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X-RAY SUB-MICRON TOMOGRAPHY AS A TOOL FOR THE STUDY OF ARCHAEOLOGICAL WOOD PRESERVED THROUGH THE CORROSION OF METAL OBJECTS*

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Wood preserved in the corrosion layer of two early medieval iron objects was examined using X-ray tomography. A state-of-the art multi-resolution X-ray tomography set-up (http://www.ugct.ugent.be) provided virtual cross-sections of the archaeological wood samples at sub-micron resolution. These were compared with scans of samples of similar modern wood. These scans demonstrate the power of sub-micron X-ray tomography for wood identification, although the process of mineralization pushes this technique to its limits. Furthermore, this technique facilitated appraisal of the mineral content of the archaeological wood, which is useful in selecting the most appropriate strategy for the (preventative) conservation of the archaeological object.

KEYWORDS: WOOD-IDENTIFICATION, ARCHAEOLOGY, X-RAY TOMOGRAPHY, MINERALIZATION, METAL CORROSION

INTRODUCTION

Decomposition of wood is an inevitable process at archaeological sites, resulting in a loss of valuable information for the understanding of material culture, technology and vegetation in the past. Primarily, the interpretation of archaeological wood relies on its taxonomic identification. This is only possible when wood is preserved in such a way that a minimum of its diagnostic anatomical characteristics can be observed and quantified.

Under anaerobic conditions—for example, below the groundwater table and when covered by sediment—the degradation of wood is minimal, as it is only affected by bacteria and actinomycetes (Blanchette 2000; Huisman *et al.* 2008). In those specific environments where microbial and fungal activity is limited, wood can be preserved for centuries. However, when exposed to atmospheric conditions, both physical processes (mainly collapse induced by water loss) and biological processes degrade the microscopic and macroscopic wood structure. When infested by wood-rotting fungi, the mechanical and physical properties are rapidly modified, resulting in a total loss of the wooden remains. Charcoal, on the other hand, is relatively inert and can be preserved in the soil for extremely long periods, regardless of its position relative to the

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groundwater table (Braadbaart and Poole 2008; Scott 2010). Most wood recovered from archaeological sites is, therefore, either waterlogged or charred.

In both types of archaeological wood, the anatomical features required for identification are generally well enough preserved. Waterlogged wood can easily be cut for the preparation of thin sections, to allow the examination of these features using transmitted light microscopy. On the other hand, the preparation of thin sections from charcoal cannot be made without laborious embedding procedures (Igersheim and Cichocki 1996). However, in most cases, sizable charcoal fragments can be identified by studying fresh break surfaces using reflected light microscopy.

In particular cases, where both metal objects and wood are in close contact, wood (and other organic material) can be preserved in a special way. In fact, the corrosion of metal can actually induce a gain in information (Huisman 2009). By anodic and cathodic processes, wood can be coated or become (partially) replaced by metal oxides and/or salts. In the case where copper is present, the corrosion inhibits fungal decay of organic matter. As a result, the organic material itself is preserved. This is in contrast to iron corrosion, which produces coatings of iron on decaying organic matter. The end result is an imprint of the original material in the corrosion layer, while no original material is left (Cronyn 1990). Both processes can result in the preservation of the microscopic structure, provided that the corrosion advances more rapidly than biological deterioration (Keepax 1975, 1989; Watson 1988). In some cases, the preserved mineralized wood fragments can be identified using light microscopy or scanning electron microscopy (SEM). However, mineralized wood is generally very brittle and in most cases only small fragments are preserved. Also, it often crumbles to powder when touched. Therefore, identification using conventional light microscopy (both transmitted and reflected light) is rarely possible or very laborious and highly destructive for the material studied (Keepax 1975). Also for SEM, fragments of the mineralized wood need to be sampled and broken at least three times to obtain a transverse (Tv), radial (R) and tangential (Tg) section (Ponting 2004). This requires both skill by the operator and sizable wood remains that allow the production of cross-sections on which diagnostic, wood-anatomical features can be observed. Despite the irregular surface of the cross-sections obtained by breaking or splitting, SEM is in some cases able to yield images that provide enough detail to allow species identification.

To overcome these practicalities, recent advances in X-ray tomography, such as sub-micron tomography and phase contrast (enhanced) tomography, open up new perspectives, as they offer the possibility of obtaining visual access to the three conventional planes of the wood (Tv, R and Tg) on a microscopic scale and only require extremely small samples (Van den Bulcke *et al.* 2009; Mayo *et al.* 2010). Therefore it was decided to examine a selection of wood fragments preserved in the corrosion layer of metal objects from a Merovingian burial ground from Broechem (Belgium). The use of laboratory-based systems for the microscopic observations of anatomical features on wood preserved in the corrosion of archaeological metal objects offers a very convenient alternative to synchrotron beam lines to which access is limited.

MATERIALS AND METHODS

The Merovingian cemetery at Broechem

The studied material was excavated at the Merovingian (late fifth to seventh century AD) cemetery of Broechem, Belgium, where 388 inhumation graves have been recorded so far (Debruyne and Annaert 2009; Annaert 2010). Many of the graves contain grave goods, such as knives, buckles, fibulae, axes, spearheads and fire steels. These objects are made out of iron, bronze or silver, and

Figure 1 *(a) The long* seax *and the* francisca *axe from the Broechem cemetery; (b) a detail of the axe-eye of the* francisca*, where wood is preserved; and (c) a detail of the sheath of the long* seax*, where wood is preserved in the corrosion layer (© H. Denis, Flanders Heritage Agency).*

on many of them the remains of organic material, such as wood, leather, textiles or horn, are preserved in the corrosion layer. When focusing on the microstructure of the organic material, it was observed that wood fragments were still recognizable.

The studied wood remains come from the corrosion layer on two iron objects from this archaeological site (Fig. 1 (a)). In the axe-eye of a *francisca* (08-BROE-1984), a throwing axe, mineralized wood fragments from the handle were preserved (Fig. 1 (b)). The other studied wood fragment comes from the corrosion on the sheath of a long *seax* (German: *langsax*) (07-BROE-1330) (Fig. 1 (c)).

X-ray sub-micron tomography

The X-ray sub-micron computed tomography (CT) infrastructure employed in this paper was assembled at the Ghent University Centre for X-ray Tomography (UGCT; http://www.ugct.ugent.be). The scanner is similar to the one described in Masschaele *et al.* (2007) and Van den Bulcke *et al.* (2009), and has a generic CT scanner control software platform (Dierick *et al.* 2010). It is a state-of-the art multi-resolution X-ray tomography set-up, specifically designed to permit very high resolution scans of small objects as well as scans of larger objects. For this study, the focus was on small objects, aiming at the highest resolvable detail. As such, all samples were scanned with similar settings for the open nanofocus X-ray tube (Hamamatsu L1711), reaching a focal spot size <1 μm, which is required to achieve the high resolution presented in this paper. The beam of X-rays emitted by this X-ray tube consists of a spectrum of X-ray photons with different wavelengths (energy) and therefore it is called polychromatic. The specific nature of the archaeological samples necessitated both hardware and software corrections for a series of possible imaging artefacts, which are elaborated on in the discussion section. On average, each sample took 2–3 h of scanning, resulting in scans with an isotropic voxel pitch (i.e., the length of a side of the volume element) of approximately 0.75 μm.

Sample size and handling

Dry samples of approximately 1 mm^3 were taken from the wood preserved in the corrosion of both selected metal objects (Fig. 2), by using a scalpel under a stereomicroscope. They were adhered to a sample holder (Ø 0.9 mm) in order to manipulate and position the sample accurately between the X-ray source and detector.

Furthermore, comparable fragments were taken from modern wood samples (*Ilex aquifolium* L. and *Prunus avium* (L.) L.) and scanned using the same device and reconstruction parameters in order to serve as a reference for the archaeological samples. These modern wood samples are

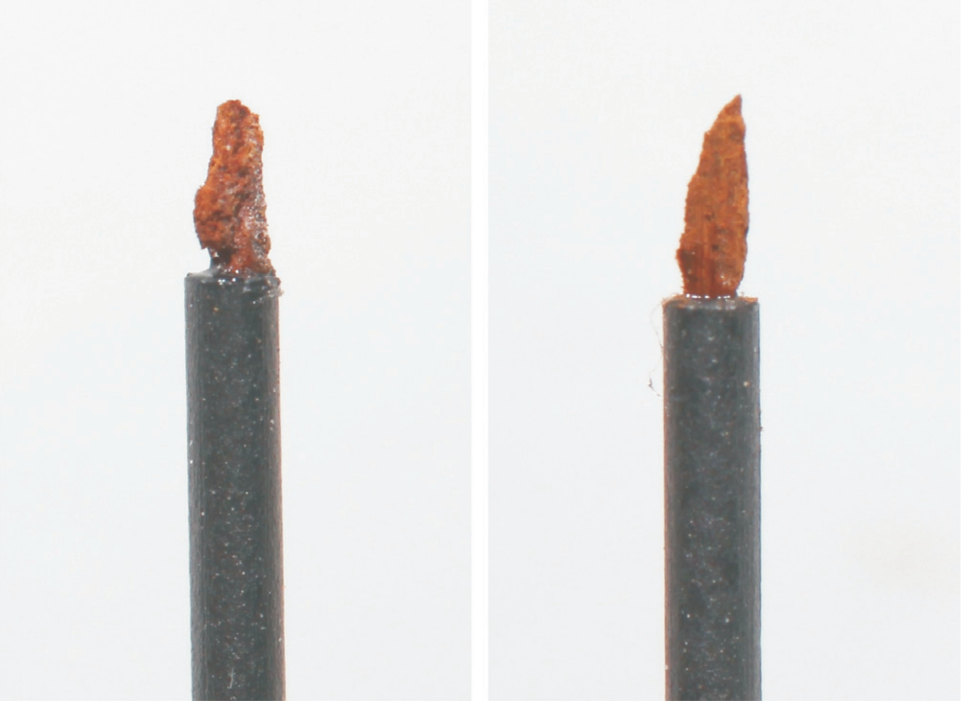

Figure 2 *Two samples for X-ray sub-micron CT of wood preserved in the corrosion layer of two iron objects from the Merovingian cemetery at Broechem. The fragments are mounted on small cylindrical sample holders (Ø 0.9 mm).*

expected to provide the best possible result in terms of visualizing wood anatomical features by means of X-ray sub-micron tomography, since they are not degraded by biological or physical processes.

Visualization and wood identification

The reconstruction of the individual scans was performed using Octopus, a tomography reconstruction package for parallel and cone-beam geometry (Vlassenbroeck *et al.* 2007). All samples were phase contrast filtered using the Bronnikov Aided Correction method (BAC; De Witte *et al.* 2009). Virtual slices with transverse, tangential and radial orientation were produced and visualized in Morpho+ (Brabant *et al.* 2011) by proper rotation of the volume. Furthermore, 3D representations of the internal structure of the wood were generated using VGStudio MAX®.

All wood anatomical features were recorded according to the IAWA list of microscopic features for hardwood/softwood identification (Wheeler *et al.* 1989; Richter *et al.* 2004). Taxonomic identification was performed using identification keys (Schweingruber 1990), illustrated atlases of microscopic thin sections (Schweingruber 1990; Wagenführ 2007) and online databases of wood anatomical descriptions accompanied by images showing anatomical details (‘Inside Wood’, http://insidewood.lib.ncsu.edu/search [2010] and ‘Wood anatomy of Central European species’, www.woodanatomy.ch [2010]).

RESULTS

Taxonomic identification of the (archaeological) wood samples

The X-ray sub-micron CT scans of both modern and archaeological samples yield images on which the internal structure of the wood is clearly visible (Figs 3 and 4). An isotropic voxel pitch of $0.75 \times 0.75 \times 0.75\ \mu m^3$ results in scans of sub-micron resolution.

On both types of sample (archaeological and modern wood), the presence and arrangement of vessels is visible on virtual Tv sections (Figs 3 (a) and 4 (a)), although in one of the archaeological samples, vessel arrangement is partly obscured by the deposition of metal oxides in the lumina of both vessels and fibre-tracheids (Fig. 3 (a)). Furthermore, the height and width of wood rays can easily be observed (Tg section). The heterogeneity of the rays (R section) can be determined and specified according to the presence and arrangement of procumbent, and/or squared and upright cells (type I, II or III).

Although not all wood anatomical features could be observed, both archaeological samples could be identified to species or genus level. The wood from the axe-eye of the *francisca* is believed to be made out of holly (*Ilex aquifolium* L.). The wood is characterized by a dense structure with small vessels, which are arranged in radial rows (Fig. 3 (a)), and by many rays that are 4–8 cells wide and often up to 4 mm high (Fig. 3 (d)). The multiseriate rays are heterogeneous (type II and III), and uniseriate rays consist exclusively of upright cells (Fig. 3 (c)). However, some other diagnostic, microscopic features for *Ilex aquifolium* are much harder to observe on the obtained sub-micron CT scans of the archaeological wood samples. For instance, the type of the perforation plates between the vessel elements was very hard to determine. Only on very few locations within the scanned sample could remains of the scalariform perforation plates be observed. Although the total number of bars could not be counted on the archaeological samples, a virtual 3D view of modern *Ilex* wood demonstrates that this type of perforation plate can indeed be detected (Fig. 5). Even more difficult is the visualization of spiral thickenings. Although these

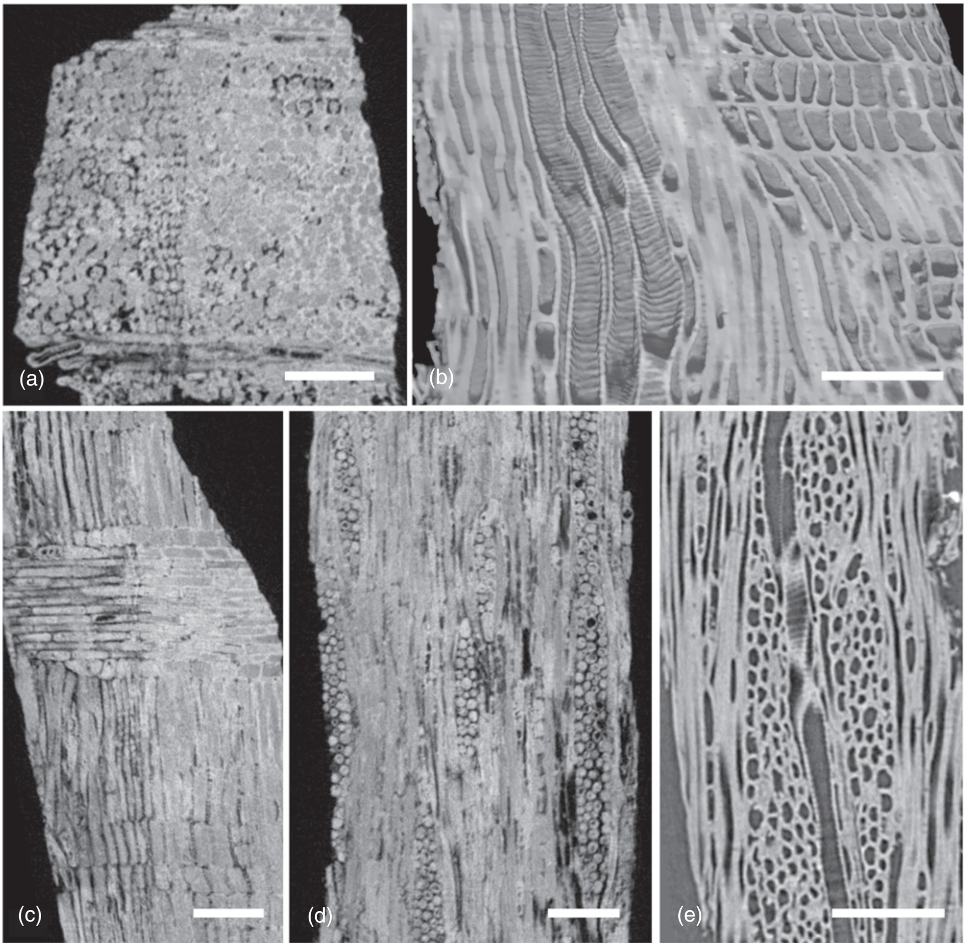

Figure 3 *Virtual sections and 3D representations of the modern* Ilex aquifolium *sample and the archaeological wood preserved in the corrosion layer of the Merovingian* francisca*: (a) a transverse section of the archaeological sample; (b) a 3D view of the modern* Ilex *sample, showing vessels with spiral thickenings; (c) a radial section of the archaeological sample with heterogeneous rays; (d) a tangential view of the archaeological sample; (e) a detail of a tangential section of the modern* Ilex *sample. The scale bars represent 100 μm.*

helical ridges on the secondary cell wall of the vessels are clearly visible on modern wood samples of *Ilex aquifolium* (Fig. 3 (b)), they could not be observed on the archaeological samples. Nevertheless, spiral thickenings were observed on wood samples from the same metal object with conventional reflected light microscopy.

The scanned wood sample from the shaft of the long *seax* has been identified as *Prunus* sp. On virtual Tg slices, it is clear that the rays are 2–4 cells wide and 15–30 cells high (Fig. 4 (b)), although single-row rays are also present. The heterogeneous rays predominantly belong to type I, with one row of squared cells bordering the ray (Fig. 4 (d)). Perforation plates were not clearly visible, but probably belong to the simple type. Again, spiral thickenings could not be visualized on the virtual slices, but were observed on other small wooden samples from the same metal object with reflected light microscopy. Also, on the scans of the modern *Prunus* samples, spiral

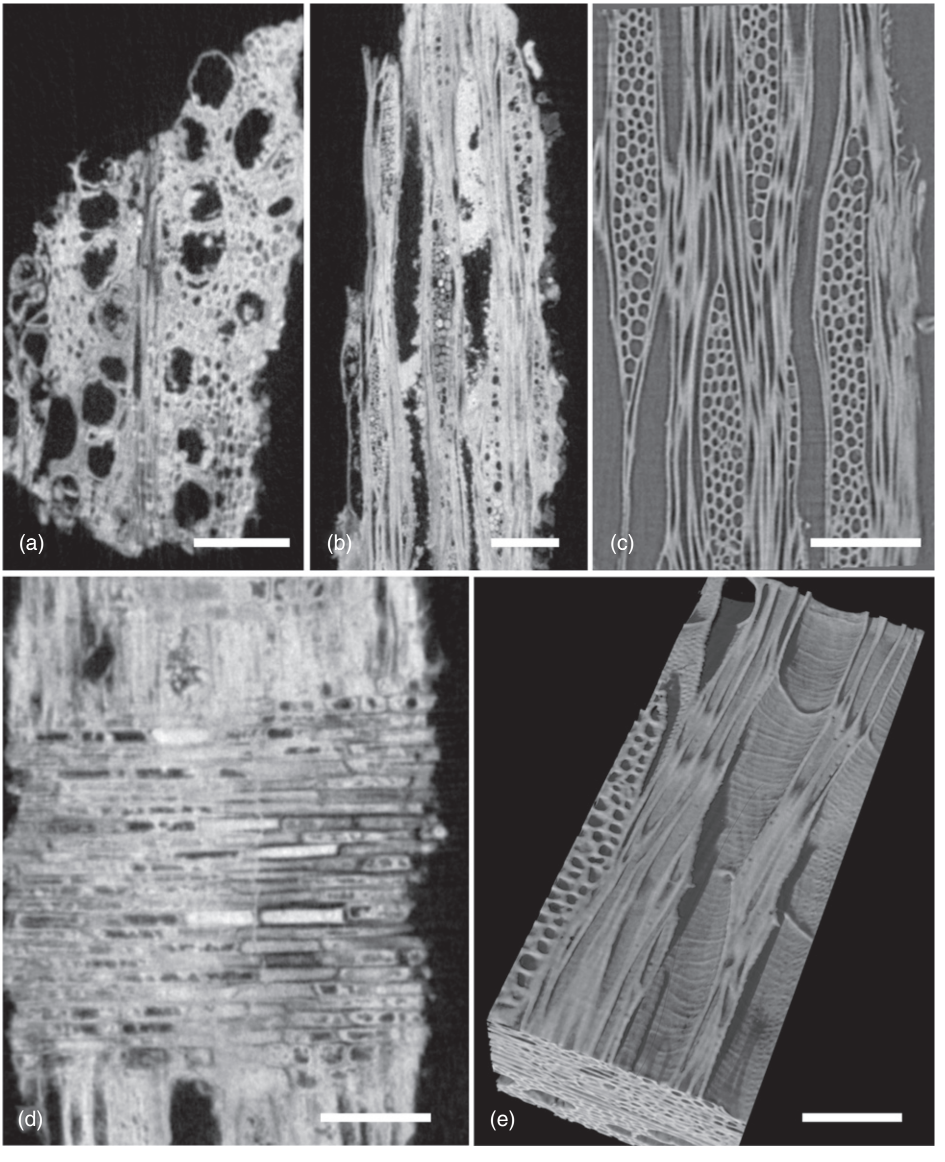

Figure 4 *Virtual sections and 3D representations of the modern* Prunus avium *sample and the archaeological wood preserved in the corrosion layer of the sheath of a Merovingian long* seax*: (a) transverse and (b) tangential sections of the archaeological sample; (c) a tangential section of the modern* Prunus *sample; (d) a radial section of the archaeological sample, showing a heterogeneous ray; (e) a 3D view of the modern* Prunus *sample with spiral thickenings on the vessel walls. The scale bars represent 100 µm.*

thickenings are clearly visible (Fig. 4 (e)). Furthermore, the size and arrangement of the pits between vessel elements can be quantified (Fig. 4 (e)). It should be noted that visualization of the volume in 3D while reslicing in different directions and tuning the position and intensity of the light source offers a powerful tool for the identification of diagnostic features.

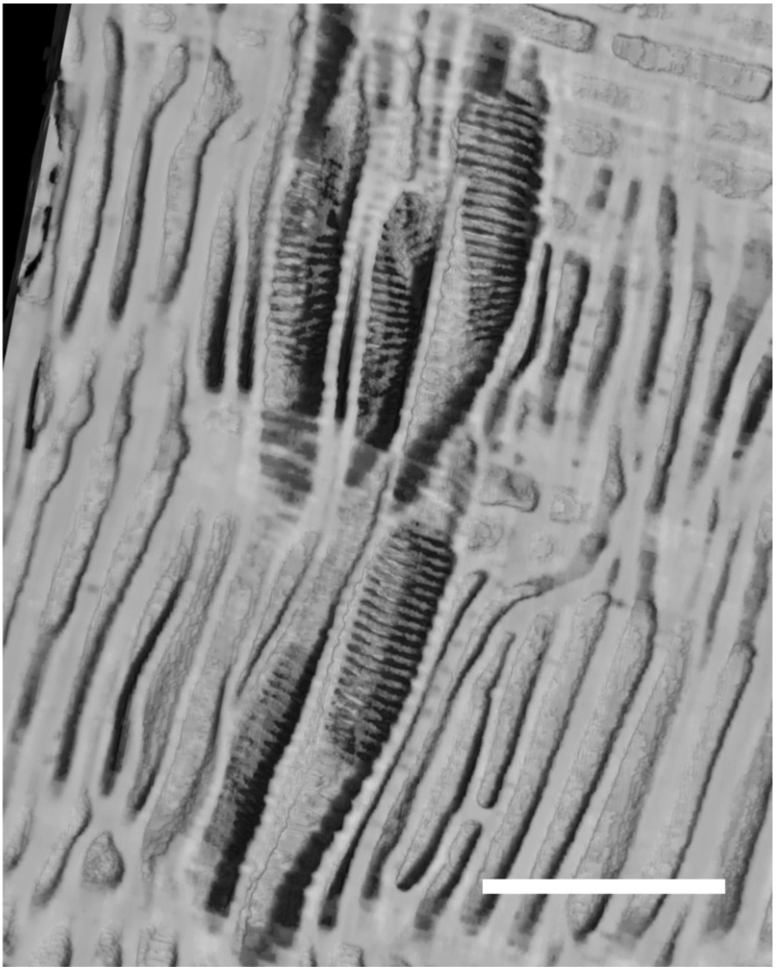

Figure 5 *A 3D view of scalariform perforation plates on the modern* Ilex aquifolium *sample. The scale bar represents 80 µm.*

Interference of X-rays with minerals/metals

Visualization of the archaeological wood samples under study is accompanied by a series of imaging artefacts. Apart from the standard workflow in tomography, specific issues need to be addressed for these samples. One of the more complex phenomena that arises, especially at high resolution, is the so-called phase contrast effect (Wilkins *et al.* 1996). Conventional tomography is based on the attenuation of X-rays along straight lines through an object. However, not only the amplitude of the X-ray beam changes but also its phase, due to differences in refractive index inside the object. This causes very small angular deviations of the X-rays and results in visible image contrast if the resolving power of the scanner is sufficient. Although this effect can be exploited to enhance visualization in some cases (Boone *et al.* 2009), reconstructing such mixed projections, a combination of attenuation as well as phase contrast, with conventional algorithms

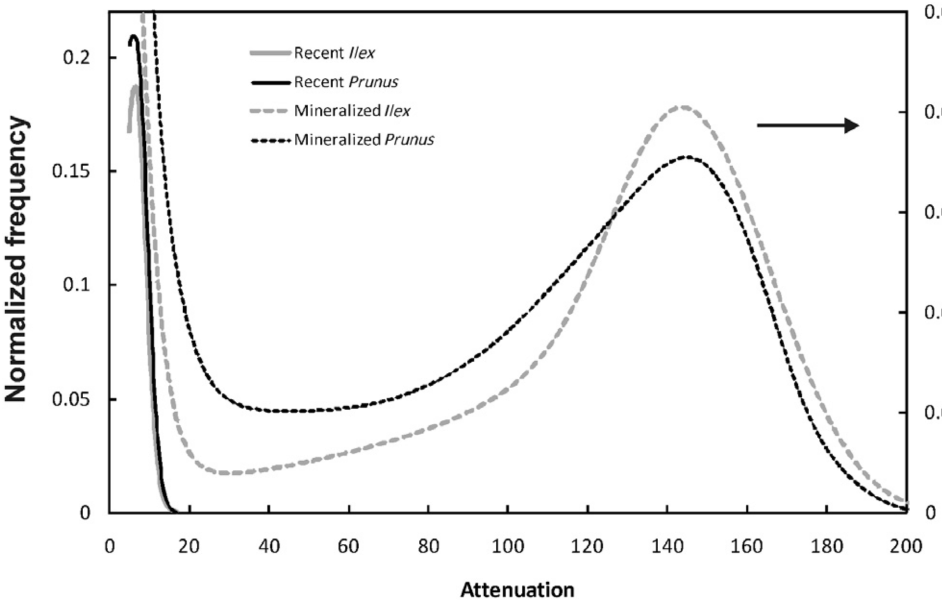

Figure 6 *Attenuation of both archaeological and modern wood samples after scanning and reconstruction, using exactly the same experimental set-up and software parameters (left Y-axis, modern wood; right Y-axis, mineralized wood).*

results in artefacts in the reconstructions. Therefore, BAC phase contrast filtering is applied, using a dedicated software algorithm (De Witte *et al.* 2010).

However, the main challenge with this kind of specimen is the corrosion-induced preservation, causing the filling of lumina and partial or complete substitution of the original organic matrix, which has a rather low attenuation, with mineral/metallic compounds with a much higher attenuation. In such cases, the use of a polychromatic source in combination with the energy dependency of X-ray absorption causes beam hardening. As such, the reconstructed images can show a gradient in grey values and additional smearing. Both hardware and software measures are used to cope with this phenomenon. Prior filtering of the X-ray beam suppresses the lower-energy part of the spectrum. This is to reduce problems caused by the polychromaticity of the detected spectrum. Additional software-based correction was performed to deal with the remaining artefacts (Vlassenbroeck *et al.* 2007).

As mentioned before, the filling of lumina, coating of cell walls or replacement of the original organic matrix by metal oxides and/or salts results in an altered attenuation of the material. Consequently, this shift in attenuation can be considered as a measure for the mineral content of the examined fragments. Therefore, all samples were scanned and reconstructed using exactly the same experimental set-up as well as software parameters for reconstruction. Volumes were segmented from air by straightforward manual thresholding and the normalized histogram of the attenuation coefficients was calculated (Fig. 6). Apparently, the sample identified as *Ilex aquifolium* has a slightly higher mineral content compared to the sample identified as *Prunus* sp., yet it is important to stress that this is merely a qualitative rather than a quantitative measure. Both modern wood samples lie on the far left of the attenuation scale, which indicates one of the main problems when there is a mixture of organic and metallic materials. The distribution of the archaeological samples clearly overlaps on the left-hand side with the scans of modern wood, and thus only when little organic material remains can it be obscured.

DISCUSSION

The obtained X-ray sub-micron CT scans on modern wood samples clearly demonstrate the power of this technique for wood anatomical studies and taxonomic identification. On scans of both *Ilex aquifolium* and *Prunus* sp., diagnostic features for species identification could be observed and quantified. Furthermore, the 3D model of the scanned samples makes a meticulous preparation superfluous, since the Tg, R and Tv planes can all be accessed on virtual slices. Together with the required minimal sample size (~1 mm^3), the non-destructive nature of X-ray sub-micron CT makes this technique most appropriate for wood identification on valuable wooden cultural heritage artefacts (e.g., Mizuno *et al.* 2010). However, the extremely small sample size can have some disadvantages for the study of wood anatomical features. For instance, for (semi-)ring-porous species, the obtained samples are potentially too small for the arrangement of the vessels to be observed. Nevertheless, this can be tackled easily by scanning more voluminous samples.

In the specific case where wood fragments are preserved in the corrosion of archaeological metal objects, some diagnostic anatomical features may remain undetected in the resulting scan. Due to the interaction of the X-ray beam with metallic inclusions in the sample, some degree of detail is lost, although some of these features could have been (partially) degraded before completion of the preservation process and, as such, absent on the samples that were X-ray scanned. Wood anatomical features such as perforation plates and spiral thickenings are indeed visible on the modern wood samples, but are very hard to detect on the mineralized wood samples. For instance, on modern wood samples, scalariform perforation plates can be identified and the number of bars counted (Fig. 5). This feature was only rarely observed on the sub-micron CT scans of archaeological wood. Nevertheless, this type of perforation plate was observed on wood samples from the same metal object with conventional reflected light microscopy. The same applies to the presence of spiral thickenings, where we obtain a good visualization of these helical ridges on the secondary cell wall of the vessels in modern wood samples. As such, the scans of the modern *Ilex aquifolium* and *Prunus avium* samples provide evidence that spiral thickenings can indeed be visualized by X-ray sub-micron CT. Finally, the specificity of the corrosion process probably has led to the deposition of a metallic coating layer on the helical ridges, as such making them very difficult to see. Such deposition layers are visible in Figures 4 (a) and 4 (b).

The applied *a priori* X-ray beam filtering and software-based corrections in order to diminish artefacts are always a trade-off between noise reduction and partial information removal. However, this kind of beam-hardening correction does not work perfectly when dealing with strongly attenuating inclusions such as metals or minerals. The specificity of the material under study as such leads to non-optimal beam-hardening corrections. The practical implementation of newer simultaneous algebraic reconstruction algorithms developed for graphical processing units (GPU; De Witte *et al.* 2010) are a major advantage, coping with some of the above-mentioned problems that will improve the quality of reconstruction in the near future.

The taxonomic identification of the wood remains associated with the archaeological metal object immediately demonstrates the value and importance of wood identification for the interpretation of the archaeological record. Both identified taxa, *Prunus* sp. and *Ilex aquifolium*, provide wood with a distinctive colouring, respectively reddish and white, that is also very hard and that has excellent properties for wood carving and making handles. However, *Ilex* in particular is often associated with ritual contexts (Gale and Cutler 2000), and the record of archaeological artefacts made of *Ilex* is actually rare. More information on the selection of wood taxa for weaponry in Merovingian grave contexts will become available when a representative sample of this type of grave goods has been examined.

CONCLUSIONS

X-ray sub-micron CT has proven to be a promising new technique for the study of recent wood (Van den Bulcke *et al.* 2009) and ancient wooden objects (Mizuno *et al*. 2010), as well as recent and archaeological charcoal (Bird *et al*. 2008). The results presented here demonstrate that X-ray sub-micron CT can also be useful for the study of wood preserved in the corrosion layer of metal objects recovered during archaeological excavations. In particular, the very small sample size and the ability to create virtual sections without having to break or cut the sample are great advantages of X-ray sub-micron CT over conventional techniques such as light microscopy and SEM. Theoretically, it should be possible to visualize imprints of other organic materials, such as leather and textiles, in corrosion. However, at this point it is rather premature to point out this possibility without any feasibility tests.

It should be clear that taxonomic identification of mineralized wood fragments pushes X-ray sub-micron CT to its limits. In order to visualize all diagnostic, wood anatomical features, sub-micron resolution is required. To achieve this level of detail, using a laboratory-based set-up with a polychromatic X-ray source, phase contrast filtering is required. Furthermore, the inclusion of metallic components in the organic matrix makes X-ray sub-micron CT scanning even more complex, and requires additional filtering while scanning and correction algorithms in the reconstruction of individual scans. Even with this state-of-the art set-up, a number of wood anatomical features remain very hard to visualize, particularly those that seem to be related to the mineral content of the studied material. However, it may become possible to tackle these problems in the future, as this technique is developed further.

Also, for the first time, the mineral content of archaeological wood preserved through corrosion was appraised by using X-ray sub-micron CT. Although the approach presented here should be considered as a qualitative assessment, it provides information that might be useful in selecting the most appropriate strategy for the preventative (i.e., the environmental conditions under which objects should be stored) and structural conservation of archaeological objects.

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