Preparation of wood with pulsed UV-laser ablation for characterisation of the wood structure

Jimmy Johansson, Dick Sandberg
Växjö University, Technology and Design
SE-351 95 VÄXJÖ, Sweden

Abstract
In contrast to mechanical techniques involving cutting with a knife, cutting with a microtome or grinding, pulsed UV-laser ablation is an irradiation technique where a so-called Excimer laser is used, and the process is adiabatic, which means that no or a very little chemical change occurs in the material being processed. Cross sections of samples of Scots pine and Norway spruce which had been exposed outdoors have been prepared with the help of an Excimer laser (wavelength 248 nm) for study in a microscope.

UV-laser ablation has been found to be a suitable method of preparation for wood when it is desired to obtain thin sections with little influence on the material, and particularly when dealing with brittle materials, e.g. archaeological wood, biologically-attacked wood or, as in this case, wood which has been exposed outdoors.

1 Introduction
Microtoming and cutting by hand with a razor blade are today the two main techniques for the preparation of samples of wood material for microscopy studies, but there are two general problems with these methods. Firstly, it is usually necessary to soften the samples by wetting and in some cases by heating. Secondly, the specimens are affected physically by the mechanical force applied. The cut area is often also limited. In neither case can it be guaranteed that the method of preparation does not generate artefacts in the material. This is particularly true of brittle wood material.

It has long been known that when wood is exposed to light and especially to UV-radiation (wavelength \(10^{-8} - 10^{-7}\) m) the energy taken up by the wood surface will initiate chemical reactions which change the properties of the wood. One of the most obvious signs of these reactions is the grey discoloration which almost always develops on untreated wood surfaces exposed outdoors. The phenomenon is usually referred to as photochemical degradation [1].

Fengel and Wegner [2] have described the structural changes which occur in the cell wall as a result of UV-irradiation. All the wood components suffer changes, but the chemical structure of lignin means that it is an especially strong absorber of radiation with this wavelength, so that lignin is rapidly degraded. Of the total absorption coefficient, lignin is responsible for 80–95 %, carbohydrates for 5–20 % and extractive substances for about 2 % [3]. Pure cellulose is a poor absorbent. Irradiation with UV-radiation thus leads to a degradation of the cell-wall which is linked to the lignin concentration in the cell-wall, and this leads to a delignified wood material. The degradation is also dependent on a number of environmental factors such as moisture, temperature and the availability of oxygen. The formation of free radicals is an important stage in the degradation process.

If wood is exposed to outdoor conditions without the protection of some kind of surface treatment, e.g. a paint layer, considerable changes occur with time in the surface layer (to a depth of up to 2 mm from the surface) as a consequence of photochemical or mechanical degradation and, in certain cases, biodegradation. The wood material in this surface layer becomes very brittle and easily disintegrates on processing. Investigations have shown that the tensile strength of a thin external layer exposed to UV-irradiation decreases in a few months to less than 20 % of the original tensile strength [4, 5].

In the preparation of samples in order to study the weakened surface layer, e.g. through microscopy, it is difficult to open the structure without creating artefacts. This applies particularly in the study of end-grain wood sections.

In this investigation UV-laser ablation has been tested for as a method of preparation of the brittle surface of weathered samples of wood for microscopy studies.
2 UV-laser ablation for characterisation of the wood structure

The first trials in which the Excimer laser was used for the so-called UV-laser ablation of wood surfaces were carried out more than ten years ago [6]. The original purpose of using this technique was to remove the ca. 0.1 mm thick surface layer of crushed fibres created by mechanical treatment such as sawing and planing, Figure 1 left. In this way, the undamaged structure of the wood is revealed and the wood can be prepared for surface treatment or impregnating operations. A surface containing crushed fibres also moves in an uncontrolled manner, leading to so-called fibre-rising which is a major problem in the surface treatment of wood. This problem can be eliminated if the crushed fibre layer can be removed. In UV-laser ablation, the photons break chemical bonds in the nano-structure. This leads to a local pressure which accelerates the expulsion of particles and gases from the surface with a speed of up to 8 times the speed of sound in air. When a material is ablated, two different directions of the laser beam – perpendicular to or parallel to the ablated surface – can be used. The main difference between the results of laser ablation perpendicular to and parallel to the surface is that the former gives a somewhat undulated surface, due to differences in density. If the UV-laser ablation is extended after the crushed layer of fibres has been removed, a "washboard" structure appears which follows the density of the annual rings, since it takes a longer time to remove the high-density latewood than the earlywood. The surface will then be microscopically smooth but in a macroscopically way structured according to the wood density. The resulting texture can be used to determine the density in a single annual ring, Figure 1 right.

To obtain a smooth surface, the ablation shall take place parallel to the surface. When ablated parallel to the surface, the surface become both micro- and macroscopically smooth, but this direction of ablation gives grooves in the surface. These grooves are caused by particles of high speed that are accelerated from the surface or are a result of the profile of the laser beam or the mask. This phenomenon is discussed in [8]. In the case of ablation parallel to the surface, the laser beam can also cut deep into the wood material and thus lead to two ablated surfaces.

![Fig. 1](image.png)

**Fig. 1** Left: Planed radial section of pine with crushed fibres, 213 x magnification. The wood surface in the lower part of the picture has been ablated with UV-laser, which has removed most of the fibre layer which was compressed during planing [7]. Right: An edge of an end-grain/flat-grain section of wood at 24.6 x magnification which has been partly UV-laser ablated, perpendicular to the flat-grain section. The latewood hills, and the earlywood valleys, due to the fact that less dense material is easier to ablate than more dense material, are easily seen [6].
3 Sample preparation

In this test, fully quarter-sawn and plain-sawn boards of pine (Pinus sylvestris L.) and spruce (Picea abies Karst.) have been used. The specimens were weathered for 61 months and, after weathering, all the samples were conditioned at a temperature of 20°C and a relative humidity of 65%. Specimens with an approximate size of 10 x 10 x 10 mm³ were sawn and the cross sections of these small specimens were prepared with the help of UV-laser ablation with the laser beam parallel to the cross section. An excited dimmer laser (Ecimer laser) Questek 2520 νβ type was used and the wavelength was 248 nm, the energy 500 mJ/cm² and the frequency 5 Hz. The wood surfaces were investigated by environmental scanning electron microscopy (ESEM).

4 Microscopy studies

Figure 2 shows a characteristic pattern of degradation of radial sections with very brittle wood material. To the left, we can see a typical crack in the annual ring border. In this region, the change in density is very abrupt; in latwood, the density is about 900 kg/m³ and in earlywood only 300 kg/m³. To the right in Figure 2, we see latwood that is left when the low density earlywood has been eroded. On a macro-level these peaks of latwood make the surface wash-board-like.

Figure 3 shows ablated cross-sections of weathered tangential sections. The laser beam has cut the very brittle weathered tangential layer without disturbing the degraded wood structure. Since it has not been necessary to apply any moisture to the wood in the preparation, the crushed cell layer which was caused by planing of the layer prior to the outdoor exposure has been retained, Figure 3(c). All sections shown in Figure 2 and 3 are complicated to prepare without artefacts for microscopy studies with the help of traditional techniques. The latwood peak shown in the right-hand picture in Figure 2 is only 0.15 mm in width and extremely brittle.

By using the laser ablation technique in the preparation of samples for microscopy investigations, two important sources of undesirable artefacts are excluded, viz. the softening operation and the application of a mechanical force to the sample. A possible weak boundary layer, caused by the laser ablation, is of minor importance if the purpose is to investigate a surface under ordinary microscopy.

Fig. 2 Cross-section view of a weathered radial section. To the left: crack at the annual ring border at 175 times magnification. To the right: latwood strip in 265 times magnification, where the surrounding earlywood has eroded.
5 Conclusion

UV-laser ablation is a sample preparation technique for microscopy studies and is an especially suitable method of the preparation of wood when it is desired to obtain thin sections without artefacts and particularly when dealing with brittle materials, e.g. archaeological wood, biologically-attacked wood or, as in this case, wood which has been exposed outdoors. The main advantages are that the method gives a sample surface with a minimum of artefacts, it is a fast preparative technique and it requires relative little experience compared to the traditional preparation methods. The wood samples do not have to be moistened or dried, and the prepared area is not limited in size.

6 References