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Supplementary issue paper

A preliminary examination of *urushi*-based conservation options for the treatment of photodegraded Japanese lacquer using scanning electron microscopy and profilometry

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Urushi is the Japanese term for the sap of the lacquer tree *Toxicodendron vernicifluum*, a natural crosslinking polymer that has been used for thousands of years as a durable decorative coating. Photodegradation combined with fluctuations in relative humidity cause the formation of microcracks that lead to a reduction in gloss and eventual loss of the surface. In Japan, historic lacquer objects are often conserved with *urushi*-based methods called *urushi-gatame* and *suri urushi*. This paper compares the potential effectiveness of these treatments for enhancing the long-term preservation of historic photodegraded Japanese lacquer surfaces. Images of naturally aged lacquer samples were made with a scanning electron microscope (SEM) before they were treated using *urushi-gatame* and *suri urushi*. Samples were then artificially aged, imaged with the SEM, and assessed using a vertical scanning interferometry profilometer. The results indicated that *urushi*-based treatments may be problematic for export- type lacquers with a proteinaceous ground. While further work is required, this preliminary study suggests that, excluding the formation of new microcracking, three dilute *urushi* applications may represent the minimum *urushi*-based treatment required to enhance the long-term preservation of a photodegraded lacquer surface significantly.

Keywords: Lacquer conservation, *Urushi*, *Urushi-gatame*, *Suri urushi*, Treatment asessment

Introduction

Japanese lacquer (*urushi*) is a natural thermoset polymer produced from the sap of *Toxicodendron vernicifluum* (formerly *Rhus verniciflua*) trees. It has been used for thousands of years as a durable decorative coating for objects made from wood, leather, ceramics, and other materials (Takahashi, 2005). The filtered raw sap (*ki* or *kijomi*) *urushi* is used in the manufacture of lacquer objects, particularly for the ground layers. It can also be processed to produce *kijiro urushi*, which has a reduced water content, is more transparent and polymerizes more slowly. Processing may involve *nayashi* (heating/dehydration) and *kurome* (stirring/homogenization) (Webb, 2000; Heckmann, 2002).

When new, lacquer is a resilient material with a high resistance to water, acids and alkalis (Nagase, 1986). It is susceptible to progressive degradation by visible light and ultraviolet (UV) radiation in conjunction with fluctuations in relative humidity (RH). This causes the formation of pinholes, by decomposition and volatilization of the polymerized *urushi* (Kenjo, 1988), which initiate microcracking of the surface and associated loss of gloss (Vogl, 2000; Elmahdy *et al.*, 2011; Liu *et al.*, 2011; Thei, 2011). As damage proceeds, microcracks propagate both vertically and horizontally leading to incremental loss of the surface and associated decoration such as *makie* particles and *nashiji* flakes (Yamashita & Rivers, 2011a). Progressive photodegradation is also associated with increasing sensitivity to solvents and fluctuations in RH (Ogawa *et al.*, 1998; Obataya *et al.*, 1999, 2002; McSharry *et al.*, 2007, 2011). Obataya *et al.* (2002) inferred that raw *urushi* is more susceptible to photodegradation than processed *urushi*.

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Low RH causes moisture loss from the surface of the *urushi*, the contraction of which is restrained by the substrate (Ogawa & Kamei, 2000, Bratasz *et al.*, 2008). The surface of the *urushi* contracts to a greater extent than the lower layers, producing stress across the surface. Vertical cracks occur when the stress in the *urushi* exceeds its failure strength. This causes initial fracture, followed by curling and delamination of the surface from the substrate. The process is the same at both macro and micro levels (Ogawa *et al.*, 1998; Kitsunezaki, 1999; Ogawa & Kamei, 2000).

Until recently, Western conservators viewed photodegradation as regrettable but did not generally undertake conservation of the surface unless degradation had progressed to the point where the surface had become powdery. In such cases, a photochemically stable, re-soluble material such as Paraloid® B-72 was likely to be used. In contrast, in Japan, conservation of light-damaged surfaces with fresh *urushi* was seen as a crucial part of a conservation treatment, essential to the long-term preservation of lacquer (Yamashita & Rivers, 2011a), although synthetic resins were also used on occasion (Williams, 2008; Yamashita, 2009).

Urushi-gatame and *suri urushi* describe techniques that are used in both the manufacture and conservation of lacquer in Japan (Yamashita & Rivers, 2011b). In this paper, these terms are applied in their conservation context. Thus, *urushi-gatame* involves the application of *urushi* diluted with a hydrocarbon solvent to the photodegraded lacquer surface. The first dilute *urushi* application is intended to penetrate to the bottom of the cracks and, if they are deep enough, be absorbed into the ground, sealing the bottom of the crack. After the diluent has evaporated, all excess *urushi* is removed from the surface and the object is humidified to allow the *urushi* that remains to cure. This process may be repeated several times, with subsequent applications accumulating within the microcracks, progressively filling them, although *urushi* is always removed from the surface of the object before it has polymerized. This consolidation treatment aims to stabilize a photodegraded surface while limiting alterations to its appearance. It was developed in Japan to combine the principle of minimal intervention with the use of *urushi*-based treatments.

Suri urushi involves the application of *urushi* to the lacquer surface, which is then gently wiped with a tissue or soft cloth to ensure that the layer of *urushi* left behind on the surface of the object is very thin and even. While the amount of *urushi* applied to the surface may vary, it is implicit that a *suri* treatment will result in deposition of new material on the surface, with a concomitant increase in gloss.

Suri urushi and *urushi-gatame* are best understood as a continuum, rather than as exclusive concepts. The treatments can be undertaken separately, in sequence, or as a hybrid — for example the use of a *suri* technique in combination with dilution with a solvent will both consolidate microcracks and deposit new material on the surface. Several layers of either technique can be applied to a surface: repeated *urushi-gatame* treatments will progressively fill microcracks, while multiple *suri urushi* layers will fill cracks and progressively increase gloss. Use of *urushi-gatame* and *suri urushi* in conservation treatments has been widely documented in Japan (National Research Institute for Cultural Properties, 2001–2012) and, more recently, in the UK (Yamashita & Rivers, 2011b; Coueignoux & Rivers, 2015). Both types of treatment are irreversible once the *urushi* has cured.

A small preliminary study by Keneghan (2011) indicated that, in terms of preservation of a photodegraded lacquer surface, *suri urushi* may be more effective than a minimal two layer *urushi-gatame* treatment and that such a minimal *urushi-gatame* treatment was better than no treatment at all. Coueignoux (2011) assessed the change in appearance caused by the application of *urushi-gatame*, *suri urushi* and various synthetic resins to photodegraded pigmented lacquer. It was reported that the application of *urushi*, a brown resin, did not result in a shift in colour and that the change in gloss was dependent on both the initial degree of photodegradation and the amount of *urushi* that was applied to the surface during treatment.

This paper compares the potential effectiveness of these treatments for enhancing the long-term preservation of historic photodegraded Japanese lacquer surfaces. Naturally aged *urushi* samples were imaged using a SEM. They were then treated, imaged with the SEM, artificially aged and, finally, imaged again. The depth of the microcracks was measured using a vertical scanning interferometry (VSI) profilometer at each stage. The SEM images and VSI data were used to compare the effects of *urushi*-based treatments and assess the implications for the conservation of photodegraded Japanese lacquer surfaces.

Method

Samples

The naturally aged samples tested in this study were taken from the lacquered wood frame of a nineteenth century Japanese screen, as used by Keneghan (2011). The stratigraphy of the samples consisted of a softwood substrate, two proteinaceous ground layers (~100 μm thick in total), a thin black layer consistent with abrasive residues and a black lacquer layer (35–50 μm thick). Samples were taken from the top rail of the frame, where it exhibited the most photodegradation. Each treatment type used three samples, each with an area of approximately 10 mm^2. Owing to limited availability of suitable naturally aged lacquer for destructive testing, only one untreated sample was artificially aged alongside the treated samples, for comparison.

Scanning electron microscopy

The samples were imaged using a variable-pressure SEM (VP-SEM). The co-ordinates of the image locations were recorded using the microscope software, ensuring that the same area on each sample could be revisited at each stage of the treatment and ageing process. A feature or shape on the surface was also used as a reference to ensure that the before and after images were precisely matched. The SEM was used in variable pressure mode at 60–70 Pa to eliminate the need to make the surface conductive by coating the samples, as such a coating would have precluded subsequent treatments, and an accelerating voltage of 10 kV was used. The samples were imaged before treatment, after the *urushi* treatment had been applied and cured, and after six weeks of artificial ageing in a xenon arc chamber. Six measurements were taken from the SEM images for each microcrack type to provide a mean width.

Profilometry

The depth of the cracks and the surface roughness of the samples was measured with a VSI profilometer, which operates by splitting a white light beam onto a reference surface and the sample. The degree of fringe modulation in the recombined reflected beams is measured to produce surface roughness data. It was not possible to revisit the exact area each time, as was done in the SEM, so a random point at the centre of each sample was used. The data from VSI were used to calculate the average area surface roughness, R_a, as defined by the ASME B46.1 standard (ASME B46.1, 2002), given as:

$$R_a = \frac{1}{N}\sum_{i=1}^{N}|Z_i| \qquad (1)$$

where Z_i is the distance from the measured point to the mean plane and N is the number of data points. The area scanned was 235×315 µm. VSI was undertaken before treatment, after treatment, and after three and six weeks of ageing.

Treatments

Table 1 shows the details of the *urushi-gatame* and *suri urushi* treatments compared in this study. Processed *urushi* (*kijiro*) was used for all treatments to limit variables and because it is the most photochemically stable type (Obataya *et al.*, 2002). The aromatic hydrocarbon solvent Solvesso® 150ND was used to dilute the *urushi* for application, while the aliphatic hydrocarbon Exxsol DSP 80/100 (known as ligroine in Japan) was used to remove excess *urushi* from the surface for the *urushi-gatame* treatment (Yamashita & Rivers, 2011b; Coueignoux & Rivers, 2015). After each application of *urushi*, the samples were humidified at 68–70% RH at room temperature (~18°C) for two or four weeks to polymerize the *urushi*.

The first treatment tested was *urushi-gatame*, in which dilute *urushi* is deposited into the microcracks and any excess removed from the surface. This treatment was derived from the *urushi-gatame* method used on the Mazarin Chest (Yamashita & Rivers, 2011b). A mixture of one part *kijiro urushi* to six parts solvent (w/w) was brushed across the surface. The sample was left for 30 minutes to allow the solvent to evaporate and was then wiped using a few drops of solvent on a Kimwipe® 7102 low-lint tissue to remove *urushi* from the surface. The samples were humidified for two weeks. A second application using one part *urushi* to four parts solvent (w/w) was brushed across the surface, left for 30 minutes and wiped as above. The samples were then humidified for four weeks to allow the *urushi* to polymerize.

Three variations of *suri urushi* were tested. *Suri* 1 was based on the consolidation method used on the fragile surface of a small *nashiji* (pear skin) box by Coueignoux and Rivers (2015). *Urushi* diluted 1:4 (w/w) in solvent was applied to the surface. After the solvent had evaporated, samples were humidified for two weeks to allow the *urushi* to polymerize. A second application of *urushi* diluted 1:4 w/w in solvent was applied, solvent allowed to evaporate, and then a Kimwipe tissue wrapped around a fingertip was rolled gently over the surface to simulate the process of consolidating a photodegraded surface with lifting *nashiji* flakes. The samples were then left in the humidifier for four weeks to allow the *urushi* to polymerize.

Suri 2 used three applications of dilute *urushi* — 1:4, 1:4, 1:2, *urushi*:solvent (w/w). The surface was wiped over gently with a tissue and humidified after each application. *Suri* 3 was similar to a traditional Japanese restoration process. A thin coat of undiluted *urushi* was applied to the surface and the sample gently rubbed using a tissue to remove the excess, leaving a thin layer of *urushi* behind. The samples were humidified for two weeks. The process was repeated and the samples humidified for four weeks to allow the *urushi* to polymerize.

Artificial ageing

After treatment and humidification/curing, the samples were artificially aged for six weeks in a xenon arc weatherometer fitted with an indoor daylight filter at 50 °C, 12% RH and an irradiance level of 0.5 Wm^{-2}. This artificial ageing method was

Table 1 Summary of the treatments applied to the lacquer samples

Treatment	1st Application			2nd Application			3rd Application			Ageing	Relative quantity of ***urushi*** used
	Dilution (***urushi***: solvent w:w)	***Urushi*** removed from surface?	Humidification (weeks)	Dilution (***urushi***: solvent w:w)	***Urushi*** removed from surface?	Humidification (weeks)	Dilution (***urushi***: solvent)	***Urushi*** removed from surface?	Humidification (weeks)	Artificial ageing (weeks)	
None	–	–	–	–	–	–	–	–	–	6	0
Urushi-gatame	1:6	Yes: surface wiped with tissue and Exxsol DSP 80/110	2	1:4	Yes: surface wiped with tissue and Exxsol DSP 80/110	4	–	–	–	6	0.343
Suri 1	1:4	No: surface undisturbed	2	1:4	No: tissue rolled over surface without solvent	4	–	–	–	6	0.4
Suri 2	1:4	No: surface wiped with tissue, without solvent	2	1:4	No: surface wiped with tissue, without solvent	2	1:2	No: surface wiped with tissue, without solvent	4	6	0.75
Suri 3	Undiluted	No: surface wiped with tissue, without solvent	2	Undiluted	No: surface wiped with tissue, without solvent	4	–	–	–	6	2.0

based on research in which it had reproduced the surface morphology that most closely resembled the effects of natural ageing (Thei, 2011).

SEM results

SEM images from the surface of the samples are shown at ×100, ×500, and ×1000 magnification. The white box in Fig. 1A shows the location of the ×1000 images, in this case Fig. 1E and F. The relative locations of the high magnification images are consistent in all the subsequent SEM figures, although not specifically marked. The lower magnification images allow comparison of the effects of treatment and ageing on overall morphology, while the higher magnification images allow comparison of the effects of treatment and ageing on individual microcracks and islands.

A general rule of crack propagation is that cracks that form early in the process of degradation are wider and deeper than those that form at a later stage (Groisman & Kaplan, 1994). As a result, two groups of microcracks can be seen in the SEM images — a wider group over 6 μm in width, examples indicated with white arrows in Figs. 1A and 2F, and a narrower group between 2 and 6 μm in width, indicated with blue arrows in Figs. 1A and 2F. A glossary of terms used to describe the observed microcracks appears at the end of this article.

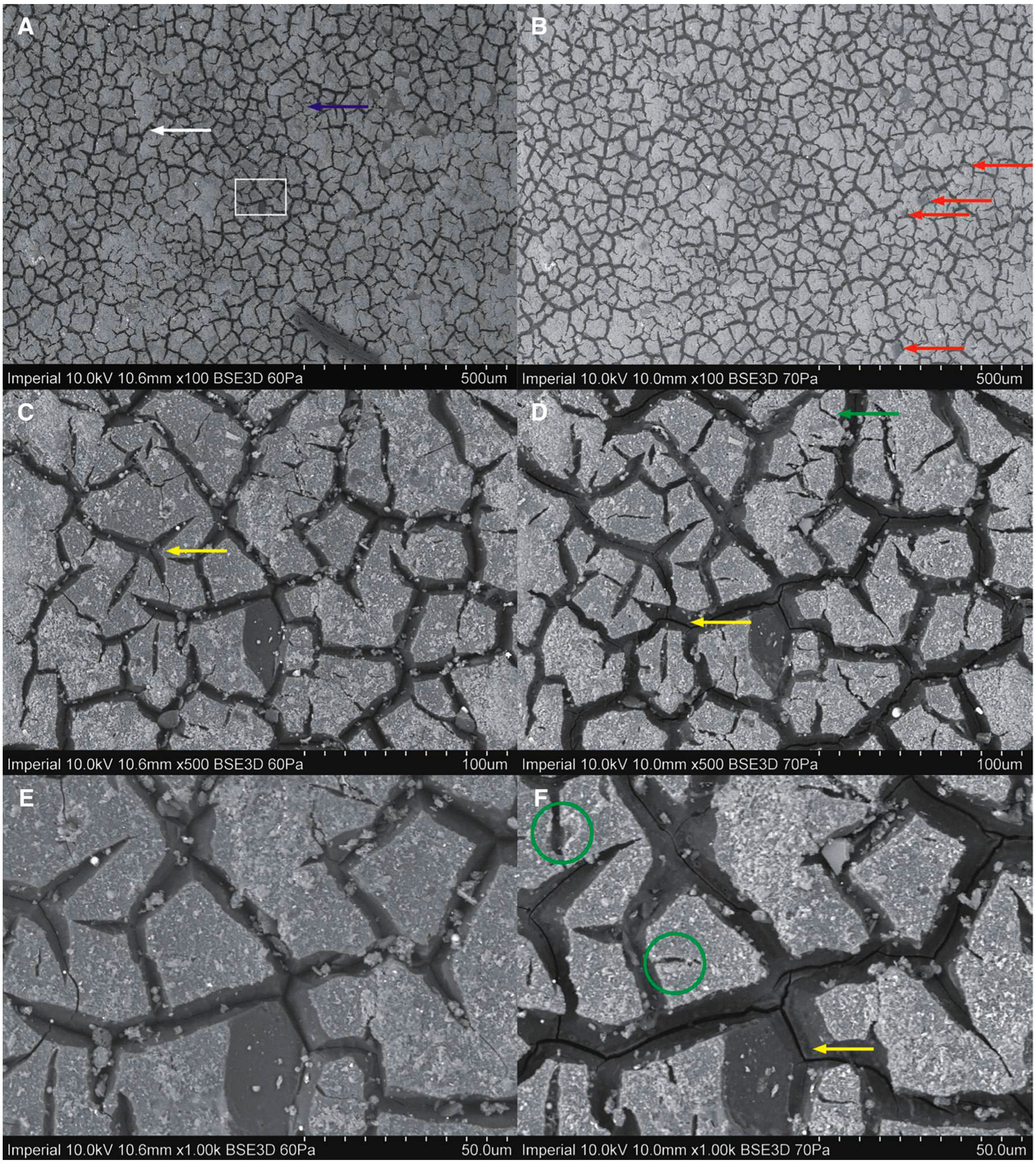

Figure 1 SEM images of the untreated naturally aged control sample before (left column A, C, E) and after artificial ageing (right column B, D, F) at increasing magnification: ×100 (top row A, B); ×500 (middle row C, D); and ×1000 (bottom row E, F).

Untreated sample

Fig. 1A is typical of the appearance of a naturally aged *urushi* surface viewed using the SEM at low magnification, with V-shaped microcracks between irregularly shaped islands and an overall 'mud-crack' pattern. Such mud-crack patterns are typical in desiccation cracking (Corte & Higashi, 1960). Darker areas within islands, for example that seen at the bottom centre of the white box in Fig. 1A, are areas where lateral microcracking had resulted in loss of original surface. Fig. 1B shows the untreated sample after six weeks of artificial ageing. The surface retained a random mud-crack pattern, and the microcracks appeared wider overall. In a few areas part of the lacquer surface of an island had been lost (indicated by red arrows).

Fig. 1C and D shows the untreated sample at ×500 magnification before and after six weeks of artificial ageing respectively. Microcrack widening and losses from the island edges as a result of further ageing are apparent. Hairline cracks (less than 2 μm wide) that had partly extended into islands in the unaged surface had lengthened and in some cases now bisected them (green arrow, Fig. 1D). These hairline cracks generally occur perpendicular to the wide cracks, as a crack will form perpendicular to the tensile stress in the *urushi*, and the tensile stress in the *urushi* will be parallel to the wide crack. Further fissures could be seen at the bottom of many of the deeper microcracks after ageing (yellow arrow, Fig. 1D). Although it is possible that some new fissures formed as a consequence of the progression of the ageing process, several could be seen before ageing (e.g. yellow arrow, Fig. 1C) and it is more likely that the fissures became apparent because the microcracks had widened rather than that they were all newly formed. The change in width of these fissures after ageing was proportional to the change in width of the 'parent' crack — fissures in narrow microcracks remained relatively narrow after ageing while those in the wider group of microcracks widened proportionately more.

Fig. 1E and F shows the untreated sample at ×1000 magnification before and after six weeks of artificial ageing. Narrow fissures could be seen at the bottom of many of the deeper microcracks before ageing. After ageing, hairline cracks within the islands had lengthened and widened (indicated by the green circles in Fig. 1F). Loss of material from the bottom of wider fissures had formed a secondary v-shape at the bottom of some microcracks (e.g. yellow arrow, Fig. 1F).

In summary, the ageing process was characterized by increases in the width, depth and sometimes length of microcracks accompanied by the erosion of material from the top and edges of the islands with a corresponding reduction in their size. These changes were evidence of the incremental damage and loss of original surface that occurs to lacquer surfaces as a result of progressive photodegradation.

Urushi-gatame

Fig. 2A–C shows the *urushi-gatame* treatment (two applications of dilute *urushi*, residues removed from surface, Table 1) at ×100 magnification before *urushi-gatame*, after *urushi-gatame*, and after *urushi-gatame* and six weeks of ageing respectively. Fig. 2B shows that the application of *urushi-gatame* did not change the overall morphology of the sample, although the *urushi* will undergo some shrinkage on polymerization. Some changes can be seen, such as partial loss of the lacquer surface on a few islands (red arrows) and the formation of several new narrow (0.5–1.5 μm) parallel-sided microcracks (green arrows). Fig. 2C shows the sample after six weeks of artificial ageing. The surface retained a random mud-crack pattern with no distinct features that stood out from the surface as a whole, although the microcracks appeared wider overall.

Fig. 2D–F shows the *urushi-gatame* treatment and ageing sequence at ×500 magnification. At higher magnification it can be seen that the loss of surface from the edge of islands after the application of the *urushi-gatame* noted in Fig. 2B was accompanied by additional smaller losses (Fig. 2E, red arrow). Although fissures at the bottom of several microcracks were in many cases more obvious in the images taken after *urushi-gatame*, in most cases these fissures were apparent before treatment. Artificial ageing caused microcrack widening and losses from the island edges. The parallel-sided microcracks that formed after treatment (Fig. 2E, green arrows) widened substantially after further ageing. Some fissures that were apparent before and after treatment (an example is indicated by the yellow arrow in Fig. 2E) were less obvious after ageing, although they were still barely visible as hairline microcracks. This may be because a nearby crack has become wider and deeper, thus reducing the local strain on the fissures, and hence these fissures close slightly.

Fig. 2G–I shows the *urushi-gatame* treatment and ageing sequence at ×1000 magnification. Overall, application of *urushi-gatame* did not appear to reduce the width of the microcracks significantly, suggesting that *urushi* had been deposited at the bottom of the microcracks rather than on the sides. The width of some hairline cracks (originally approximately 0.5 μm) appeared to have reduced (e.g. Fig. 2H, green circles). After six weeks ageing (Fig. 2I) these hairline cracks had reappeared but were not significantly wider than in the original untreated surface.

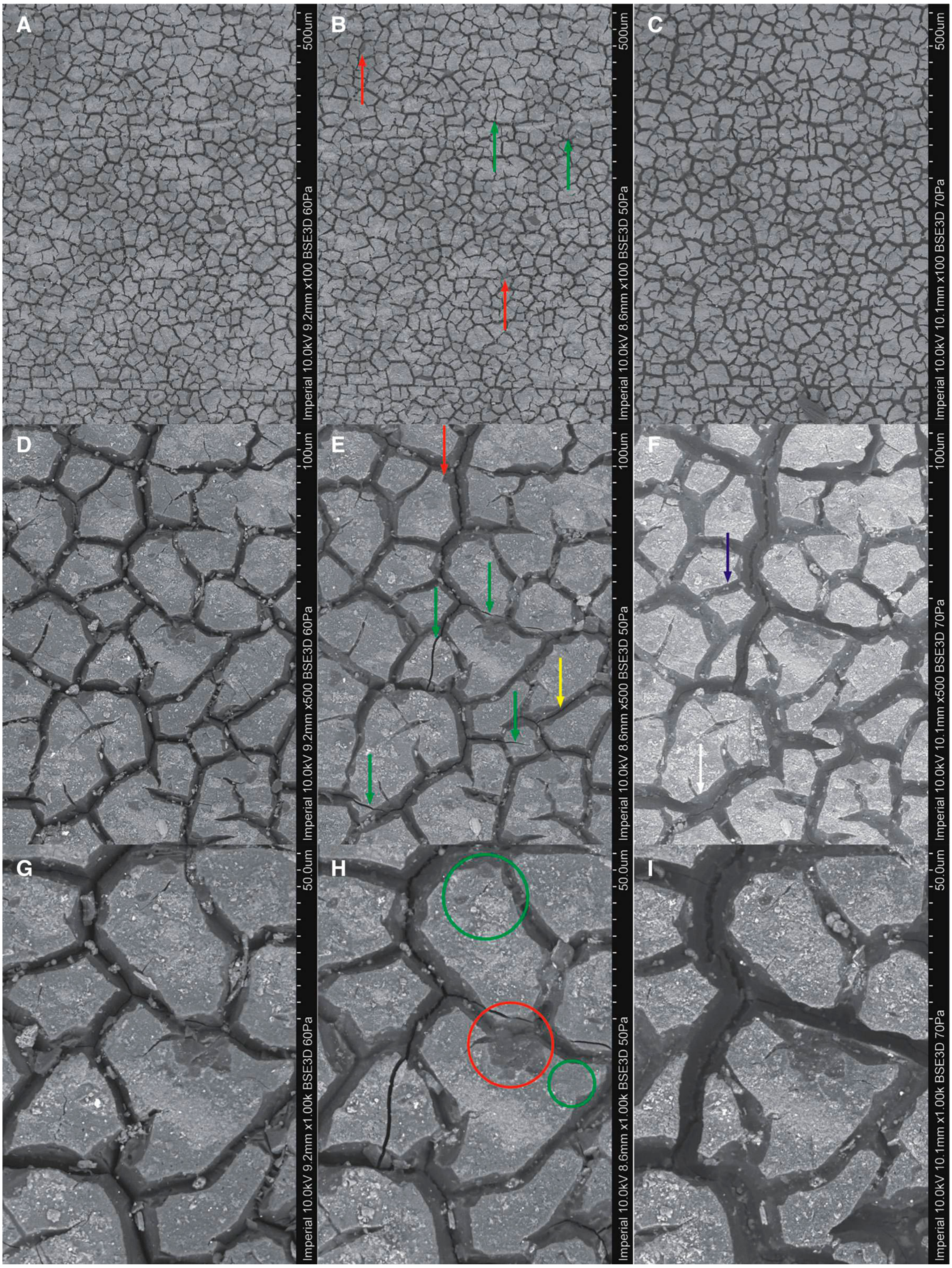

Figure 2 SEM images of *urushi-gatame* treated sample before treatment (left column A, D, G), after treatment (middle column B, E, H), and after treatment and ageing (right column C, F, I) at increasing magnification: ×100 (top row A, B, C); ×500 (middle row D, E, F); and ×1000 (bottom row G, H, I).

Comparison of the appearance of Fig. 2G and H shows that wiping the sample with Exssol® DSP 80/110 was effective in removing excess *urushi* from the surfaces of the islands: *urushi* remained only in the cracks and lower lying areas. The appearance of the area indicated by the red circle in Fig. 2H was smoother after *urushi-gatame* treatment, probably because this slightly recessed area was undisturbed by the cloth used to remove excess *urushi* from the surface. The removal of *urushi* from the surface of the islands supports the assertion made by Japanese conservators that *urushi-gatame* does not significantly alter the appearance of the object.

Suri urushi *1*

Fig. 3A–C shows the *suri urushi* 1 treatment (two applications of dilute *urushi*, Table 1) and ageing sequence at ×100 magnification. Treatment with *suri urushi* 1 did not change the gross morphology of the sample. In contrast with the *urushi-gatame* treatment, the use of *suri urushi* 1 did not result in any losses from the surface of the lacquer islands. Darker areas seen in Fig. 3B are either the result of *urushi* deposition (red arrow) or dust (blue arrow); as with *urushi-gatame*, several new narrow parallel-sided microcracks formed (green arrows). After ageing, the surface retained a random mud-crack pattern with no distinct features that stood out from the surface as a whole, though the microcracks appeared wider overall.

Fig. 3D and E shows an area before and after the *suri urushi* 1 treatment at ×500 magnification. *Urushi* was deposited in low-lying areas on some surfaces (Fig. 3E, red arrow). Fissures at the bottoms of microcracks appeared more obvious after treatment and some had widened (Fig. 3E, yellow arrows). Fig. 3F shows the *suri urushi* 1 sample after artificial ageing. Fissures at the bottoms of the microcracks widened in proportion to the widening of the 'parent' microcrack. *Urushi* deposited on the surface during treatment appeared to have been mostly eroded by the six weeks of ageing. The parallel-sided microcracks that formed after treatment (e.g. Fig. 3E, green arrow) widened significantly after ageing.

Fig. 3G–I shows the *suri urushi* 1 treatment and ageing sequence at ×1000 magnification. Application of *suri urushi* 1 had not reduced the width of the microcracks suggesting that, as with *urushi-gatame*, *urushi* had primarily been deposited at the bottoms of the microcracks, rather than on their sides. Treatment or the low RH in the SEM exacerbated lateral cracking — the example indicated by the pink arrow in Fig. 3H had both branched and extended in length. Loss of original material was apparent in this area after ageing. Some hairline cracks were narrower after treatment (Fig. 3H, upper green circle) but after ageing their width and length were similar to those seen before treatment. In other cases, hairline cracks widened slightly after treatment (Fig. 3H, lower green circle) and widened significantly after ageing. New hairline cracks also formed after ageing (Fig. 3I, green arrow).

Suri urushi *2*

Fig. 4A–C shows the *suri urushi* 2 treatment (three applications of dilute *urushi*, Table 1) and ageing sequence at ×100 magnification. A number of new, narrow, parallel-sided microcracks formed after treatment (Fig. 4B, green arrows) that were substantially longer than those observed on the samples treated with *urushi-gatame* and *suri urushi* 1. Darker areas within islands (Fig. 4B, red arrow) may represent loss of surface from islands, *urushi* deposition or a combination of both. After artificial ageing the sample retained a mud-crack pattern with the additional formation of island clusters, defined as a group of islands, surrounded by distinctly wider and deeper microcracks — an example is indicated in Fig. 4C by a green dotted outline. The formation of wider and deeper microcracks, which split the surface into island clusters, reduces the stress on the lacquer inside the clusters and thus not all of the existing microcracks (especially those within the clusters) deepen and widen.

Fig. 4D and E shows an area before and after the *suri urushi* 2 treatment at ×500 magnification. Treatment resulted in significant deposition of *urushi* into the microcracks and onto the surface of islands. Many new hairline cracks formed in the *urushi* deposited in the microcracks, predominantly at the interface between the newly deposited *urushi* and the edges of islands of original material (e.g. Fig. 4E and H, green arrows). These microcracks are likely to have been caused by shrinkage of the new *urushi* during polymerization and by poor adhesion between the new and the original material (e.g. due to contamination on the surface of the microcracks in the original material). Fig. 4F shows the *suri urushi* 2 sample after artificial ageing at ×500 magnification. Overall, artificial ageing caused the width of the microcracks to increase. A higher magnification view of the perimeter of an island cluster that formed after artificial ageing is indicated by the yellow arrow in Fig. 4F. The parallel-sided cracks that had formed after treatment widened substantially after ageing.

Fig. 4G–I shows the *suri urushi* 2 treatment and ageing sequence at ×1000 magnification. The degree to which the microcracks were filled by the *urushi* deposited by the *suri urushi* 2 treatment varied. This appeared to reflect the original variation in depth of the microcracks, with shallower microcracks (e.g. Fig. 4H, green circle) being filled to a greater degree by the *suri urushi* 2 treatment than deeper microcracks (e.g. Fig. 4H, yellow circles). In all cases, the level of the newly applied *urushi* in the microcracks was below that of the adjacent islands.

As with *urushi-gatame* and *suri urushi* 1, artificial ageing resulted in the formation of new hairline cracks (e.g. Fig. 4I, green circle). Ageing also caused the formation of pinholes (~1 μm diameter) in the *urushi* deposited in the microcracks during *suri urushi* 2 treatment (e.g. Fig. 4I, green arrows). Although the majority of the pinholes formed along the hairline cracks at the interface between the *suri urushi* deposited in the valleys and the edges of the islands of original material (e.g. Fig. 4I, green arrows), a few pinholes appeared towards the centre (e.g. Fig. 4I, green circle). Pinhole formation contributed to the widening of the hairline cracks and has been found

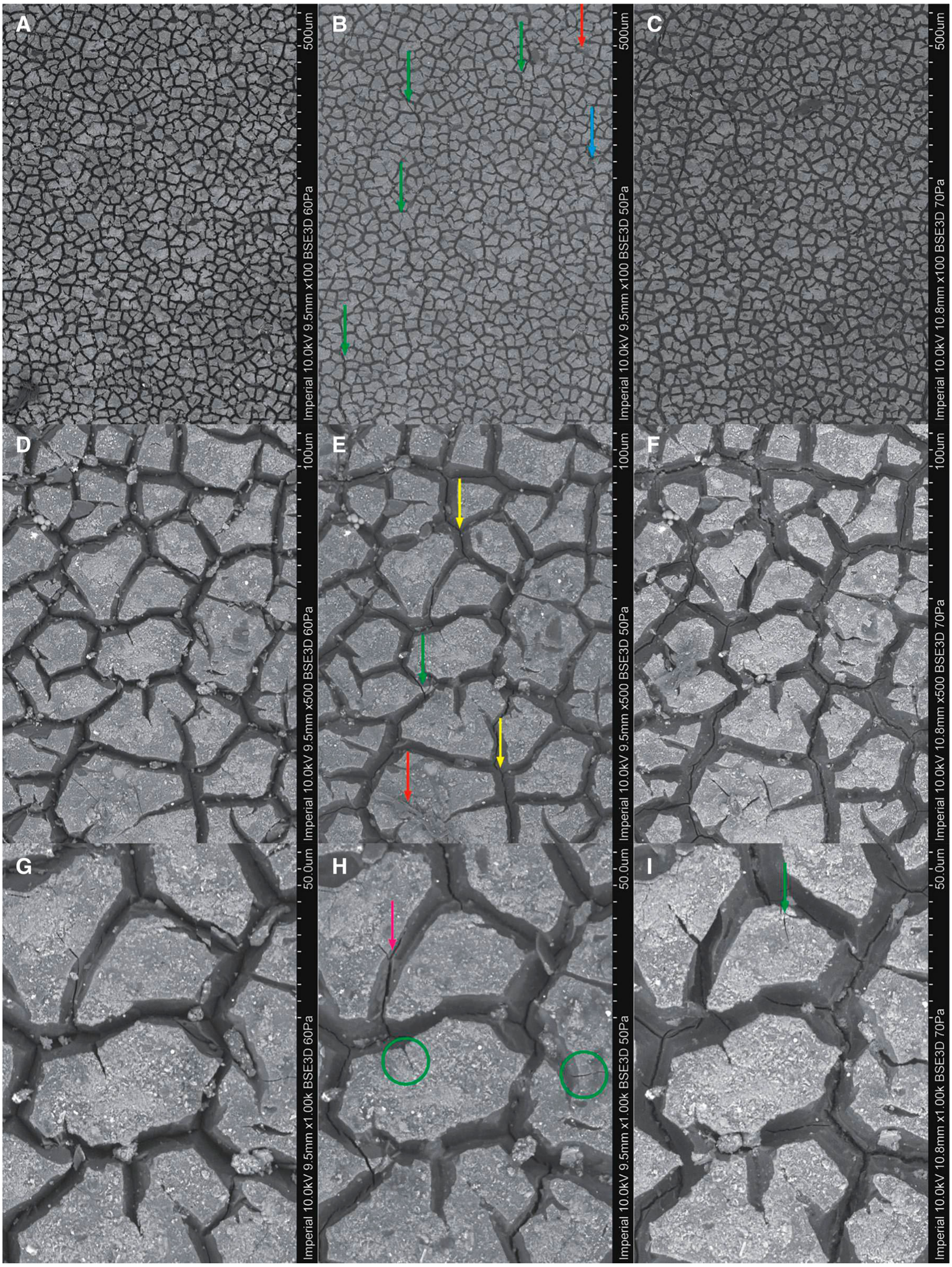

Figure 3 SEM images of *suri urushi* 1 treated sample before treatment (left column A, D, G), after treatment (middle column B, E, H), and after treatment and ageing (right column C, F, I) at increasing magnification: ×100 (top row A, B, C); ×500 (middle row D, E, F); and ×1000 (bottom row H, I, J).

to be a precursor to microcrack formation during artificial ageing studies of fresh *urushi* (Thei, 2011, p. 78).

After ageing (Fig. 4I), the microcracks that were originally shallower (Fig. 4I, green circle) appeared to have retained much of the *urushi* deposited during treatment. In contrast, significant losses had occurred to the deeper microcracks (Fig. 4F, yellow arrow and Fig. 4I, yellow circles). Since microcracks that form early in the process of photodegradation are wider and deeper in comparison to cracks that form at a later stage, it is likely that the deeper microcracks, filled or not, would exhibit a proportionally greater degree of damage as photodegradation proceeded.

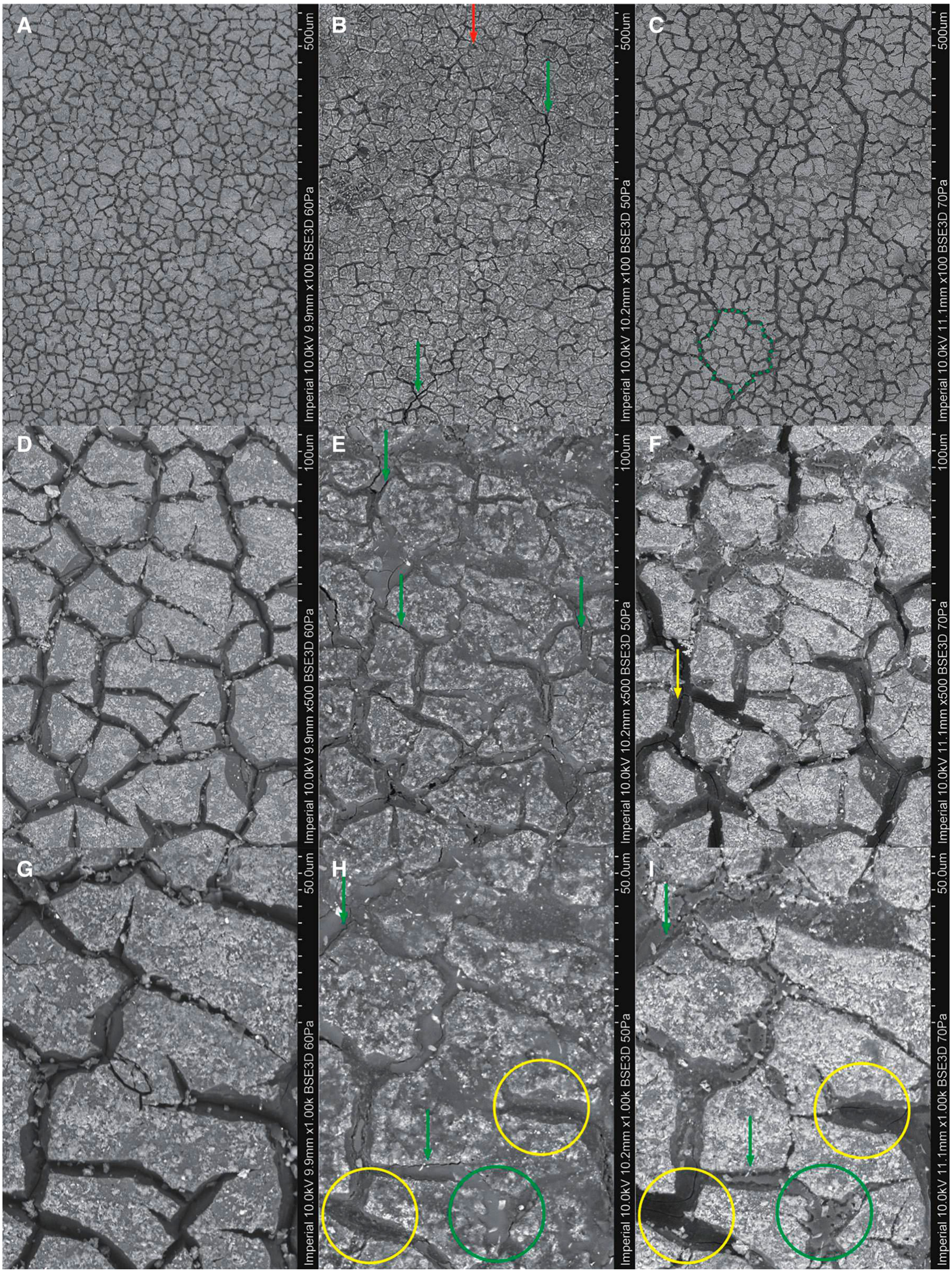

Figure 4 SEM images of *suri urushi* 2 treated sample before treatment (left column A, D, G), after treatment (middle column B, E, H), and after treatment and ageing (right column C, F, I) at increasing magnification: ×100 (top row A, B, C); ×500 (middle row D, E, F); and ×1000 (bottom row G, H, I).

Suri urushi *3*

Fig. 5A–C shows the *suri urushi* 3 treatment (two applications of undiluted *urushi*, Table 1) and the ageing sequence at ×100 magnification. As with *suri urushi* 2, darker areas in Fig. 5B may represent a loss of surface from islands, *urushi* deposition or a combination of both. Some new narrow, parallel-sided microcracks formed after treatment (Fig. 5B, green arrows). After six weeks of ageing the sample retained the overall mud-crack pattern but with the additional formation of distinct island clusters over the whole surface (e.g. Fig. 5C, area with green dotted outline).

In most cases, the parallel-sided microcracks that formed after treatment (indicated in Fig. 5B) were

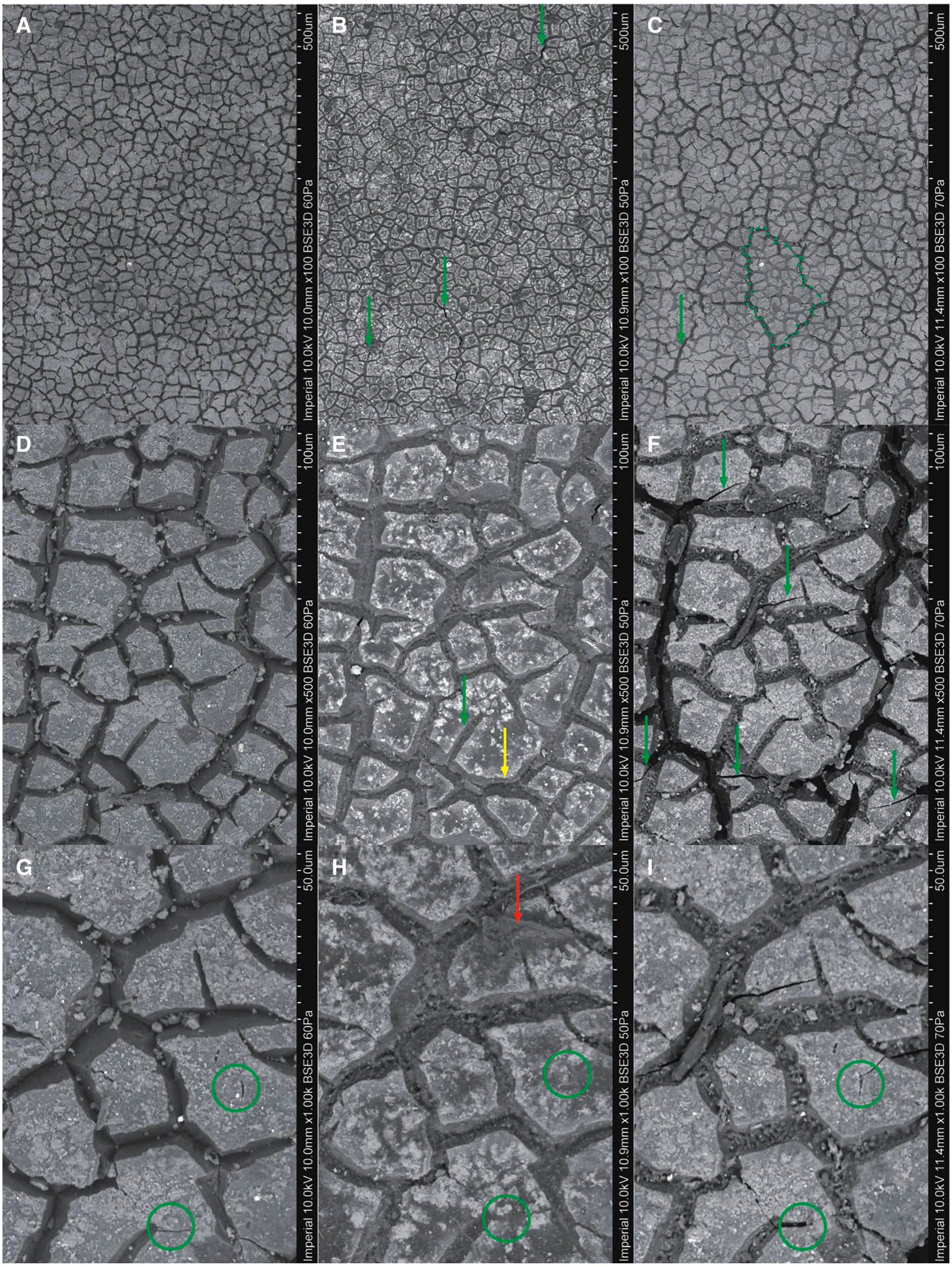

Figure 5 **SEM images of *suri urushi* 3 treated sample before treatment (left column A, D, G), after treatment (middle column B, E, H), and after treatment and ageing (right column C, F, I) at increasing magnification: ×100 (top row A, B, C); ×500 (middle row D, E, F); and ×1000 (bottom row G, H, I).**

incorporated into the island cluster perimeters. These perimeters tended to follow pre-existing microcracks, but also formed new pathways across previously intact islands (e.g. Fig. 5C, green arrow). After ageing, the parallel-sided microcracks that had formed after treatment widened, with those microcracks that were part of the island cluster perimeters widening more significantly than those within the clusters.

Fig. 5D–F shows the *suri urushi* 3 treatment and ageing sequence at ×500 magnification. Treatment resulted in significant deposition of *urushi* onto the surface of the islands and into the microcracks. The *urushi* deposited into the microcracks was noticeably rougher in texture than seen in *suri urushi* 2 treatment, due to the higher viscosity of the undiluted *urushi* used for *suri urushi* 3. As with *suri urushi* 2, the degree to which the microcracks were filled by the *urushi* deposited during treatment reflected the original depth of the microcracks, with shallower microcracks (e.g. Fig. 5E, green arrow) being filled to a greater degree than deeper microcracks (e.g. Fig. 5E, yellow arrow). In contrast with all the other treatments, in which parallel-sided cracks formed only after the treatment stage, ageing of samples treated by *suri urushi* 3 resulted in the formation of a significant number of new parallel-sided microcracks, which in most cases cleaved islands that had previously been microcrack-free (Fig. 5F, green arrows). This suggests that although much of the shrinkage stress associated with the application of new *urushi* had been relieved by the cracking associated with island cluster formation, sufficient residual stress remained to initiate further parallel-sided microcracking within individual islands.

In contrast to *suri urushi* 2, loss of newly applied *urushi* from microcracks after ageing was concentrated in the island cluster perimeter, while within this perimeter the *urushi* deposited in the microcracks remained largely intact (Fig. 5F and I) regardless of the original depth of the microcracks. After ageing, the surface of the *urushi* deposited during treatment appeared rougher and was characterized by extensive pinhole formation. In contrast to *suri urushi* 2, where pinholes had formed at the interface of newly applied *urushi* and the edges of original islands, pinhole formation in newly deposited *urushi* from *suri urushi* 3 treatment occurred primarily along the centre of the microcracks.

Fig. 5G–I shows the *suri urushi* 3 treatment and ageing sequence at ×1000 magnification. Hairline microcracks that had been visible before treatment were filled or covered by the newly applied *urushi* (Fig. 5G and H, green circles), although after ageing these had reappeared and lengthened (Fig. 5I, green circles). Larger areas of *urushi* deposited on the island surfaces during treatment (Fig. 5H, red arrow) were eroded by ageing. The appearance of the surface of the islands after ageing was similar to that before treatment, which may suggest that the *urushi* deposited on the island surfaces during treatment acted as a sacrificial layer, protecting the original surface to some degree.

Discussion of SEM results

The *urushi-gatame* method involves applying *urushi* and wiping the lacquer surface using a solvent to ensure that lacquer is left only in the microcracks. It is an adaptation of traditional materials and techniques that accommodates the conservation philosophy of minimum intervention and is intended to stabilize the object without affecting its appearance. The SEM results indicated that wiping during the initial stage/s of an *urushi-gatame* treatment may cause loss of a small amount of original surface. This could be avoided by adapting techniques from the *suri urushi* 1 treatment — i.e. avoiding any wiping action on the surface or modifying the *urushi* clearance procedure by using a rolling action rather than wiping.

Island cluster formation occurred only in the *suri urushi* 2 and 3 samples, suggesting that in these cases at least, the more *urushi* applied during treatment, the more likely that island clusters would be present after ageing. The limited range of treatments tested means that it was not possible to determine whether this was a result of the overall amount of *urushi* used in the treatment, *urushi* deposition on the surface or a combination of both. In the case of *suri urushi* 3, the similarity in the appearance (texture) of the surface of the lacquer islands before treatment and after ageing suggests that where sufficient *urushi* has been deposited it may act as a sacrificial layer during subsequent photodegradation, helping to retain original surface that would otherwise be lost. This would need to be confirmed by further study in combination with measurement of changes in appearance so that conservators and curators can balance the preservation and aesthetic implications of a potentially heavily interventive treatment.

The changes in width of various types of microcracks are set out in Table 2. By comparing the 'µm difference' columns, it can be seen that the untreated, *urushi-gatame* and *suri urushi* 1 samples formed a group in which ageing produced the same change in width of the narrow (2 µm) and wide (5 µm) groups of microcracks. *Suri urushi* 2 and 3 samples reduced the amount of post-ageing widening of these microcracks suggesting that, in terms of retention of original material, these two treatments were more effective. These observations validate the initial findings of Keneghan (2011), although this study has further developed her methodology and results.

One of the potential risks of using *urushi*-based treatments for conserving photodegraded lacquer is, however, that it involves the use of a comparatively strong material (fresh *urushi*) on a weaker material (original photodegraded lacquer). A possible consequence is that, as photodegradation progresses, damage might occur preferentially in the weaker original material. The formation of parallel-sided microcracks in all samples treated with *urushi* was unexpected. These may have been caused by shrinkage stress exerted by the curing of the newly applied *urushi* on a relatively weak, aged proteinaceous ground. While initially fairly narrow (1–2.5 µm), these cracks widened substantially after ageing (up to 600%).

Table 2 Changes in microcrack width (µm) before and after ageing

	Narrow group (2–6 µm)				Wide group (>6 µm)				Parallel-sided microcracks				Island cluster perimeter			
	Before ageing	After ageing	µm difference	% change	Before ageing	After ageing	µm difference	% change	After treatment	After ageing	µm difference	% change	Before ageing	After ageing	µm difference	% change
Untreated	3	5	2	67	8	13	5	63								
Urushi-gatame	5	7	2	40	11	16	5	55	1	7	6	600				
Suri 1	4	6	2	50	10	15	5	50	1	4	3	300				
Suri 2	5	6	1	20	9	13	4	44	2	14	12	600	7	16	9	129
Suri 3	4.5	6	1.5	33	9	12	3	33	2.5	13	10.5	420	9	16	7	78

As only one type of ground was studied it is difficult to comment on the effect of the ground layer. It may be that the strength of the ground is unimportant in the development of vertical microcracks, as the shrinkage of the wood and the properties of the *urushi* may control overall behaviour. However, a stronger or tougher ground may prevent the microcracks penetrating into the ground, reducing their width. The corollary to this is that if the microcracks penetrate less far into the structure and are narrower then there may be an increased number of microcracks formed for the same curvature of the wood substrate. If the microcracks extend into the ground, then the application of *urushi* may help to consolidate this layer, as discussed by Schellmann & Taylor (2011, 2015).

As both the parallel-sided cracks and the island cluster microcracking can be considered to constitute damage to the original material, *suri urushi* 2 and 3 can be deemed to be less successful treatments of the lacquer surface of the screen from which the samples were taken.

VSI results

The VSI profilometry measurements of crack depth before and after artificial ageing are shown in Fig. 6. VSI permits trends in the changes in crack depth to be identified, which in turn allow comparisons of the effectiveness of conservation treatments. The technique cannot consistently scan to the bottom of the microcracks due to geometric and equipment limitations; for example, if the crack is angled the vertical light beam will not reach its full depth. Fig. 6 shows the data as cumulative percentages of the data points versus the depth of the crack below the surface of the islands (hence the negative values). The plots show the proportion of cracks that are deeper than the crack depth indicated on the *y*-axis. For example, the point at the top left of the plot indicates that 10% of the microcracks are 3.2 µm or deeper (and, therefore, that 90% are less than 3.2 µm deep).

The process of photodegradation is characterized by increases in the depth (and width) of microcracks accompanied by erosion of original material. This deterioration increases permeability and therefore sensitivity to fluctuations in RH, as well as increasing unwanted physicochemical interactions with many solvents used in conservation treatments. The VSI measurements can help to identify treatments that reduce or inhibit the effects of photodegradation by indicating those cases in which the crack depths before and after ageing are similar. In Fig. 6, this would be a treatment for which the before ageing (solid) and after ageing (dashed) plots of the same colour are close together and where the after ageing (dashed) plot lies above that for the untreated and aged sample (indicated by the black dashed line). In

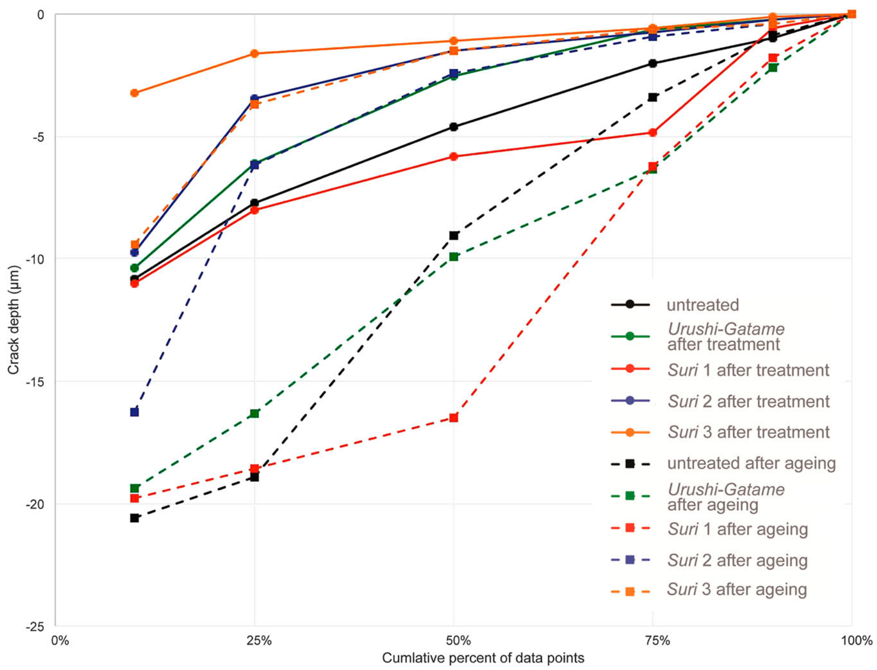

Figure 6 Plot showing the effect of the various treatment methods on crack depth.

Fig. 6, the treatments that meet these requirements are *suri urushi* 2 and 3. A less successful treatment would be indicated by a large difference between the before and after ageing data, and a plot for the aged sample that lies below the plot for the untreated and aged sample, i.e. the *urushi-gatame* and *suri urushi* 1 treatments.

Table 3 uses the VSI data to give the measured depths for a cumulative percentage of data points at the 10 and 50% levels. These are the values at which 10 and 50%, respectively, of the measured cracks are deeper than the given crack depth, measured in microns. The 10% data characterize the deepest micro-cracks, which are generally the parallel-sided and island cluster perimeter microcracks. The use of the 10% data points reduces the effect of any measurement artefacts. The 50% data characterize both the deeper and shallower microcracks, including the shallower microcracks within the island clusters. A successful treatment would give rise to a smaller difference between the depth before and after ageing, tabulated in the columns headed 'µm difference' in Table 3.

Comparison of the pre-ageing 50% data shows that all the treatments reduced the depth of the shallower cracks. The effect was less pronounced for the deeper cracks (10% data) due to their greater volume and depth. The most significant change was for the *suri urushi* 3 treatment, where the use of the undiluted *urushi* had a greater filling effect and hence most reduced the depth of the cracks. The *suri urushi* 2 treatment, which used three applications of dilute *urushi*, reduced the depth more significantly than the *suri urushi* 1 treatment, which used two applications. The *urushi-gatame* and *suri urushi* 1 treatments left the crack depth almost unchanged compared to the untreated sample, because comparatively little *urushi* was used in the treatment and the *urushi* was deposited in the deepest parts of the cracks where it was most difficult for VSI to measure.

For the deeper cracks (10% data), comparison of the post-ageing data shows that, in terms of the difference in microcrack depth measured after ageing, the untreated sample, *urushi-gatame* and *suri urushi* 1 treatments all showed a similar degree and larger

Table 3 Changes in microcrack depth (µm) before and after ageing

	10%				50%			
Cumulative % of data points	Before ageing	After ageing	µm difference	% Change	Before ageing	After ageing	µm difference	% Change
Untreated	10.8	20.6	9.8	90	4.6	9.0	4.4	96
Urushi-gatame	10.4	19.4	9	87	2.6	9.9	7.3	281
Suri urushi 1	11.0	19.8	8.8	80	5.8	16.5	10.7	184
Suri urushi 2	9.7	16.2	6.5	67	1.5	2.5	1.0	67
Suri urushi 3	3.2	9.4	6.2	194	1.1	1.5	0.4	36

amount of change in comparison to the *suri urushi* 2 and 3 treatments. The *suri urushi* 2 treatment produced a small depth difference after ageing and the smallest percentage change. The *suri urushi* 3 treatment produced the smallest depth difference after ageing but the largest percentage change. The large percentage change reflects the markedly lower starting point of this treatment (because the *suri urushi* 3 treatment had filled the microcracks to the greatest extent) and may also reflect the formation of the comparatively deep island cluster perimeter microcracks observed in the SEM images.

The VSI measurements confirmed the trends suggested by the SEM images and showed that the microcracks deepened and widened when the samples were artificially aged. The data also showed that the *suri urushi* 2 and 3 treatments were better than *urushi-gatame* and *suri urushi* 1 at reducing microcrack depth before ageing and producing the smallest change in microcrack depth after ageing.

Surface roughness

The effect of treatment and ageing can also be examined using derived surface roughness data (Table 4) incorporating roughness before and after ageing for each treatment type, as well as the difference after ageing measured in microns and as a percentage. Roughness was calculated across the whole area of analysis, so both the microcracks and the islands were measured. A higher R_a value in Table 4 indicates a rougher surface and therefore a less successful treatment in terms of retention of original material.

An R_a of 1.6 μm was measured for the untreated sample before ageing. The roughness of the *urushi-gatame*, *suri urushi* 1 and *suri urushi* 2 treatments (1.5–2.1 μm) before ageing were not significantly different from the untreated sample when natural variation is taken into account. However, the *suri urushi* 3 treatment reduced the roughness to 1.1 μm, the lowest R_a value recorded, due to deposition of relatively large areas of smooth fresh *urushi* on top of the islands and in the microcracks.

Table 4 Changes in surface roughness after ageing

	Mean R_a (μm) before ageing	Mean R_a (μm) after ageing	μm difference	% Change
Untreated sample	1.6	4.0	2.4	150
Urushi-gatame	1.8	4.8	3.0	167
Suri urushi 1	2.1	5.3	3.2	152
Suri urushi 2	1.5	2.6	1.1	73
Suri urushi 3	1.1	2.1	1.0	91

Artificial ageing increased the roughness of all the samples. The roughness increased because the microcracks became wider and deeper. Much or all of the *urushi* deposited on the islands during treatment was also degraded and/or eroded, causing the surfaces of the islands to become rougher. The samples may be divided into two groups: those where there was a significant increase in roughness after ageing: untreated, *urushi-gatame* and *suri urushi* 1; and those with a comparatively small increase in roughness: *suri urushi* 2 and 3. Indeed, for *suri urushi* 3 the roughness after ageing is approximately equal to that of the untreated sample or the samples treated by *urushi-gatame* and *suri urushi* 1 before ageing.

The roughness measurements confirmed the trends suggested by the width and depth data and the qualitative differences in roughness observed in the SEM images. They suggest that the *suri urushi* 2 and 3 treatments were more effective than *urushi-gatame* and *suri urushi* 1 in slowing the rate of damage, as measured through changes in roughness caused by post-treatment artificial ageing.

Conclusions

This study has compared the potential effectiveness of *urushi*-based treatments for enhancing the long-term preservation of historic photodegraded Japanese lacquer surfaces. As a preliminary study, it has validated a methodology that combined SEM imaging with VSI profilometry and established the need for further research.

SEM imaging showed that artificial ageing of an untreated naturally aged lacquer surface produced an increase of around 65% in the width and 93% in the depth of random mud-crack pattern microcracks. This increase measured the erosion of material from the edges of the islands with a corresponding reduction in their size. These changes were evidence of the incremental damage and loss of original surface that occurs as a result of progressive photodegradation of lacquer surfaces. As the surface is slowly eroded, so too is much of the decorative, aesthetic, historical, technical, and cultural information and value contained in lacquer objects.

Measurements of changes in the width and depth of the random mud-crack-type microcracks suggested that a two-step *urushi-gatame* treatment and a similar two-step *suri urushi* (*suri urushi* 1) treatment produced very similar results to the untreated sample — i.e. their preservation effect was at best minimal. However, it is worth bearing in mind that changes in microcrack width and depth are only one aspect of *urushi*-based treatments — anecdotal evidence has suggested that a two-step *urushi-gatame* treatment significantly reduces physicochemical interactions with solvents used during conservation (Yamashita & Rivers, 2011b).

A three-step dilute *urushi* treatment (*suri urushi* 2) and a two-step undiluted *urushi*-based treatment (*suri urushi* 3) both reduced the amount that the random mud-crack-type microcracks widened and deepened after artificial ageing. However, both these treatments were associated with the formation of new microcracks as island clusters. Through the deposition of *urushi* onto the surface, both treatments are also very likely to change the appearance of the object, in particular to increase the gloss and reduce the overall impression of age.

All the treatment types caused the formation of new narrow (0.5–1.5 μm) parallel-sided microcracks in the surface, which widened substantially (up to 600%) after artificial ageing. The most likely cause is shrinkage stresses exerted by the curing *urushi* in combination with the relative weakness of the proteinaceous ground layers. Proteinaceous ground layers are hygroscopic and can suffer damage and failure as a consequence of fluctuating RH (Michalski, 1991). These factors may also have played a role in the formation of island clusters. Parallel-sided cracks were not caused by the SEM beam, which would have damaged the surrounding surface had this been the case. Both the parallel-sided and island cluster types of microcracking can be considered damage to the original material. These results are significant because they may indicate that *urushi*-based treatments can be problematic for export-type lacquerware with proteinaceous grounds.

SEM imaging provided a very useful visual reference for many of the changes associated with each of the treatments. While microcrack width measurements taken from images necessarily incorporated the potential for a wide margin of error, the data proved consistent with the VSI results. By highlighting the range of different types of changes, for example the formation of parallel-sided microcracks and island clusters, SEM facilitated a more nuanced interpretation of the VSI data than might otherwise have been possible.

In terms of assessing the efficacy of *urushi*-based treatments for the conservation of photodegraded lacquer surfaces, this preliminary study has raised more questions than it has answered. It is clear that further research is required to disentangle both the variables in this study and its limitations. A first step would be to compare surfaces that lie above a lacquer-based ground layer rather than the proteinaceous ground used in this study. Ideally, naturally and artificially aged samples would be tested; each offers particular advantages or disadvantages, and alone are likely to provide incomplete and possibly misleading answers.

A wider range of treatments should be tested in order to separate the variables that may have contributed to these results. It would be helpful to test the effect of the amount of material deposited from different treatments — for example nearly six times as much *urushi* was deposited during the *suri ursuhi* 3 treatment in comparison to *urushi-gatame* and *suri urushi* 1. The addition of further steps to the *urushi-gatame* treatment, as reported by some Japanese practitioners (e.g. Matsumoto, 2010) might enhance preservation without compromising the aesthetics of the object. Comparison with multi-stage dilute and undilute *suri urushi* treatments, particularly correlated with the survival of decoration and changes in gloss levels, would help conservators and curators balance the physical preservation of the object with aesthetic changes. Finally, comparison with similar treatments that utilize high molecular weight, photochemically stable synthetic resins such as Paraloid® B-72 should be included.

Minimal intervention, in the form of the *urushi-gatame* and *suri urushi* 1 treatments outlined in this paper, may not deliver sufficient stabilization/retention of original material. While further work is required, this preliminary study suggests that, excluding the formation of parallel-sided cracks and island clusters, three applications of dilute *urushi* may represent the minimum *urushi*-based treatment required if the long- term preservation of a photodegraded lacquer surface is to be enhanced significantly.

Glossary of microcrack terminology

Wide — over 6 μm in width, examples indicated with white arrows in Figs. 1A & 2F

Narrow — between 2 and 6 μm in width, indicated with blue arrows in Figs. 1A & 2F

Lateral — a crack that propagates parallel to the surface

V-shaped — a crack that narrows to a point at the bottom

Parallel-sided — a crack with parallel sides that does not narrow to a point at the bottom

Fissure — a secondary crack that forms after artificial ageing at the bottom of an existing v-shaped crack

Hairline — a crack less than 2 μm in width that forms on the surface

List of suppliers

SEM (Hitachi S-3400N): Hitachi High-Technologies Europe GmbH, Whitebrook Park, Lower Cookham Road, Maidenhead, Berkshire SL6 8YA, UK.

Xenon arc ageing chamber (QSun Xe-3H): Q-Lab Europe Ltd., Express Trading Estate, Stone Hill Road, Farnworth, Bolton BL4 9TP, UK.

VSI (Wyko NT9100 with Vision version 4.10 software): Veeco Instruments Inc., Corporate Headquarters, Terminal Drive, Plainview, NY 11803, USA.

Solvesso® 150ND and Exxsol® DSP 80/110: ExxonMobil, ExxonMobil House, Ermyn Way, Leatherhead, Surrey KT22 8UX, UK.

Kimwipe® 7102 low-lint tissue: Kimberly-Clark Ltd, 1 Tower View, Kings Hill, West Malling, Kent ME19 4HA, UK.

Health and safety

Liquid *urushi* is an allergen that may produce a mild to severe immune response in susceptible individuals. Protective clothing and nitrile gloves should be worn to prevent contact dermatitis; a high standard of studio hygiene in essential. Hypersensitive individuals may also need to avoid exposure to the fumes of uncured *urushi*. An individual risk assessment incorporating a safe system of work is essential.

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