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# SCANNING ELECTRON MICROSCOPIC EXAMINATION OF ARCHAEOLOGICAL WOOD MICROSTRUCTURE ALTERED BY CONSOLIDATION TREATMENTS

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## Abstract

The scanning electron microscope was used in a study of consolidative treatments for archaeological wooden objects. The surfaces of specimens taken from ancient Egyptian wood artifacts (Dynasty XII, ca. 1991-1786 B.C.) were examined for evidence of microstructural deterioration prior to consolidation with paraffin wax, a typical field treatment. The microstructural appearance of wood subjected to treatments used in conservation was investigated. The distribution of a wax and an acrylic resin used in consolidation was studied, as well as the success of consolidant removal by solvent extraction, and the effects of further treatment with an acrylic resin.

SEM examination revealed significant mechanical, microbiological, and structural damage prior to consolidation. After wax consolidation, microstructure was obscured and surface appearance was significantly altered. Solvent extraction of wax was found to be incomplete. Acrylic resin treatment allowed handling of samples without apparent damage, while maintaining the visibility of wood structure and satisfactory surface appearance. Damage to wood microstructure was observed when solvent alone was used.

KEY WORDS: Consolidant; Archaeology; Paraffin; Wax; Wood; Yew Wood; Egyptian; Deterioration; Scanning electron microscope.

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## Introduction

Many consolidants have been applied to fragile materials in an attempt to infuse them with additional strength. Molten waxes have been used extensively as consolidants on excavation sites in an attempt to prevent loss and damage to fragile archaeological artifacts including objects of material culture as well as skeletal remains. W.M. Flinders Petrie, who excavated a large part of the Metropolitan Museum's Egyptian collection, is noted for the use of paraffin wax to consolidate Egyptian wood artifacts in the field (10). Although this practice did prevent structural disintegration and loss of decorated surfaces during removal, transport, and storage of objects from the field, in many cases it resulted in unexhibitable material. During the late 19th to early 20th centuries, large quantities of artifacts were excavated in Egypt and shipped to Europe and the United States. Often they were prepared for the rigors of travel by the application of molten wax. Where surfaces were not completely consolidated, fragile areas were often completely lost while wax-saturated areas were obscured by dirt and debris. The 1902 accession photograph of a painted wooden model sarcophagus in the collection of the Boston Museum of Fine Arts is an example of an object treated with wax in the field (Fig. 1). The artifact dates from the 30th Dynasty (378-341 B.C.), and was found at Abydos, Egypt.

The preservation of such material was not aided by the poor storage conditions in which these objects were often housed after excavation. The deteriorated condition of such objects has come to light as collections surveys were initiated or as researchers became interested in studying them. Many of these artifacts are unexhibitable unless retreated, and too fragile to handle without stabilization. Retreatment usually necessitates the removal of wax, and reconsolidation with another strengthener. Concern for the stresses this process might inflict on the microstructure of the wood prompted an investigation of the procedure by scanning electron microscopy.

The scanning electron microscope (SEM) has been used very effectively in the study of wood microstructure, both for species identification (2) and in studying aspects of wood deterioration (1). In the wood deterioration study, ancient Egyptian wood was examined by SEM to determine the nature of microstructural damage, revealing mechanical failure in the middle lamella and in cell walls, as well as partial separation between rows of tracheids. Samples appeared to be mechanically intact at the macroscopic level. However, at the ultrastructural level, deterioration was particularly evident during sample preparation, which caused damage at the areas of incipient failure noted above (1).

Uses of Wax in Conservation

Waxes have been used for a wide range of applications to both organic and inorganic materials, not only in archaeological settings. Beeswax and microcrystalline waxes are mentioned in the consolidation of fragile metals (13), and the appropriateness and ease of removal of such treatments have been explored (8,9). Waxes have been applied to buildings as preservatives (4), as well as ancient stone relief carving (5). Wax has been commonly used in the formulation of coatings, adhesives and consolidants. Its use is noted in the treatment of materials such as fossils (6), and ivory (18). Waterlogged archaeological wood has been treated by drying in alcohol, and consolidation with paraffin wax (16), in addition to the more commonly used consolidation with polyethylene glycol (7). Wax-resin mixtures have been documented in the treatment of wall paintings (3) as well as for the conservation of archaeological wooden artifacts (12). Polychromed and gilded medieval and Renaissance sculpture was commonly stabilized by wax impregnation (14).

Some of the negative aspects of these treatments have included: Alteration of surface appearance (color and gloss); difficulty of penetration; the creation of a consolidated, impenetrable surface zone with an unconsolidated core. Loose pieces are often difficult to adhere after wax consolidation. In addition, wax tends to migrate at elevated temperatures. Dust and dirt become embedded readily in wax surfaces, obscuring surface detail. On the other hand, minimal volume change is associated with the setting of waxes (15). They are generally stable chemically, and function as extremely effective moisture barriers. They are also said to protect against fungi and insects (15). Some of these qualities have certainly preserved fragile archaeological objects. However, the problems associated with wax treatments have more recently prompted the use of other resins, such as some of the solvented acrylics (17,19).

The present study was designed to determine whether consolidation treatments or their subsequent removal affected the microstructure of ancient wood specimens. Samples of untreated wood were taken from the inner coffin of Senebtisi (Dynasty XII, Lisht ca. 1991-1786 B.C., Metropolitan Museum of Art 08.200.45) for the present study. The wood was identified as yew wood, Taxus baccata, by the late Dr. Samuel Record, Yale University. Portions of this coffin had been treated in the field with boiling paraffin in an effort to secure loose gold leaf during the period between the discovery of the tomb in 1906 and the publication of the excavation report in 1916 (11).

## Materials and Methods

The views of wood samples examined included original external and cleaved internal surfaces both pre- and post-treatment. Samples were cleaved with a sharp scalpel rather than cut, sawn or ground. Samples measured approximately 2 x 3 x 5 mm. Samples were studied at each of the following stages: 1. Before treatment; 2. after wax impregnation; 3. after wax removal; 4. after re-consolidation with an acrylic resin; 5. after solvent extraction of acrylic resin; 6. after solvent extraction alone; and 7. after consolidation with acrylic alone.

Paraffin was melted in the top of a double boiler, and samples were impregnated with paraffin absorbed by capillary action from the cross-section end. Wax and acrylic consolidant were removed (steps 3,5 above) by soaking samples in five changes of 50 ml. toluene for 30 minutes per change. Re-consolidation (step 4) and acrylic consolidation (step 7) used 5% Acryloid B-72 in toluene (Rohm and Haas, Philadelphia, PA; a methyl acrylate-ethyl methacrylate copolymer). Samples were loosely covered with polyethylene to slow evaporation and prevent skin formation on wood surfaces.

Samples of each of the above conditions, 1-7, plus untreated samples, were mounted on aluminum pin-type stubs with 3M double adhesive tape and sputter coated with approximately 10 nm of Au, in a Polaron E 5100 sputter coater with cold stage and film thickness monitor. SEM operating conditions were from 5 to 20 kV, tungsten filament in an AMRay 1600T (equivalent) scanning electron microscope.

## Results and Discussion

### Untreated Wood

Samples were studied in cross, radial, and tangential section. For the sake of brevity, only the most illustrative views are shown here. Figure 2 shows an untreated sample in radial section at low magnification. Features such as homocellular rays are characteristic of the *Taxus* genus. Figure 3, a radial section at higher magnification, shows features associated with degradation. Note the fraying of wood fibrils at edges of tracheids, cracks in cell walls, and the deformation of the half-bordered pits in tracheid walls. Figure 4 shows typical softwood features such as uniseriate rays. The specimen is clearly degraded, evidenced by the proliferation of fine fungal and/or actinomycetes hyphae. Figure 5, a cross-section at higher magnification shows helical thickenings and the fungal hyphae associated with wood deterioration. This is a typical specimen, with helical thickenings present in all tracheids.

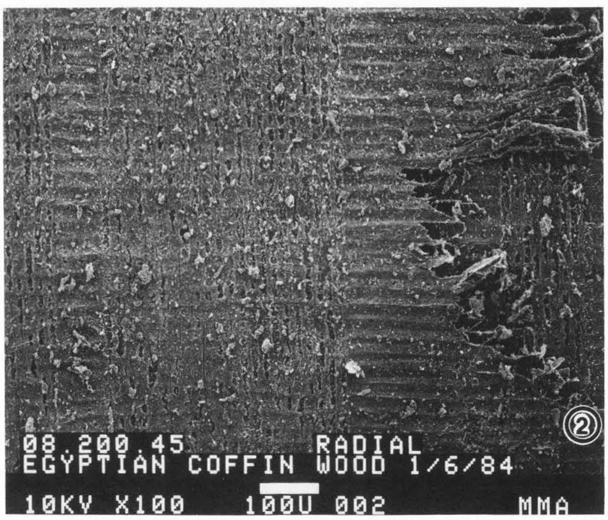




Figure 1. Accession photograph of Boston Museum of Fine Arts 02.31, painted wood model sarcophagus. The object was the gift of the Egypt Exploration Fund in 1902. The paraffin wax visible in the photograph is still present on the surface of the object, but decoration is now predominantly obscured by a thick layer of black soot adhered to the wax.

Figure 2. SEM micrograph of untreated wood sample (exterior, original surface) in radial section, illustrating surface abrasion, disruption of structure, artifacts such as dust. Regular bordered pits are present along tracheid walls. Bar = 100 μm.

Figure 3. SEM micrograph of untreated wood sample (interior, cleaved surface) in radial section, illustrating wood anatomy features such as bordered pits (arrow A), and helical thickenings (arrow B), and features associated with the degradation of wood, such as the proliferation of fungal or actinomycete hyphae (arrow C). Bar = 10μm.

Paraffin Wax Treatment

After the application of molten paraffin, the surface character of the sample was obscured completely (Fig. 6). When wax was applied in an archaeological field setting, debris such as sand, hairs and dirt often became incorporated into surface films of wax. Uneven application of wax resulted in unconsolidated areas adjacent to heavily impregnated areas. These zones react differently to environmental conditions causing splits at the interface between them, both in the wood and in painted surfaces. In addition, surfaces containing wax have a much higher index of refraction than untreated surfaces, causing them to appear "saturated" and often shiny. Although resins were occasionally used, early Egyptian artifacts were decorated primarily with medium-poor, gum-based paints, creating a dry effect very unlike the intensity and gloss produced by wax impregnation.

Paraffin Removal

Even after five successive changes of solvent, a significant amount of wax was left in the wood structure. Figure 7 shows the wood features to be predominantly obscured from view by the substantial coating of wax left after removal was attempted. A comparison between Figures 8 (untreated wood) and 9 (paraffin treated, then solvent extracted) demonstrates the degree of surface detail obscured by the wax residue. Note the proliferation of hyphae in Figure 8, and charging problems associated with the uneven wax residue after consolidant removal in Figure 9. After paraffin removal by extraction with toluene it was found that the wood could not be handled without a high probability of damage. Although a wax coating was visible at high magnifications, its consolidative effects were not adequate in the amounts present. Nevertheless, surface appearance was visually acceptable at this stage.

Consolidation with Acryloid B-72 after Wax Removal

After consolidation with Acryloid B-72 5% in toluene, gross characteristics of wood structure, such as the edges and direction of tracheids, were indistinct but still discernable (Fig. 10). Both


Figure 4. SEM micrograph of untreated wood sample (interior, cleaved surface) in tangential section, showing details of wood anatomy including uniseriate rays (arrow A), and helical thickenings (arrow B). Bar = 10 µm.

Figure 5. SEM micrograph of untreated wood sample (interior, cleaved surface) in cross section, showing helical thickenings characteristic of yew wood (arrow). Bar = 10 µm.

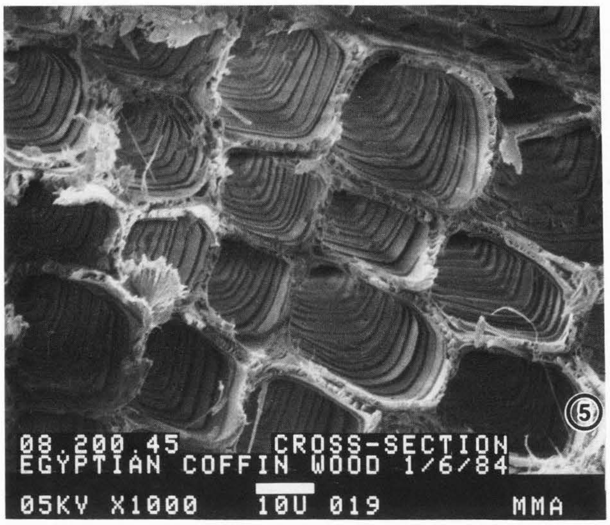
Figure 6. SEM micrograph of wood (interior surface cleaved prior to treatment) in cross section after treatment by immersion in molten paraffin wax. Note that the surface is completely obscured. Bar = 10 µm.

Figure 7. SEM micrograph of wood (interior surface cleaved prior to treatment) in cross section after attempted removal of paraffin by immersion in toluene (50 ml., for 30 minutes, repeated five times). Tracheid walls are still partially coated with wax. Bar = 100 µm.

Figure 8. SEM micrograph of untreated wood (exterior, original surface) in radial section. Fungal and/or actinomycete hyphae are clearly visible (arrow). Bar = 10 µm.

Figure 9. SEM micrograph of wood (interior surface cleaved prior to treatment) in radial section after attempted removal of paraffin as for Figure 6. Note charging problems associated with uneven wax removal (arrow). Bar = 100 µm.

Figure 10. SEM micrograph of wood (interior surface cleaved prior to treatment) in tangential section after paraffin removal and subsequent consolidation with Acryloid B-72 5% in toluene. Note formation of wax globules. Bar = 100 µm.


5
08.200.45 CROSS-SECTION
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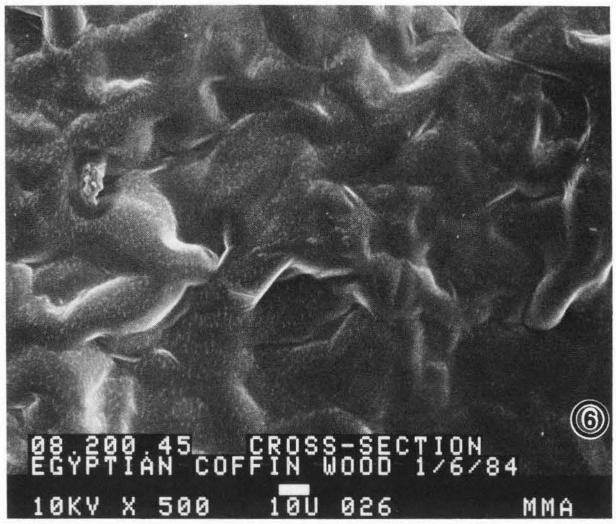
05KV X1000 10U 019 MMA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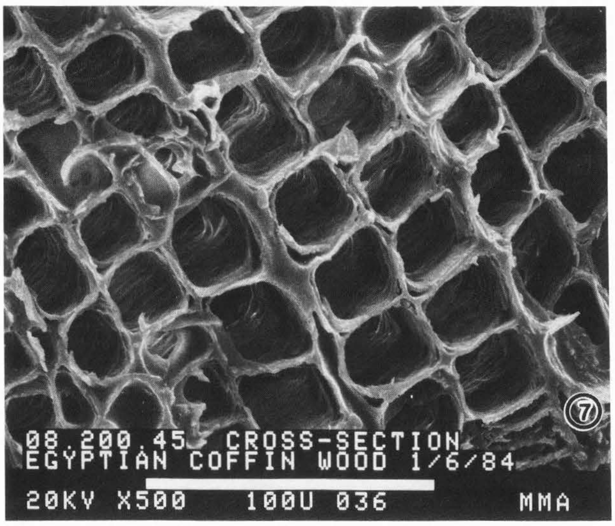
10KV X 500 10U 026 MMA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08.200.45 CROSS-SECTION
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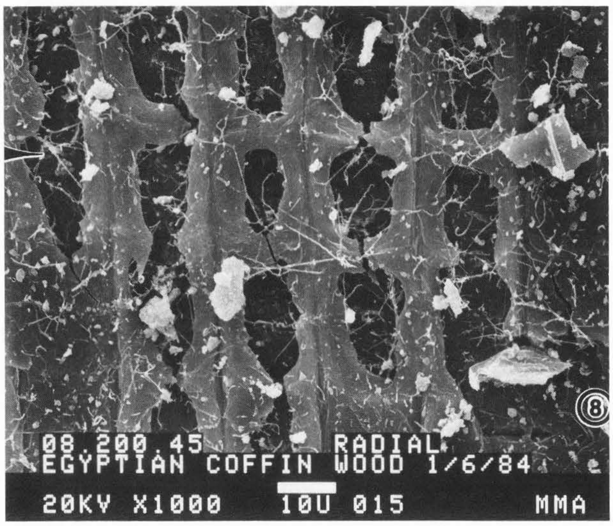
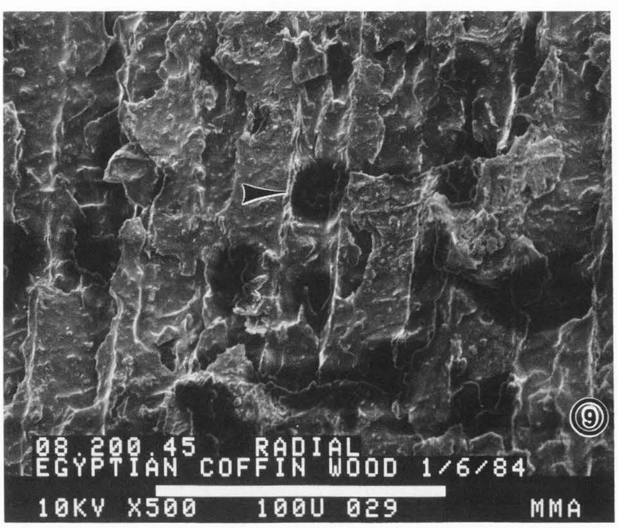
20KV X500 100U 036 MMA


8
08.200.45 RADIAL
EGYPTIAN COFFIN WOOD 1/6/84
20KV X1000 10U 015 MMA


9
08.200.45 RADIAL
EGYPTIAN COFFIN WOOD 1/6/84
10KV X500 100U 029 MMA


10
08.200.45 TANGENTIAL
EGYPTIAN COFFIN WOOD 1/6/84
20KV X500 100U 040 MMA

the wax and the acrylic resin were soluble in the solvent chosen. Coated surfaces took on a globular appearance at this stage. Reconsolidation appeared to have mobilized the wax residue, causing it to coalesce into lumps and be deposited under a film of the acrylic resin.

The formation of wax globules can be seen more clearly in Figure 11. The surface appearance of treated objects was usually satisfactory at this stage, and the wood was generally cohesive enough to be handled, albeit carefully. However, the joining of pieces of objects treated in this manner posed certain problems, as the wax residue made adhesion difficult.

Effects of Solvent Extraction on Untreated Wood

Solvent washing alone caused significant loss of definition of wood characteristics (Fig.12). This tangential view shows the disruption of tracheid walls, as well as general structural disintegration. The microstructure of the wood was severely disrupted here, possibly due to soft rot. However, the cause of deterioration could not be identified by SEM examination.

The cross section revealed the removal of a large amount of fibrous wood material and loss of surface definition (Fig. 13). The wood powdered extremely readily at this stage. Without magnification, the surface appeared desiccated and cracked.

Consolidation of Untreated Wood with Acryloid B-72 in Toluene

After consolidation with B-72, good preservation of surface characteristics was found. (cf. Fig. 14, a tangential view.) All surfaces appeared to be coated with the resin, but the coating appeared somewhat thicker in certain areas, such as tracheid walls. At higher magnifications, identifying characteristics such as the pit and aperture shape were marginally diagnostic for wood identification purposes (Fig. 15). Distinguishing features of wood anatomy were preserved, but substantial detail was obscured. However, the gross surface appearance was good for objects treated in this manner, and handling them was generally possible with a minimum of risk.

Removal of B-72

Reversibility ideally comprises one of the attributes of any conservation treatment. B-72 was removed from consolidated wood by extraction with toluene as described for paraffin removal. Figure 16 shows substantial but not complete removal of resin by solvent extraction. When compared to an untreated cross section (Fig. 17), Figure 16 clearly showed the disintegrated wood structure with helical thickenings dislodged from tracheid walls. Nevertheless, considerably more damage was caused by solvent extraction alone (see the comparison between Figure 18, consolidated with B-72, then solvent extracted, and Figure 19, which was solvent extracted only).

## Conclusions

No evidence of mechanical damage was apparent in the removal of paraffin from consolidated wood. However, removal was incomplete and surface characteristics were masked on a microscopic scale. At the concentrations which remained after removal, paraffin alone was not an adequate consolidant, and made the joining of previously paraffined pieces difficult. The addition of an acrylic resin in a solvent compatible to both consolidants resulted in improved strength and appearance of the wood, but

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Figure 11. SEM micrograph of wood (interior surface cleaved prior to treatment) in cross section after paraffin removal and subsequent consolidation with Acryloid B-72 5% in toluene. Bar = 10 μm.

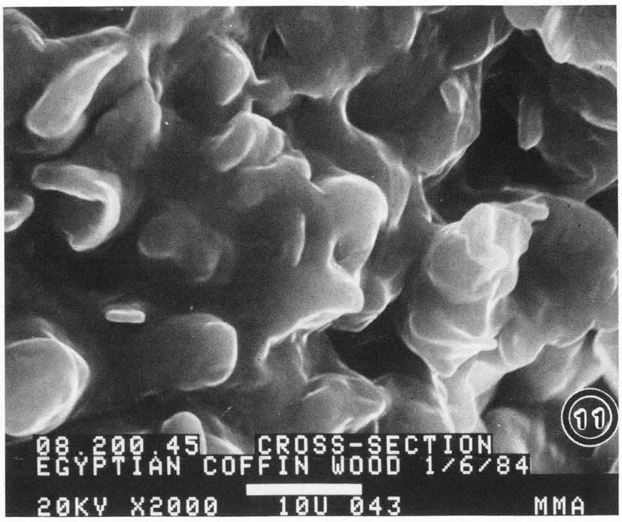
Figure 12. SEM micrograph of wood (interior surface cleaved prior to treatment) in tangential section after solvent washing only (repeated 5 times, 30 minutes each time,in 50ml. toluene). Bar = 10 μm.

Figure 13. SEM micrograph of wood (interior surface cleaved prior to treatment) in cross section after solvent washing as for Figure 12. Bar = 100 μm.

Figure 14. SEM micrograph of wood (interior surface cleaved prior to treatment) in tangential section after consolidation with Acryloid B-72 5% in toluene. Bar = 100 μm.

Figure 15. SEM micrograph of wood (interior surface cleaved prior to treatment) in radial section, although orientation is not discernable. Specimen has been consolidated as for Figure 14. Bar = 10 μm.

Figure 16. SEM micrograph of wood (interior surface cleaved prior to treatment) in cross section after consolidation as for Figure 14, and removal of B-72 by washing with solvent, as for Figure 12. Note incomplete removal and mechanical damage. Bar = 100 μm.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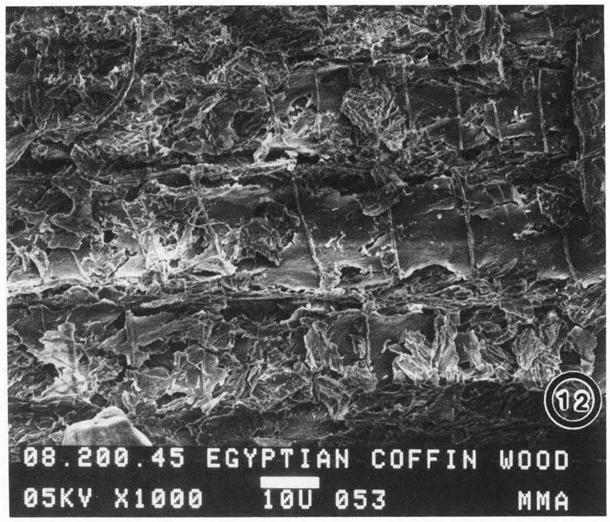
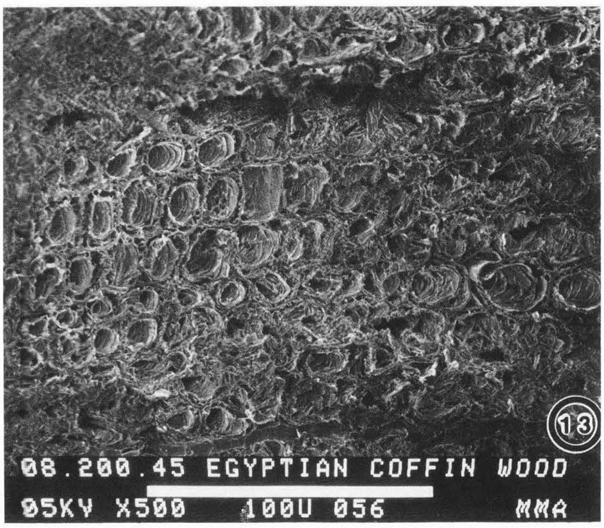
08.200.45 CROSS-SECTION
EGYPTIAN COFFIN WOOD 1/6/84
20KV X2000 10U 043 MMA


12
08.200.45 EGYPTIAN COFFIN WOOD
05KV X1000 10U 053 MMA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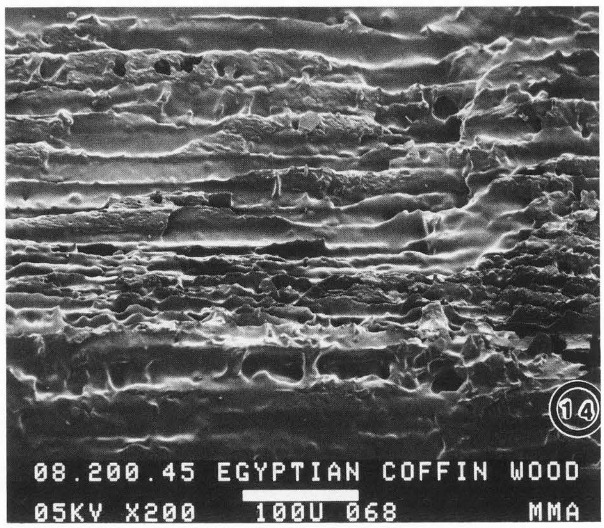
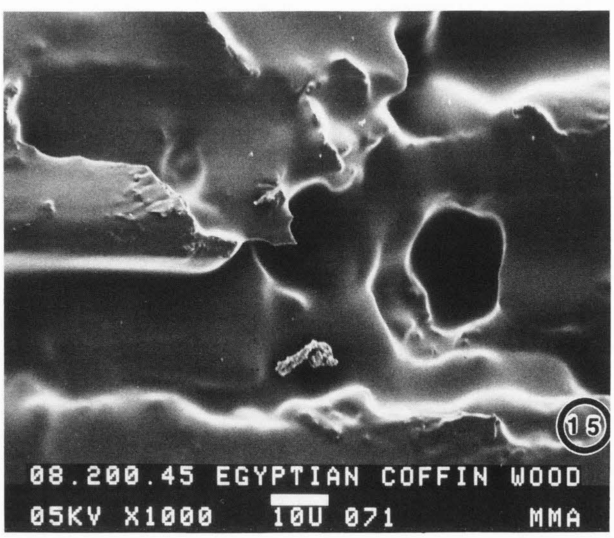
08.200.45 EGYPTIAN COFFIN WOOD
05KV X500 100U 056 MMA


14
08.200.45 EGYPTIAN COFFIN WOOD
05KV X200 100U 068 MMA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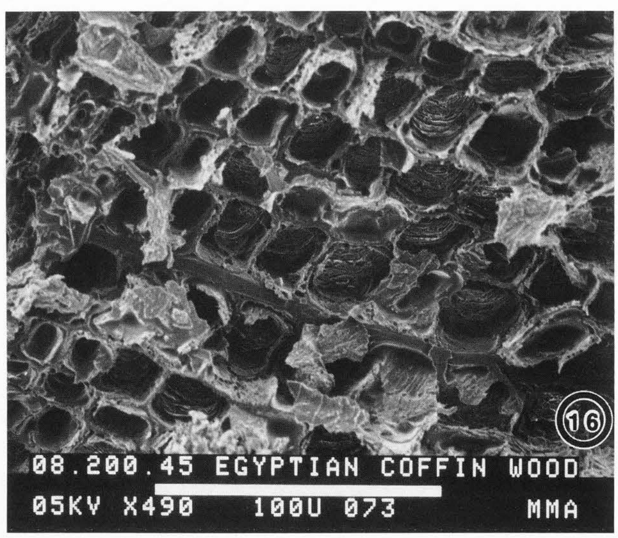
05KV X1000 10U 071 MMA


16
08.200.45 EGYPTIAN COFFIN WOOD
05KV X490 100U 073 MMA

caused the formation of localized aggregates of paraffin on a microscopic level. The acrylic resin alone imparted more strength to degraded wood samples in thinner films than paraffin did, but subsequent removal was not complete, and damage to the substrate was evident.

Acknowledgements

The authors would like to acknowledge the help of Dr. W.A. Côté for assistance with wood identification and clarifying characteristics of wood deterioration. Thanks are also offered to objects conservators at the Metropolitan Museum of Art for the development of treatments which prompted this study, and to the Department of Egyptian Art for providing test samples. The authors thank Ed Brovarski, Curator of Egyptian Art at the Boston Museum of Fine Arts for permission to publish the accession photograph of the tomb model, and to Chris Blair for photographic help.

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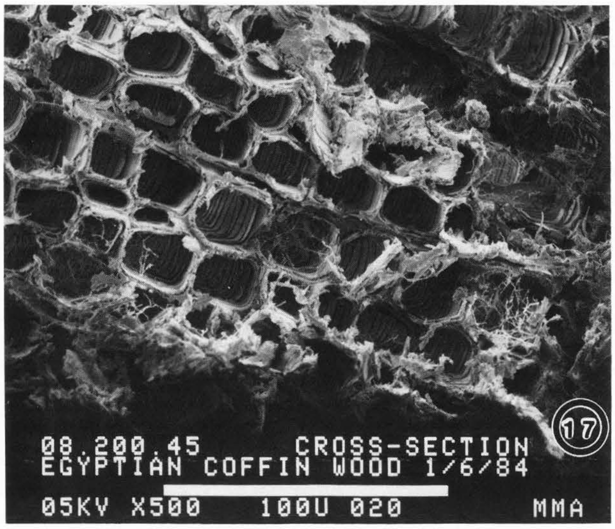
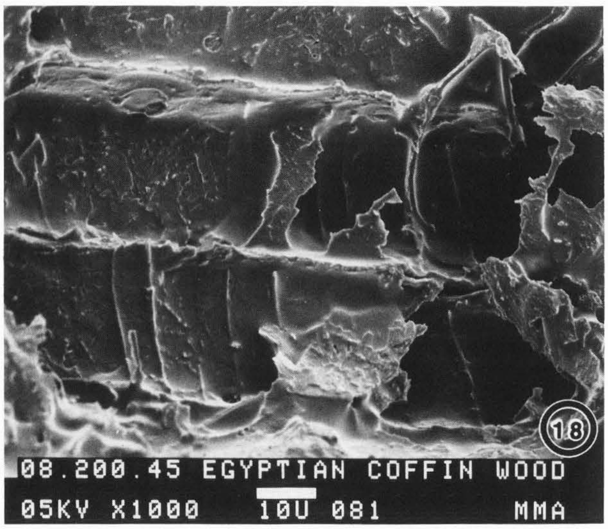
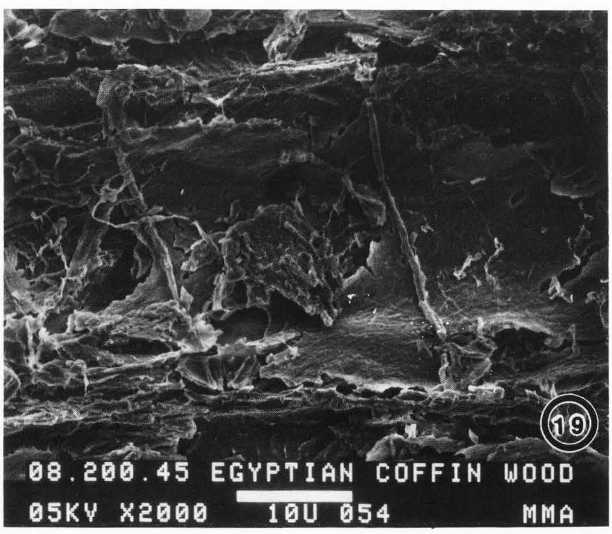
Figure 17. SEM micrograph of untreated wood (exterior original surface) in cross section. Compare to treated cross sections illustrated in Figures 16, 11, 7, and 6. Bar = 100 µm.

Figure 18. SEM micrograph of wood (interior surface cleaved prior to treatment) in tangential section after consolidation with Acryloid B-72 as for Figure 14, and solvent extraction, as for Figure 12. Bar = 10 µm.

Figure 19. SEM micrograph of wood (interior surface cleaved prior to treatment) in tangential section after solvent washing, as for Figure 12. Bar = 10 µm.

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## Discussion with Reviewers

D.P. Kronkright: ...there is no data to suggest that any strength property of wood had been changed as a result of treatment with another material....Had the authors referenced citations in which the strengthening characteristic of acrylic resins had been evaluated in a consolidation application similar to their own, the assumption of strength increase as a result of treatment would have been better supported.

Authors: It was not the intent of this paper to evaluate consolidants by making quantitative determinations of changes in the physical properties of archaeological wood subjected to various consolidation treatments. Our attempt was rather to view at high magnification alterations in surface appearance at different stages in conservation treatments currently being used often in conjunction with the results of earlier field practices. The evaluation of acrylic resins used in conservation treatments is found in several recent publications (see text reference 19, or: Schniewind AP, Kronkright DP (1984) Strength evaluation of deteriorated wood treated with consolidants. In: Brommelle NS, Pye EM, Smith P, Thomson G (eds) Adhesives and Consolidants. International Institute for Conservation of Historic and Artistic Works, London, 146-150).

A.P. Schniewind: The hyphae seen in Figs. 4 and 5 are too thin to be fungal hyphae; they are probably actinomycetes which are not known to be directly responsible for wood decay (although they may be found in association with it).

Authors: It may be that the hyphae in this wood are those from the bacteria that constitute the order *Actinomycetales*. It is also possible that they are fungi, as some fungal hyphae are 1-2 micrometers in diameter--approaching that of e.g., *Streptomyces* (ref. Hickman CJ (1965) Fungal structures and organization. In: Ainsworth GC, Sussman AS (eds) The Fungi. An Advanced Treatise. I. The Fungal Cell. Academic Press, New York, 26). Actinomycetes *have* been found to be involved in wood decay, e.g., *Streptomycetes* spp can degrade cellulosic and lignin components, *S. flavovirens* has decomposed intact cell walls of phloem from Douglas fir, and *S. badius* can degrade lignin (ref. Williams ST, Lanning S, Wellington EMH (1984) Ecology of Actinomycetes. In: Goodfellow M, Mordarski M, Williams ST (eds) The Biology of the Actinomycetes. Academic Press, New York, 491-492).

A.P. Schniewind: What mechanisms would you propose in explaining your observation that solvent alone causes more damage than a solution of B-72? Could it not be that changes such as dissolution of degradation products would be about the same considering that the solution of B72 at 5% is rather dilute, and that residual consolidant masks some of these changes where it is present?

D.P. Kronkright: Perhaps one of the...significant aspects of the research, from a conservation perspective, is the recording of damage done by solvent extraction alone. Clearly many middle lamella and primary cell wall materials were extracted during the course of treatment with solvent and equally as clear is the fact that they contributed significantly to the microstructural stabilization of the woody tissues. I would very much like a follow up study to characterize the extract and the % loss of weight of ancient wood following extraction of this type.

Authors: Although it is possible that the presence of B-72 simply masks the damage caused by solvent washing, evidence for its protective capacity might be indicated by the presence of less debris in the consolidant bath than in the solvent bath after treatment. We prefer not to speculate on the nature of the mechanisms involved in damage caused by solvent washing as compared to consolidation with 5% B-72, and would welcome further study of the subject.

C. Freedland: It would be interesting to vary extraction conditions to see if more complete paraffin removal could be achieved. Either the time of extraction could be lengthened or the temperature changed. Would there perhaps be increased solvent penetration into the wood at a paraffin melting point of 50-60 °C.?

I.B. Sachs: Apparently even after five successive changes of toluene a significant amount of wax is left in the wood structure. Has polyvinyl alcohol (15-20%) and its removal with warm water, or polystyrene with removal with benzene been tried? When I used these techniques for the replication of wood surfaces there was no residue after removal with their respective solvents.

Authors: In structuring other experiments of this nature, variables could certainly be changed to produce better results. However, our intent was to follow the changes caused by treatments as they were performed in the conservation laboratory. It is likely that prolonging solvent extraction would result in further damage to the wood microstructure. The artifacts treated in this manner would probably not be able to withstand higher temperatures, as very often polychromed surfaces are better adhered to applied wax than to wood substrates. It would be inappropriate to subject the polychromed artifacts examined here to treatment with water primarily because the gessoed and painted surfaces are quite soluble in water. Also, polyvinyl alcohol is not favored for consolidation as it tends to become insoluble in contact with many salts and pigments which may appear on museum artifacts (ref. De Witte E (1976) Polyvinyl alcohol. Some theoretical and practical information for restorers. Bulletin de L'Institut Royal Du Patrimoine Artistique, XVI Brussels, Belgium, 124). Perhaps in part due to burial conditions, archaeological wood has been noted to be significantly higher in mineral content than fresh wood (ref. Hoffmann P (1982) Chemical wood analysis as a means of characterizing archaeological wood. In: Grattan DW (ed) Proceedings of the ICOM Waterlogged Wood Working Group Conference. Ottawa, 1981. International Council of Museums Committee for Conservation Waterlogged Wood Working Group, Ottawa, Canada, 73). Polystyrene has not been commonly used to our knowledge for consolidation of wood artifacts, probably because of its light sensitivity, tendency towards brittleness and yellowing. The use of less toxic solvents than benzene is preferred in conservation treatment whenever possible.

I.B. Sachs: Under what conditions are the artifacts kept at the museum before and after researchers become interested in studying them?

Authors: Artifacts may be subjected to a very wide range of conditions between excavation and later examination, treatment and exhibition. Some storage areas may cycle between 10% and 90% relative humidity from winter to summer. Optimally, air is filtered for dust and pollutants, and temperature and relative humidity are kept stable. Temperature should range between 10 and 20 °C. Relative humidity levels for organic objects ideally range between 40-45% (ref. Thomson G (1986) The Museum Environment. 2nd Ed. Butterworths, London, 43, 104).