Investigation of Wood Materials by Combined Application of X-ray and Neutron Imaging Techniques*

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Conservation of cultural heritage is extremely important not only from a cultural point of view, but also from a practical one. It is our duty to pass on to future generations the cultural heritage left to us by our ancestors. Wood is one of the most common materials used to generate works of art which are in a state of constant change and/or deterioration. In order to optimize the knowledge of artworks together with their conservation, it is necessary to use the most advanced scientific and technological tools. In the following paper, we will show the tomographic results which can be achieved by application of complementary techniques based on the combined use of X-ray and neutron radiography as structural probes.

X-ray and neutron imaging [1-9] techniques are very important in dealing with the non-destructive investigation and conservation of cultural heritage finds, e.g. finds from shipwrecks, including parts of the ship itself [10]. Objects made from a variety of different materials might be hidden by thick heavy layers of calcareous concretions, to the point that it is impossible to gain the necessary information without seriously damaging the find. As a matter of fact, in the vast majority of cases, X-rays and neutrons may give complementary information [11]. This is certainly the case for wood samples (for example parts of sunken ships).

In what follows, we shall highlight how X-ray and neutron based techniques may be used in a complementary way to investigate the macroscopic structure of woods and to help conserve them. For example, the use of neutrons, a combination of neutron radiography (NR) and/or of neutron tomography (NT) enables the investigation of the macroscopic structure of degraded woods before and after any consolidation and/or restoration work.

In addition, neutrons are particularly sensitive to hydrogen, and therefore are well suited to investigate the presence of organic matter and its distribution in archaeological finds. On the other hand neutrons are highly penetrating probes and so are suitable for the investigation of large artworks. Compared to neutrons, X-rays have lower penetration depth (at least at commonly used energies below 150 keV) in metals and rocks, but small samples or fragments of artefacts can be imaged by X-ray photons with the additional advantage, as we shall see, of higher spatial resolution. In examining degraded woods, later in the paper, we shall discuss these complementary features. Clearly, the combined use of neutron based and X-ray based techniques, seems to be well suited for the use of cultural heritage investigations.

| X-ray and Neutron Imaging |

Over the last decade X-ray and neutron imaging methods have significantly gained importance. One of the reasons is the rapidly developing field of digital image recording and processing, which enables the computation of tomographic reconstructions from high resolution images on a reasonable timescale [12-17]. The development of new detectors with better signal-to-noise characteristics and faster read-out electronics have allowed some of the limits of conventional radiography and tomography concerning spatial and time resolution to be overcome. Furthermore, X-ray and neutron imaging are non-destructive and they are suitable for in-situ and in-operando measurements, what is of special importance in many fields of science [18-25]. Another reason are the recently introduced new image modes [26-32], which allow investigation of, e.g., crystalline structures [33-38] or magnetic materials [39-44], and that significantly extend the field of possible applications.

X-ray and neutron imaging methods are based on the detection of transmitted radiation through a defined medium (sample) by using position sensitive detectors [1]. The radiation beam is attenuated differently by the composition elements of the sample, giving contrasting variations in the recorded radiography image. For high resolution radiography purposes two kinds of radiation can be used: X-rays and neutrons.

The charge-free neutron interacts with the core of the atoms, while in contrast X-
rays interact with the charge distribution of the electron shells. Therefore the X-ray attenuation coefficients increase with the atomic number of the elements, i.e., with the number of electrons [1]. The interaction probability of neutrons with the atomic core depends on the coherent scattering length acoh, which does not show a systematic correlation with the atomic number of the element [3, 4]. As a consequence, the attenuation properties of the elements for neutrons show a non-systematic correlation with the atomic number as shown in Figure 1.

It should be pointed out that neutrons are very sensitive to some light elements like H, Li, B, for which X-rays do not provide a good contrast (low and similar atomic numbers). Additionally, neutrons easily penetrate thick layers of metals like Pb, Fe and Cu where standard X-ray imaging even with energies of several hundred keV would fail. In this context the two radiations appear to be complementary in the case of the non-destructive radiography investigations.

In the case of tomographic investigations, the sample is rotated around a defined axis and 2D projections are recorded under different rotation angles. The mathematical reconstruction of the matrix of the attenuation coefficients in the sample volume can be made using the collected set of projections. As an example, Figure 2 (see Figure 2) shows the neutron tomography image of a small object recovered from a find whose picture is shown on the left of the tomography reconstruction. It is clear that the information has been obtained without damaging the find thanks to the high penetrability of the neutrons. In this case it would have been more difficult to obtain the information using X-rays as a probe, due to the thickness of the calcareous concretion.

### Experimental and Materials

All the reported imaging experiments were performed at the Helmholtz-Zentrum Berlin für Materialien und Energie (Berlin, Germany). For neutron based techniques we used the cold neutron imaging instrument CONRAD [45]. The beam provides neutrons with wavelengths between 0.2 nm and 1.2 nm with a maximum at 0.35 nm. The point source beam geometry is determined by pinholes placed at a distance of 5 m in front of the sample. The detector system was based on a CCD camera integrated in a light tight box with a scintillator screen (20 × 20 cm²) and a mirror projecting the image from the scintillator via a lens system on the CCD chip. The 16 bit CCD camera used (Andor DV436N-BV), had a Peltier cooled chip with 2048 × 2048 pixels. The beam size of 10 × 10 cm² permitted the investigation, when necessary, of more than one sample at a time. The number of the radiographic projections was 300 over 180 degrees. The achieved spatial resolution varied, depending on the sample size from 60 µm to 100 mm. Exposure times between 2 hours and 5 hours were used per tomography experiment.

For the X-ray based technique we used an X-ray CT scanner based on a micro-focus tube (Hamamatsu, L8121-03) at 60 kV, 166 µA in small spot mode (focus: 7 µm). The images were recorded by flat panel sensor (Hamamatsu, C7942SK-05) with a pixel size of 50 µm where different magnification ratios (distances between source, sample and detector) were used providing a spatial resolution between 10 µm and 50 µm in the reconstructed tomographic data. The exposure time per X-ray tomography experiment varied between 2 hours and 4 hours depending on the sample size and applied magnification. The volumetric reconstruction was made using Octopus software and the visualization and data analysis by VGStudioMax software. The tomography slices were reconstructed in absolute attenuation coefficients so a comparison could be made of the experiments carried out on different samples and at different times.

Four samples of Pinus Silvestris, indicated as PB, PD, PE and PF, artificially degraded by brown rot, were consolidated by means of different treatments. Samples PB and PE were consolidated at 45 °C by immersion in a 7% (w/w) solution of Klucel in acetone and in ethanol, respectively. Samples PD and PF were consolidated at 22 °C by immersion in a 30% (w/w) solution of colophony in acetone and ethanol, respectively. Samples were impregnated for 8 days. During this time the impregnating solutions were replaced three times with fresh ones. At the end of the impreg-
nation, the samples were dried in a stove for about 20 days at 45 °C.

Results

As pointed out earlier, in examining items of archaeological interest X-ray and neutron based techniques may give interesting complementary information, in particular when dealing with wood samples. As an example, in Figure 3 we show the images of an internal slice of an artificially degraded sample of pine wood (Figure 3a), and of samples of the same wood consolidated by treatment with a 30% w/w solution of colophony in ethanol (Figure 3b) or with a 7% solution of Klucell in ethanol (Figure 3c). X-ray tomography (XT, Figures 3a-3c) and neutron tomography (NT, Figures 3d-3f) images were obtained on the same samples. The degradation performed by the brown rot is clearly visible in the black portion of the slices.

X-ray images (a, b) show a small portion of the slice where wood powder resulting from the degradation has been accumulating. The same regions are barely visible in the corresponding neutron images (d, e). Both XT and NT images show the layered structure of the wood with the distinct region of the earlywood (higher attenuation coefficient, light color) and of the latewood (lower attenuation coefficient, dark color), respectively. Natural voids are also clearly visible in the X-ray image.

It can be noticed that the XT images show higher space resolution, when compared with NT. On the other hand, the NT images are more sensitive to the presence of the consolidating medium (colophony or Klucell) with the result that the image is less sharp, but contains more information as far as the distribution of the consolidating medium is concerned [10]. This is clearly shown in the histograms (g, h) of the images (c, f) shown next to the images.

The complex structure of the NT histograms is a clear indication of the variety of effects which are detected by the NT image. We may expect that, in the simplest case, one would need three Gaussians to fit the histograms: one Gaussian at very low values of the attenuation coefficient, corresponding to the voids (macro and micro voids, in general), another one describing the regions dominated by the earlywood and a third one corresponding to the latewood. Eventually one more Gaussian might describe the region in between the early and the latewood. This is exactly the situation found in the case of XT.

If we now consider the NT histogram, we still find a Gaussian at low attenuation coefficient describing the contribution of the voids. The remaining portion of the histogram cannot be reproduced with only three Gaussians due to the fact that neutrons are quite sensitive to hydrogen and therefore the histograms are sensitive to the distribution of the consolidating medium inside the wood.

Evidently X-rays can clearly differentiate between the main components, but are not able to distinguish equally well the differences in the attenuation factor induced by the consolidating medium, made
of light chemical elements. On the other hand, NT images have richer details and describe the difference in the attenuation coefficients induced by the consolidating medium; in doing so, there is less information regarding the contrast between the different regions.

For further support of this deduction, histograms for XT and NT were generated for different portions of the whole slice. Of course, the choice of the different portions was made in an attempt to attribute the single Gaussians to well-defined portions of the wood slice. In particular, histograms have been generated for the latewood areas and for the earlywood areas in different portions of the slice: far from the side surface and close to the side surface (where the consolidating medium should reach higher concentrations).

Partial histograms are drawn in Figure 4. As far as the XT results are concerned, the two Gaussians identifying the latewood contribution near the centre of the slice (Figure 4b) and near the surface, are almost identical, while the corresponding earlywood contributions show a minor change in the peak position (Figure 4a).

The situation is quite different for the NT data: in this case, the contributions far from the surface and near the surface are significantly different (Figures 4c and 4d). As expected, both for earlywood and for latewood, the peak positions of the contributions for histograms which refer to portions close to the surface correspond to higher attenuation coefficients.

The results so far obtained for Pinus Silvestris were also found for other woods (fir, chestnut) [10]. In all cases, we found that, while for XT the histograms of the same material in different regions of the slice change only slightly, it is not so for the NT measurements. In these, the histograms of the earlywood near the centre of the slice differ from the histograms of the same wood near the edge of the slices. This is the same for latewood. This is of course due to the fact that neutrons are quite sensitive to hydrogen and hence may distinguish between the two zones. This means that the combined use of XT and NT is important when attempting to assess how well the consolidating material penetrates into the wood, a key point in selecting the proper consolidation medium.

To further highlight the importance of the synergic use of XT and NT, in Figure 5 we show more results obtained for the samples discussed so far. We generated histograms for a series of slices with about 0.75 mm distance between each other, but excluding the operation, the portion of the slices close to the surface (approx. 4 mm) and the region where the wood powder accumulated. The reason for not considering, in this comparison, the area close to the surface is that this area is the one which is more affected by the accumulation of the consolidating medium, and therefore the one to which neutrons are more sensitive. By not considering this area in obtaining the histograms, the fit should essentially reflect the basic structural features of the wood itself, even if some changes are
produced by the diffusion of the consolidating medium.

To fit each histogram we then used three Gaussians (corresponding to the earlywood and to the latewood, i.e., the dominating contributions, and to the intermediate region). In Figure 5 we show the peak positions obtained from the histograms obtained from the XT data for sample PB (illustration a) and PD (illustration b). Illustrations c) and d) show the peak positions obtained from the histograms obtained from the NT data for the same samples.

As expected, the trend shown for each peak does not seem to be heavily dependent on the probe (X-ray or neutron), as we did not use the portion which are more affected by the consolidating medium, although some differences can be observed. In addition, Figure 5 clearly shows that the portion of wood which is more sensitive to the diffusion of the consolidating medium, is the one corresponding to the earlywood with larger pores and a less compact structure, features which make the earlywood more penetrable.

The situation is expected to be different if greatly degraded samples are compared. This is the case for samples PE and PF, whose histograms for different sections are shown in Figure 6. Again illustrations a) and b) show histograms obtained from the XT data, sample PE (illustration a) and PF (illustration b), respectively. The corresponding histograms obtained from the NT data for the same samples, are shown in illustrations c) and d), respectively. Trends for XT and NT histograms are not quite as similar as those shown in Figure 5, and, as expected, the neutrons show a more systematic variation with the distance from the surface.

| Acknowledgements |
| This research project has been supported by the Framework Agreement between HZB, Technische Universität Berlin, Germany and Università di Palermo, Italy, by Fondi Ateneo of the Università degli Studi di Palermo and by the European Commission through the ‘Research Infrastructures’ action of the ‘Capacities’ Programme; Contract n.: CPSA_INFRA-2008-1.1.1 number 226507-NMI3. |

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| Conclusion |
| The role of different probes (X-rays and neutrons) in investigating the structure of items of archaeological interest has been highlighted by showing results obtained for wood, a rather common material in items of archaeological interest. The fundamental role of neutrons in investigating the distribution of the consolidating medium inside the wood sample together with the good spatial resolution shown by X-rays (even in heavily consolidated samples where the neutron image is not equally sharp), suggest that, whenever possible, both techniques should be used. This would mean benefiting from their complementary features when used in the field of cultural heritage. |

| Abstract |
| Charakterisierung von Holz mit komplementären Röntgen- und Neutronenbildungsverfahren. Die Konservierung von Kulturgütern ist von großer Bedeutung nicht nur in kultureller Hinsicht, sondern auch unter praktischen Gesichtspunkten. Wir müssen uns der Aufgabe stellen, unseren Nachfahnen das kulturelle Netz weiterzugeben, das uns von unseren Ahnen überliefert wurde. Das Holz, eines der gebrauchlichsten Materialien für Kunstwerke, ist einem ständigen Prozess unterworfen, der zu Veränderungen und/oder seinem Zerfall führt. Um die Kenntnis von historisch-künstlerischen Fundstücken zu optimieren und ihre Konservierung zu ermöglichen, ist die Anwendung der besten wissenschaftlichen sowie technischen Technologien unerlässlich. In den Folgenden zeigen wir einige der computertomographischen Ergebnisse, die durch Anwendung komplementärer Messtechniken bei kombinierter Verwendung von Röntgenstrahlen und Neutronen erzielt werden können. |
TOMOGRAPHY OF WOOD

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DOI 10.3139/120.110553
Materials Testing 56 (2014), 3, page 224-229 © Carl Hanser Verlag GmbH & Co. KG
ISSN 0025-5300

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