as free films and on canvas, and second, to understand how the mechanical properties of each layer and the adhesion between them affect the behavior of combined layers. This work forms part of the author's continuing research into the applicable methods of measuring interfacial interactions of artists' materials (e.g., peel and shear forces, adhesion, and delamination), with the long-term objectives of predicting problems and finding the most appropriate approaches to mitigate them.

The choice of paints for testing was based on those paints that are most readily available commercially and are among the best selling at the main art suppliers in London for students and professionals. The primers tested were Spectrum Thixotropic Alkyd, Winsor & Newton Alkyd, Golden Acrylic Gesso, and Roberson Acrylic (table 1). The top paint layers consisted of Michael Harding Artists Oils, Liquitex Acrylics, and Winsor & Newton Griffin Alkyds (table 2).

Table 1 Primer samples tested.

Primer Brand and Name	Pigment	Binder[a]	Extender[a]	Manufacturer's Recommended Use
Golden Gesso Primer	TiO_2	p(BA/MMA) acrylic	Silica	Priming all surfaces for oil and acrylic paint
Winsor & Newton Oil Painting Primer	TiO_2	Alkyd	Aluminum silicate	Priming all surfaces for oil and alkyd
Spectrum Thixotropic Alkyd	TiO_2	Alkyd	None detected	Priming all surfaces for oil and alkyd
Roberson Oil Primer	TiO_2	Modified oil alkyd	Aluminum silicate	Priming canvas and wood for oil
Roberson Acrylic	TiO_2	p(BA/MMA) acrylic	Calcium carbonate	Priming all surfaces for oil, acrylic, and tempera

[a] Identified by FTIR

Table 2 Paint samples tested.

Artist Paint	Stated Pigment	Media
Liquitex High Viscosity	Cadmium selenosulfide (red)	Acrylic emulsion
Liquitex High Viscosity	Copper phthalocyanine (blue)	Acrylic emulsion
Golden Heavy Body	Cadmium red	Acrylic emulsion
Golden Heavy Body	Phthalocyanine blue	Acrylic emulsion
Winsor & Newton Finity	Cadmium red	Acrylic emulsion
Winsor & Newton Finity	Phthalocyanine blue	Acrylic emulsion
Winsor & Newton Griffin	Cadmium sulfoselenide (red)	Long-oil modified alkyd (extender dolomite[a])
Winsor & Newton Griffin	Copper phthalocyanine (blue)	Long-oil modified alkyd (extender dolomite[a])
Michael Harding Artists	Cadmium sulfoselenide (red)	Acid-refined linseed oil
Michael Harding Artists	Chlorinated copper (blue)	Acid-refined linseed oil

[a] Identified by FTIR.

Interestingly, those primers that are recommended for use with oil and alkyd paint layers on a variety of supports—canvas, fiberboard, wood, and metal—are alkyd based. Alkyd artists' paints formulated for the upper paints layers are often used by artists because of their short drying times, and sometimes as a method to block-in the design on top of the primer before returning to oil paint for the final design layers. Both alkyd and oil media are known to become brittle with age, and hence if they are used together on a canvas support, which can undergo large deformations, there is a substantial possibility of cracking in both preparation and design layers.

Sample Preparation

The oil, alkyd, and acrylic primings were prepared with subsequent paint layers of phthalocyanine blue and cadmium red in oil, acrylic, and alkyd media. The priming and paint layers were prepared as composites on canvas and as free films.

Free films were cast onto polyester sheets, and composites were prepared on stretched 12 ounce cotton duck using a paint film applicator. The priming and paint layers were applied at thicknesses of 100 and 200 microns, respectively. The samples were left to dry for six days in ambient conditions, after which free films were produced by peeling the paint film away from the polyester sheet and the composites on canvas were removed from their stretchers. Both were cut into 25 × 150 mm strips.

In addition, a previous set of samples had been prepared on stretched canvas, with the layers applied by brush in accordance with the manufacturer's instructions. The crack initiation results only from these earlier samples are reported in this paper. The full results from that work are reported elsewhere (Gregg 2002; Young et al. 2004).

Investigating the mechanical properties of paints necessitates an artificial aging program to evaluate their long-term properties. This is especially true where curing or aging leads to cross-linking or increased crystallinity of the molecular structure. These changes are likely to cause important changes to the macro-mechanical properties of the materials. For the films on canvas, half of the samples were aged in the dark as controls, while the other half underwent thermal and light aging (60° C for 91 days, of which 30 days included exposure to 14,000 lux). For the free films, only thermal aging was undertaken. Three sets were aged at 60° C and 55% relative humidity (RH). One set was the control and the other two were aged for 50 and 122 days, respectively. All samples were preconditioned for 72 hours at 55% RH before testing.

The data for the 122-day thermal aging is that considered in this paper. This aging regime was chosen because it enabled a direct comparison with data from research into the mechanical properties of traditional nineteenth-century oil grounds (Carr et al. 2003).

Tests

The interfacial interaction between the layers of a painting depends upon stiffness, strength, flexibility, and adhesion. In relation to canvas paintings, these terms can be understood as follows: *Stiffness* relates to how taut a painting is when tensioned. *Ultimate tensile strength* (UTS) is the resistance to fracture under tension. Both properties are measured by tensile testing. *Flexibility* is the ability to bend without fracture; it was tested using a mandrel test (Bend Test BS 3900) for paint and varnishes, and for fabric by determining the bending length and flexural rigidity using test BS 3356:1990. *Adhesion* is the resistance to delamination between layers; it was tested by Cross-Hatch BS EN 3900-E6/Pull-Off BS EN 24624.[1]

Tensile testing was performed on an Instron 4301 at a speed of 5 mm/minute at 20° C ± 2° C and 55% RH ± 2%. Sample size was the same for free films and composites. Films were tested to 20% strain or to their UTS, whichever occurred first. Composites were tested to UTS. For the canvas composite samples, the strain at which cracks first appeared was recorded as the crack initiation (CI), based on careful observations during the testing (fig. 1).

The mandrel tests used a rig consisting of metal rods (mandrels) with diameters from 1 to 23 mm. Each sample was bent around the rods to an angle of 180 degrees for 2 seconds with the primer layer inside. Samples were deemed to have failed at the largest diameter at which cracking occurred. If a sample had not cracked on the mandrels, it was subject to a 180-degree fold test. The test procedure was to fold the sample back on itself in one direction and then in the other direction repeatedly for up to 100 folds or until failure, whichever occurred first. Because the maximum tensile and compressive strains this test exerts are on the free surfaces, cracking was assessed by visual inspection for a surface crack in either the primer or pigmented layer. From the previous tests it had been found that larger diameter rods resulted in very few failures and hence there was no discrimination between samples. Further mandrel tests and 180-degree fold tests were repeated on the samples after an additional two years of natural aging with rod diameters of 1–6 mm.

Adhesion tests were performed using the Cross-Hatch method. The two-layer free films were adhered to a flat steel plate with Permabond E32A8B and left to cure for 24 hours. The upper paint layer was then cut into small squares using a tool with serrated teeth equally spaced 2 mm apart. This method reduces the lateral bonding within the layer and promotes delamination between the layers. The degree of delamination of the layer is classified by a visual assessment of the degree of paint loss from each square compared to a standard. The Pull-Off test was used for comparison of two sample types. The test consisted of a free film sample with primer and paint layers adhered with Permabond E32A8B onto two stubs, which were then coupled to a tensile tester. Tensile testing was performed on an Instron 4301 at a speed of 5 mm/minute at 20° C ± 2° C and 55% RH ± 2%. The test measured the tensile force required to separate the two paint layers.

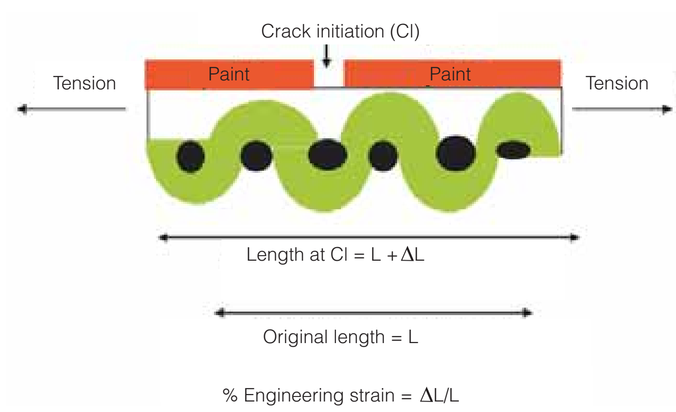


FIGURE 1 Crack initiation.

Results

This paper concentrates on the analysis of the results from the free films with a red paint layer and compares it to data from previous testing of the same layer structure on cotton duck canvas, the full results of which are published elsewhere (Young et al. 2002). Stress and strain were calculated from the load extension data of each sample, using the initial gauge length and the measured thickness.[2] For most external forces exerted on a stretched painting, typically the strain experienced will be on the order of 0.1–10%. In this strain range, calculation of engineering strain is appropriate (see fig. 1). The results are plotted in figures 2–6, grouped by the primer. As would be expected from viscoelastic materials, there is no clearly defined linear region from which to calculate the elastic modulus.[3] Therefore, the secant modulus at 0.5% and 5.0% strain,[4] has been calculated from the stress-strain plots (summarized in table 3). The percentage of strain at which crack initiation occurs for the composite samples and the mandrel and fold test results for the free films are also summarized in table 3. (For clarity, all paint film layers that are not primer are described below by their media, rather than by manufacturer, as there is only one in each case.)

Acrylic Gesso–Acrylic Paint Samples

Comparison of the secant modulus at 5% strain shows that Golden gesso primer (fig. 2) and Roberson acrylic primer (fig. 3), with an acrylic paint layer, had the lowest stiffness of all the paint combinations, in the range 0.02–0.04 MPa. After aging, there appears to be a very slight increase in stiffness, but this change is within the error of the experimental measurement. For all these samples, cracks were not observed after mandrel tests or after 100 folds of a fold test. Thus, it is considered that a significant change in flexibility did not occur after aging. This result is consistent with the composite sample results, for the same paint combinations, where crack initiation did not occur before failure of the complete composite. It is worth noting that the cotton duck canvas without primer or paint layers, typically exhibits a strain of approximately 25% before failure. This sets an upper limit on the percentage strain for crack initiation.

Acrylic Gesso–Oil Paint Samples

The results for the unaged Golden gesso with an oil paint layer are similar, with a secant modulus at 5% strain of 0.03 MPa, no failure after 100 folds, and no crack initiation before composite

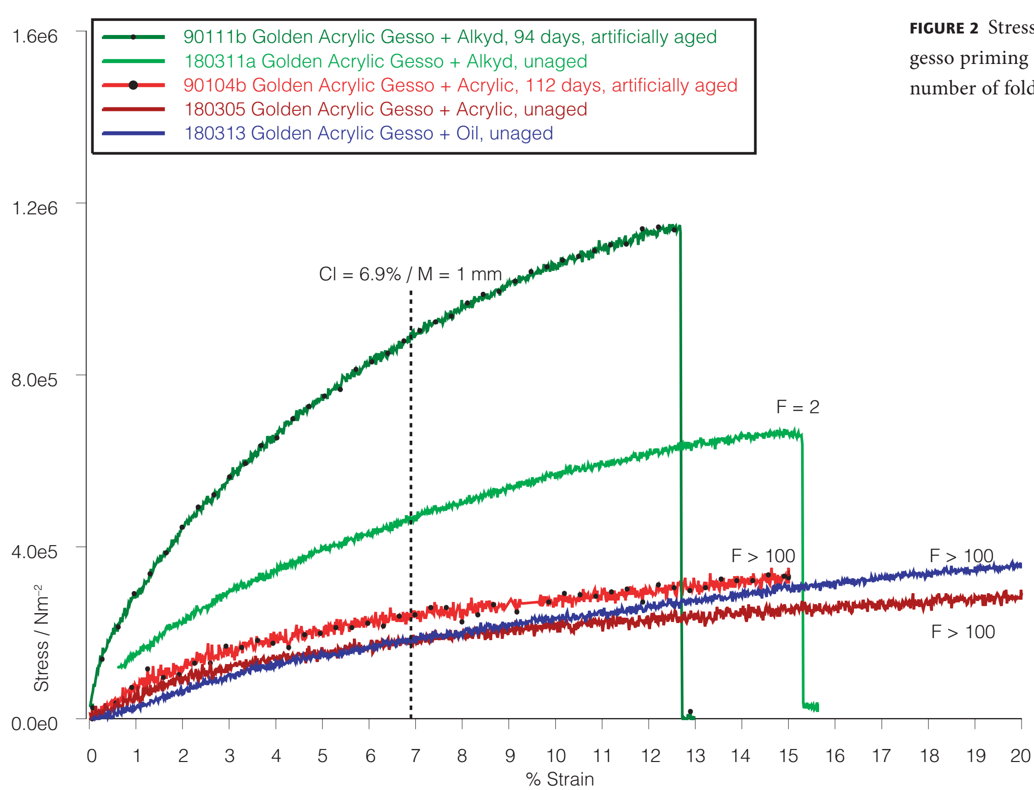


FIGURE 2 Stress-strain curves for Golden gesso priming plus one paint layer. F = number of folds.

Table 3 Testing results.

Priming Layer	Paint Layer	Thermal Aging (days)	Secant Modulus (MPa) at 0.5% strain (error ± 0.03 MPa)	Secant Modulus (MPa) at 5.0% strain (error ± 0.02 MPa)	Crack Initiation Strain (error ± 1%)	Mandrel Failure Diameter (mm)	Folds to Failure
Golden Acrylic Gesso	Winsor & Newton Griffin Alkyd	94	0.36	0.15	6.9%	1	n/a
Golden Acrylic Gesso	Winsor & Newton Griffin Alkyd	None	0.21	0.08	None	No failure	2
Golden Acrylic Gesso	Liquitex Acrylic	112	0.11	0.04	None	No failure	>100
Golden Acrylic Gesso	Liquitex Acrylic	None	0.06	0.03	None	No failure	>100
Golden Acrylic Gesso	Michael Harding Artists Oil	112; remains attached to substrate	Not tested	Not tested	Not tested	Not tested	Not tested
Golden Acrylic Gesso	Michael Harding Artists Oil	None	Insufficient data above baseline	0.03	None	No failure	>100
Roberson Acrylic Primer	Winsor & Newton Griffin Alkyd	94	0.08	0.11	1.4%	No failure	1
Roberson Acrylic Primer	Winsor & Newton Griffin Alkyd	None	0.08	.09	8.0%	No failure	1
Roberson Acrylic Primer	Liquitex Acrylic	112	Insufficient data above baseline	0.04	None	No failure	>100
Roberson Acrylic Primer	Liquitex Acrylic	None	Insufficient data above baseline	0.02	None	No failure	>100
Winsor & Newton Alkyd Primer	Winsor & Newton Griffin Alkyd	64	1.06	Failed at 4% strain	1.8%	1	n/a
Winsor & Newton Alkyd Primer	Winsor & Newton Griffin Alkyd	None	0.24	0.17	1.9%	4	n/a
Winsor & Newton Alkyd Primer	Liquitex Acrylic	112	Insufficient data above baseline	0.14	1.4%	Not tested	n/a
Winsor & Newton Alkyd Primer	Liquitex Acrylic	None	Insufficient data above baseline	0.05	19.0%	No failure	3
Spectrum Alkyd Primer	Winsor & Newton Griffin Alkyd	94	0.07	0.18	3.3%	Not tested	n/k
Spectrum Alkyd Primer	Winsor & Newton Griffin Alkyd	None	0.05	0.01	8.0%	No failure	1
Spectrum Alkyd Primer	Liquitex Acrylic	112	0.17	0.08	3.3%	4	n/a
Spectrum Alkyd Primer	Liquitex Acrylic	None	0.05	0.05	None	No failure	2
Spectrum Alkyd Primer	Michael Harding Artists Oil	82	Insufficient data above baseline	0.4	9.4%	1.5	n//a
Spectrum Alkyd Primer	Michael Harding Artists Oil	None	Insufficient data above baseline	0.1	11.6%	1.5	n/a

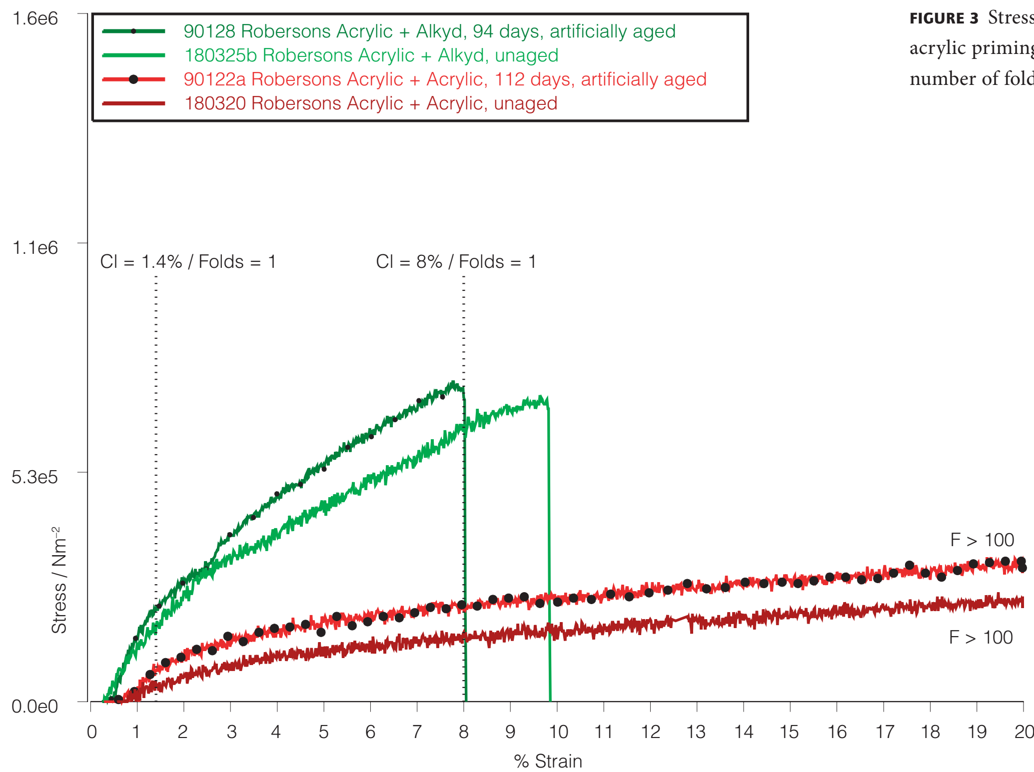


FIGURE 3 Stress-strain curves for Roberson acrylic priming plus one paint layer. F = number of folds.

failure. This would be expected because at the time of testing (twenty-one days and two years), the oil paint film can still be considered to be "young," and therefore not fully cross-linked, and when young oil paint's stress-strain behavior is similar to that of acrylic films. Unfortunately, the artificially aged acrylic primer–oil films could not be detached from the polyester substrate (for reasons yet to be investigated), and therefore, no direct comparison could be made with aged acrylic primers with acrylic films. However, from the previous testing of the oil paint film, one would have expected the two-layer samples to have increased in stiffness and reduced in flexibility (Carr et al. 2003).

Acrylic Gesso–Alkyd Paint Samples

For the samples of alkyd paint layers on Golden gesso, there was a doubling in stiffness, to 0.08 MPa, when compared to the acrylic, and a further increase after aging to 0.15 MPa. At 0.5% strain, a similar trend can be seen (fig. 2). Strain to failure of the aged sample was at 12.7% strain. Crack initiation at 6.9% strain occurred for the equivalent canvas composite sample. Strain to failure of the unaged sample was at 15.4% strain. Thus, although the acrylic primer can withstand over 25% strain without failure, the addition of the alkyd layer causes premature failure. The unaged alkyd sample failed after two folds of a fold test; the aged sample failed at a diameter of 1 mm on the mandrel tester. Thus, as expected, significant increase in stiffness and reduction in flexibility had occurred with the addition of an alkyd layer.

For the Roberson acrylic primer (fig. 3), the addition of the alkyd paint did not cause a change in stiffness after aging (0.08 MPa at 0.5% strain). At 5% strain an increase in stiffness, to 0.09 MPa, did occur when compared to the acrylic (0.02 MPa), and a small increase occurred after aging (0.11 MPa). But it can be seen from the curve for the aged acrylic-alkyd sample in figure 3 that some failure in the sample is occurring at approximately 2.5% strain, where a dip in the curve occurs, and thus the stiffness at 5% corresponds to a sample with a cracked topcoat. This correlates with the observed crack initiation of the canvas composite at 1.4% strain. Although no obvious dip occurs for the unaged sample, the strain to failure is 10%, which is close to the observed crack initiation value for the composite at 8% strain. The alkyd layer has caused premature failure in the acrylic layer, which has reduced the strain to failure. Interestingly, this alkyd paint layer has had a greater influence on the Roberson primer than on the Golden gesso primer, which could be the result of greater interfacial interaction and therefore better adhesion between the paint and the Roberson primer. Both unaged and aged samples failed after one fold of the fold test. Overall, the Roberson acrylic primer–alkyd combination produces a less extensible paint film than the Golden gesso equivalent. This could be caused by higher levels of calcium carbonate extender, higher pigment volume concentration, and greater interfacial interaction between the two layers.

Alkyd Primer–Alkyd/Acrylic/Oil Samples

One can see from comparison of the curves for Winsor & Newton alkyd primer (fig. 4) and Spectrum alkyd primer (fig. 5), both of which had a Winsor & Newton alkyd paint layer on top, that some failure in the samples has occurred at approximately 1–3% strain, where dips or inflections in the gradient occur. Thus, it is very hard to discern a valid place to compare the secant modulus. Nevertheless, if one assumes failure has not occurred before 1%, then it can be seen with reference to figures 4–5 and table 3 that Winsor & Newton alkyd primer with a Winsor & Newton alkyd paint layer results in the stiffest combination, at 0.5% strain with moduli of 0.24 MPa unaged and 1.06 MPa aged. Surprisingly, the Spectrum alkyd primer has a stiffness (0.05 MPa) close to that of the acrylic primers when unaged. This could be explained by the fact that no extender was detected in the primer by Fourier transform infrared (FTIR) analysis.[5] Once the primer is aged, the stiffness increases to 0.07 MPa, which is not significant, but the reduced strain to failure and observed cracking implies this value is inaccurate. The observed crack initiation for the Winsor & Newton alkyd-alkyd and Winsor & Newton alkyd-acrylic canvas composites are at 1.4% and 1.8%, respectively, which correlates very well with the observed inflections of the free film curves at 1.4% and 1.6% respectively (fig. 4). The aged free films failed after mandrel tests at a diameter of 1 mm for Winsor & Newton alkyd-alkyd and 4 mm for Winsor & Newton alkyd-acrylic and Spectrum alkyd-acrylic. Unaged equivalents all failed after one fold of the fold test. The observed crack initiation for both the Spectrum alkyd-alkyd and Spectrum alkyd-acrylic canvas composites is at 3.3%, this correlates with the observed inflections of the free film curves at 0.8% and 3.2% respectively (fig. 5).

The curves shown in figure 6 compare samples with oil and acrylic layers on top of Spectrum alkyd primer. As before, the stress-strain curve for the unaged oil is similar to that of the acrylic film. However, for the aged sample, the stiffness at 5% strain is 0.1 MPa, which is significantly different than for

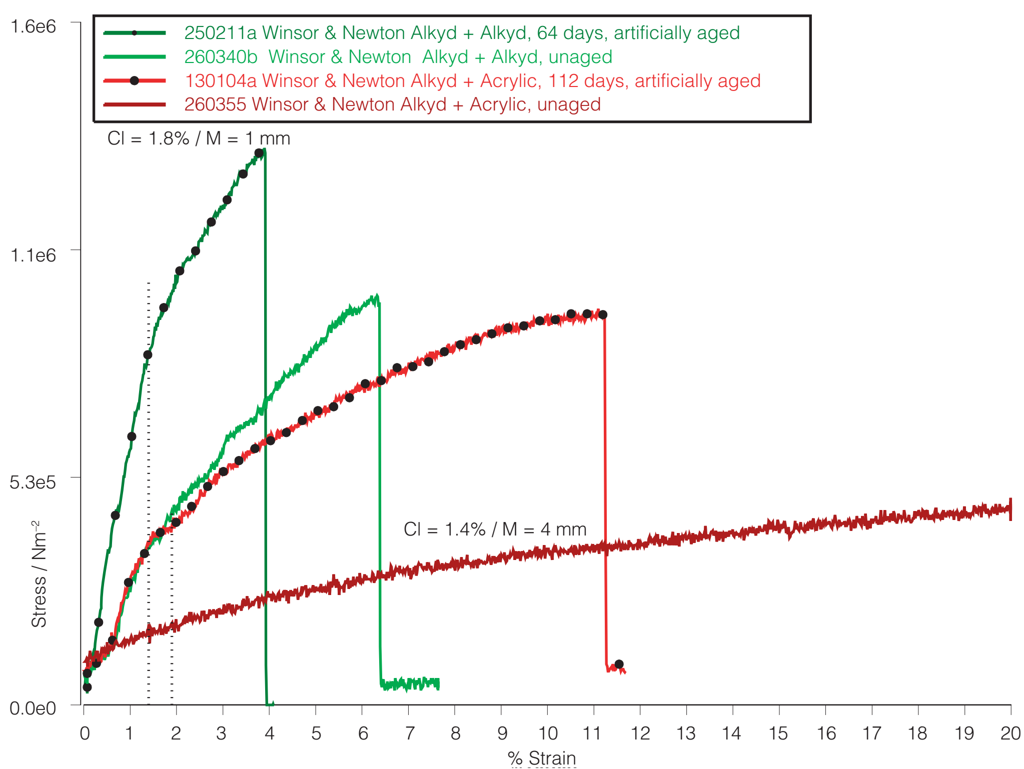


FIGURE 4 Stress-strain curves for Winsor & Newton alkyd priming plus one paint layer. F = number of folds.

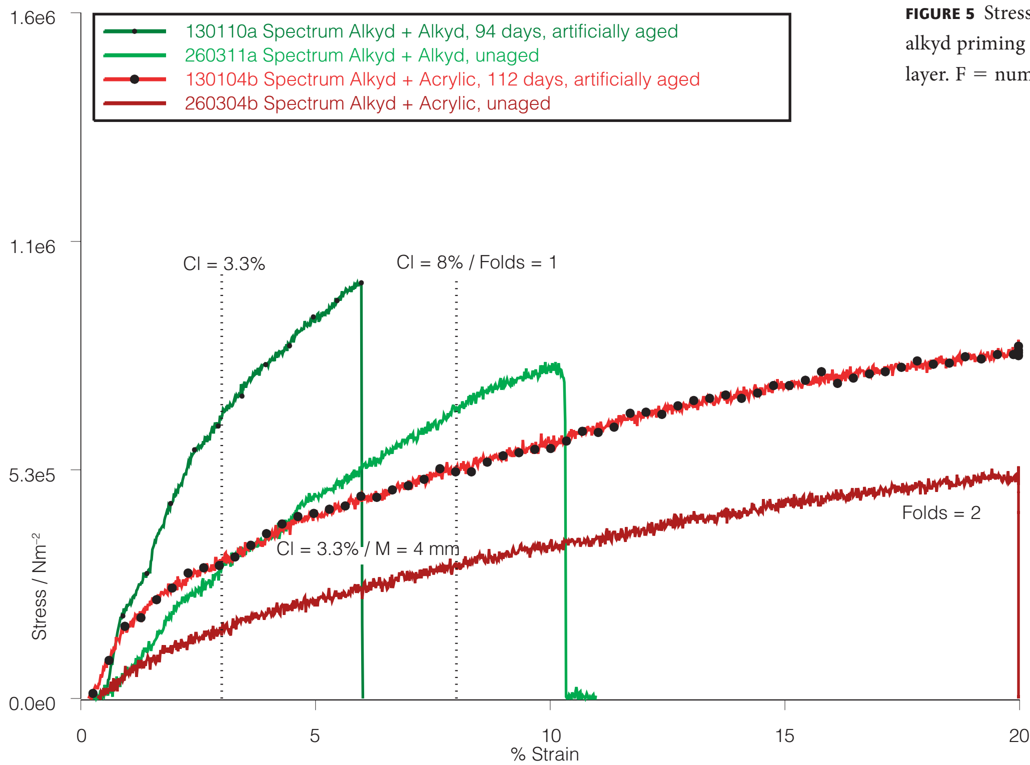


FIGURE 5 Stress-strain curves for Spectrum alkyd priming plus alkyd or acrylic paint layer. F = number of folds.

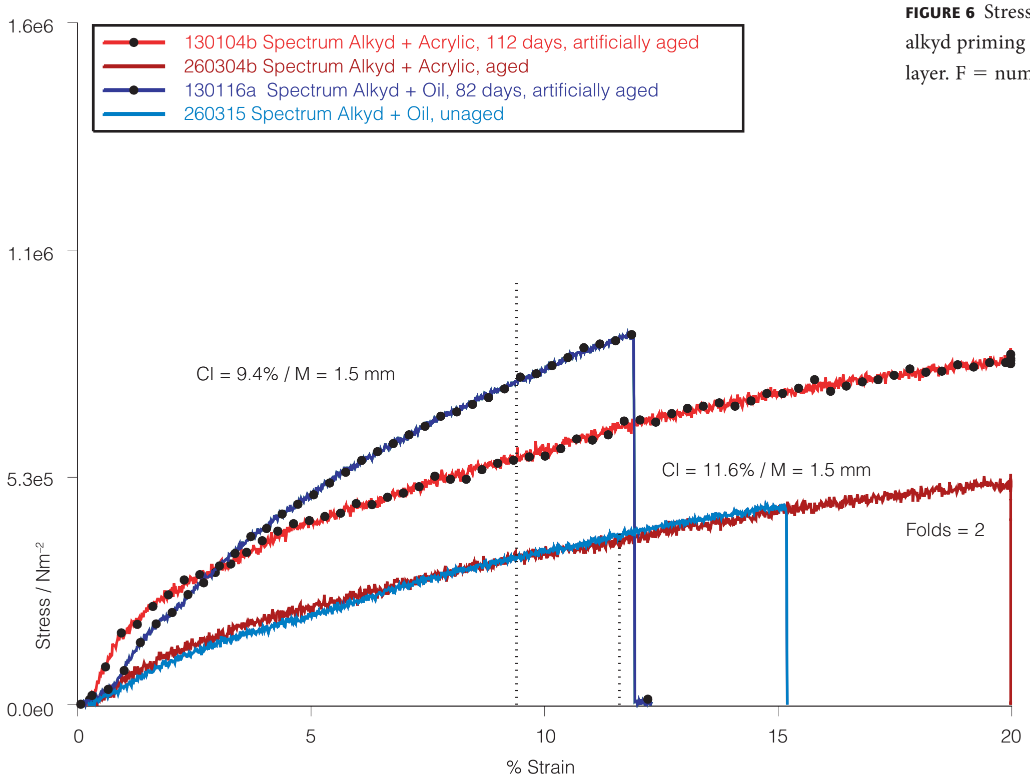
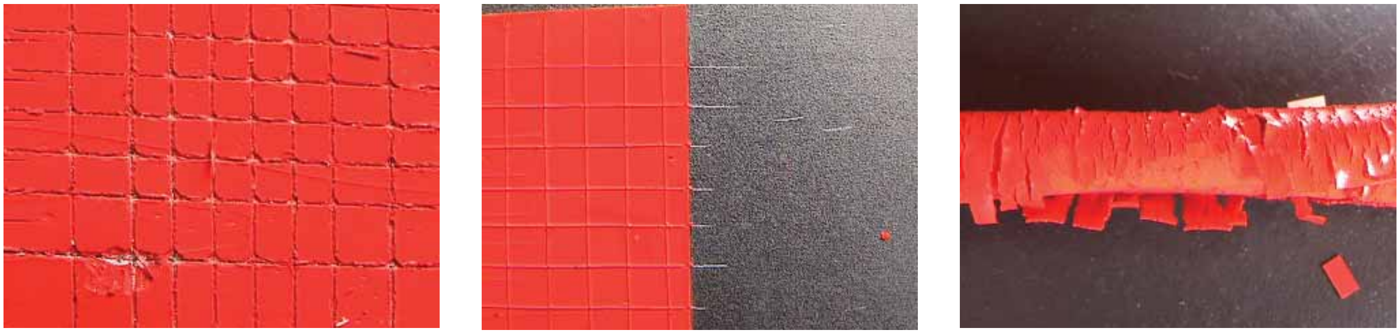


FIGURE 6 Stress-strain curves for Spectrum alkyd priming plus acrylic or oil paint layer. F = number of folds.



(A) (B) (C)

FIGURE 7 (A) Cross-hatch adhesion, unaged oil-acrylic; (B) cross-hatch adhesion, unaged acrylic-acrylic; (C) tensile testing, unaged oil-acrylic.

an acrylic layer. The acrylic–alkyd primer combination went beyond 20% before the complete composite failed, whereas the oil–alkyd primer failed at 12% (aged) and 15.5% (unaged), which correlates well with the crack initiation for the canvas composites of 9.4% strain and 11.6% strain, respectively.

Adhesion Tests

The results from the adhesion tests were unsatisfactory, as in all but a few cases there was no significant difference in the cross-hatch results. The test is difficult to reproduce systematically. The oil paint films were the only ones to show any difference in the degree of loss of paint, whether unaged or aged (figs. 7A and B). This difference was consistent with observations from the tensile testing of these films where peeling away of the oil film from the primer occurred in the unaged samples (fig. 7C). This behavior was also observed for the composite samples. The pull-off tests were also in inconclusive. Bubbles or unevenness in the films or in the adhesion to the stubs led to premature failure. At this stage more testing is continuing to find an experimental procedure that is reproducible. So far, the mandrel bend tests have given a greater understanding of the adhesion between layers, as this test encompasses adhesion, flexibility, and extensibility.

Discussion

There is no obvious incompatibility when combining layers of the different media tested, such as separation of media or stress drying cracks. Acrylic films have the lowest stiffness and least tendency to fracture. A significant change in properties was not found for aged acrylic emulsions. They survived multiple bending and high strains with eventual failure above 25% strain (above the breaking strain of the canvas), resulting in ductile fracture.

Initially, alkyd primers are not significantly stiffer than acrylic primers, but they are less flexible. They do increase in stiffness with aging, and aging reduces the strain to failure of the acrylic paint layer. Significant increase in stiffness and reduction in flexibility occurs with the addition of an alkyd layer. As would be expected, acrylic-alkyd films exhibit brittle fracture at low strains when aged. Reduction in stiffness resulting from fracture in the alkyd or oil layer in the free films corresponds to cracking in the same paint layer of the canvas composite, at approximately the same strain. The load is then shared between the cracked and complete extensible acrylic layer—in all cases premature fracture on the acrylic layers occurs when testing free films. These fractures occur at relatively low strains and are not visible if there is an acrylic paint layer on top, thus, they present an underlying problem.

When "young," oil paint films have similar stress-strain behavior as acrylic films. It is suggested that delamination due to poor adhesion with an alkyd or acrylic primer might present more of a problem when they are in this stage of maturation. Once they have aged, oil paint films cause premature failure of the acrylic primer. This correlates with the adhesion tests, which show the unaged paint films to have relatively poor adhesion. However, adhesion improves as the film matures, thus increasing the interfacial interaction and tendency to promote and propagate fractures.

Conclusions

Bend tests in conjunction with tensile tests have been found to be more useful than cross-hatch tests for understanding the interfacial interaction of these films because they encompass adhesion, flexibility, and extensibility. They highlight combinations of materials that are particularly vulnerable and require further and detailed analysis. Also, the tests replicate more closely a painting on a stretcher, for instance, the bending of the tacking margin around a stretcher member. Visual observation during testing was invaluable for understanding the mechanisms of failure and cannot be underestimated. Such observation also is the crucial link with the real paintings encountered by conservators.

The research is continuing with development of the pull-off adhesion tests and steady state peel testing. The BS adhesion tests are essentially performance tests for a defined set of samples. Other adhesion tests, such as the tapered double cantilever beam (TDCB) test (Moore, Pavan, and Williams 2001) enables the data to be compared with adhesion theory and, although these tests do not represent the real situation, they allow predictions to be made under a defined set of conditions. An investigation into their applicability for conservation mechanics is under way.

Although not directly measured in these tests, it is clear that creep in the acrylics at room temperature and brittle fracture at low temperatures are more of an issue than straining or bending of the films. However, the next phase of the research will continue, with further characterization of a greater range of artists' acrylics and comparison with household acrylic emulsions as composite layers to corroborate the findings reported in this paper. Similarly, a comparison of artist alkyd primers and household alkyds used as artists' materials will be made. In addition, an investigation will be made into the change of properties and degradation of the canvas when acrylic emulsions of differing formulations and dilutions are used directly on the canvas.

Acknowledgments

The author would like to thank Rebecca Gregg, Tom Learner, Maria Charalambides, and Roy Moore.

Notes

1 The fold tests did not follow a standard.

2 Stress = Force divided by the loading area of the sample. Strain = Change in length of the sample under a load, normally expressed as a percentage of the original length.

3 The elastic modulus is a ratio of stress, within proportional limit, to corresponding strain.

4 The secant modulus is not restricted within the proportional limit; it is the slope of a line from the origin to a specified point on the stress-strain curve:

$1 \text{ MPa} = 1 \times 10^6 \text{ Nm}^{-2}$

5 FTIR was carried out on a Nicolet Avatar 360 FTIR spectrometer using a diamond cell placed in a Spectra Tech IR plan microscope. Sixty-four scans were performed at 4cm^{-1} resolution.

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