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INVESTIGATIONS OF SURFACE-TREATED WOOD BY A MICRO-INDENTATION APPROACH: A SHORT REVIEW AND A CASE STUDY

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Abstract: *Over the past 50 years, instrumented indentation, in which an indenter tip probes the specimen surface with a regulated force and displacement to characterize its mechanical properties, has evolved. With the development of atomic force microscopy, instrumented nanoindentation has been extensively applied, and instrumented indentation in the micro range (micro-indentation) has been somewhat overshadowed by nanoindentation. However, micro-indentation remains an extremely useful technique that has the potential to investigate various properties of surface-treated wood. In the first, overview-type part of the paper, instrumented micro-indentation is described and some examples of its possible applications in the field of wood science and technology are presented. To illustrate the importance of this experimental technique, some results of micro-indentation experiments carried out in our wood surface treatment laboratory are shown in the second part of the paper. The objects of the tests were untreated Scots pine wood and Scots pine wood with surface deposits of SiO_2 or ZnO. The differences between the mechanical properties of early and latewood could be clearly distinguished by the micro-indentation technique. On the other hand, the influence of ZnO and SiO_2 particles on mechanical properties as a function of indentation depth could not be detected by this technique.*

Key words: *Scots pine wood, ZnO and SiO_2 particles, instrumented micro-indentation, mechanical properties.*

1. Introduction

1.1. General Aspects

Recently, a new micro-scratch and micro-indentation depth-sensing device has been introduced in our laboratories. Because of this, an extensive literature review was conducted on the possible applications of instrumented micro-indentation / micro-scratching techniques in the field of wood science and technology. Some of the results of this

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review are presented in the first part of the paper. In addition, several preliminary tests were conducted with the new instrument. In the second part, the results of micro-indentation investigations of untreated pine wood and pine wood with surface deposits of SiO_2 and ZnO particles are presented.

1.2. Micro-indentation in the Research of Wood, Surface-Treated Wood, and Wood Coatings

Indentation is the essential phase of various non-cutting methods for determining the hardness of a material. The Brinell hardness test uses a small ball of steel or tungsten carbide to cause the plastic deformation of a material [16]. The other hardness tests that are also used are the Vickers, Berkovich, Knoop, or Rockwell tests. These methods are distinguished by the material, size, and shape of the indenter's tip [4]. The result of a hardness measurement is a single hardness value calculated from the depth of penetration of the indenter into the specimen [30]. Since the hardness values are determined after the tip has been removed, the potential elastic deformation is not taken into account when measuring with a conventional standard method [14], and the hardness is also load-dependent [16]. In addition, the measured/calculated hardness is also affected by the depth of indentation as well as the force with which the tip is pressed into the material, which is known as the indentation size effect [19]. Further, when performing a small-scale indentation test, the problem of accurately measuring the defects caused by the tip of the indenter may arise [9].

The answer to the problem is the determination of the hardness of materials by instrumented indentation, alternatively called the depth-sensing indentation method [30]. Here, the applied force and movement of the indenter are simultaneously controlled and measured during the whole process of hardness measurement (and similarly during the scratching experiment). Forces of a few μ and depth in the nm range can be precisely controlled [4]. The instrumented indentation method also allows the determination of some other material properties, such as indentation modulus, and, most importantly, the optical measurements of the indentations are not a prerequisite [14]. The technique for determination of elastic modulus and hardness by instrumented indentation on a small scale was developed and applied by Oliver and Pharr [21].

Standard ISO 14577 [14] considers instrumented indentation tests and takes into account both plastic and elastic deformations, and specifies three ranges of instrumented indentation tests.

In addition, Part 1 of the ISO 14577 [14] describes the specific requirements for the nanoscale, but we do not repeat them here because this paper is an overview of micro-indentation testing applications. In the macro range, the indentation force is between 2 N and 30 kN; in the micro range it is less than 2 N and the displacement is greater than 0.2 μm; the nano range is defined by the displacement of the tip, which is less than 0.2 μm.

Figures 1 to 3 show the typical results of an experiment with depth-sensitive indentation.

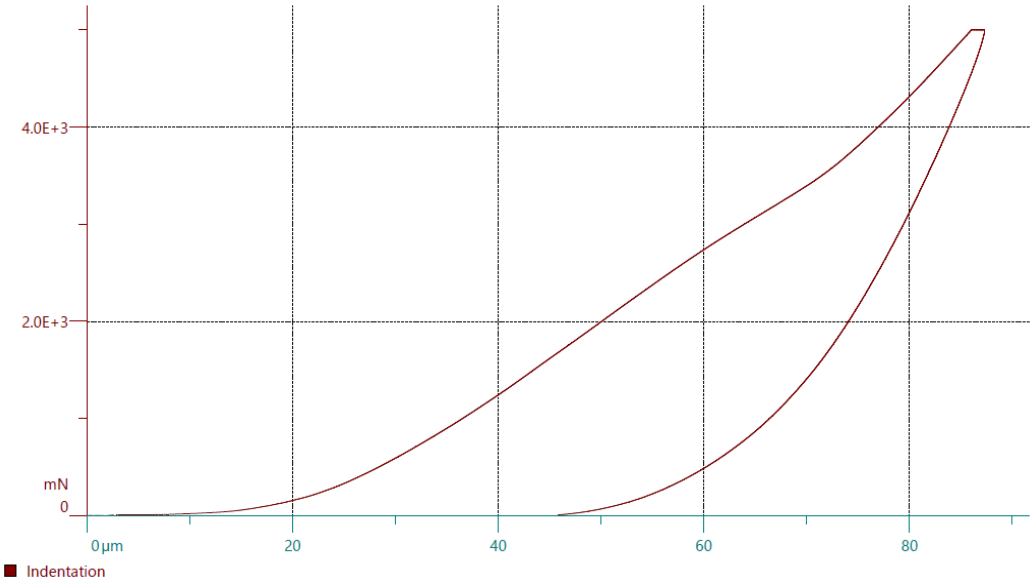


Fig 1. *A typical example of a force-depth curve (pine wood, semi-radial surface, earlywood)*

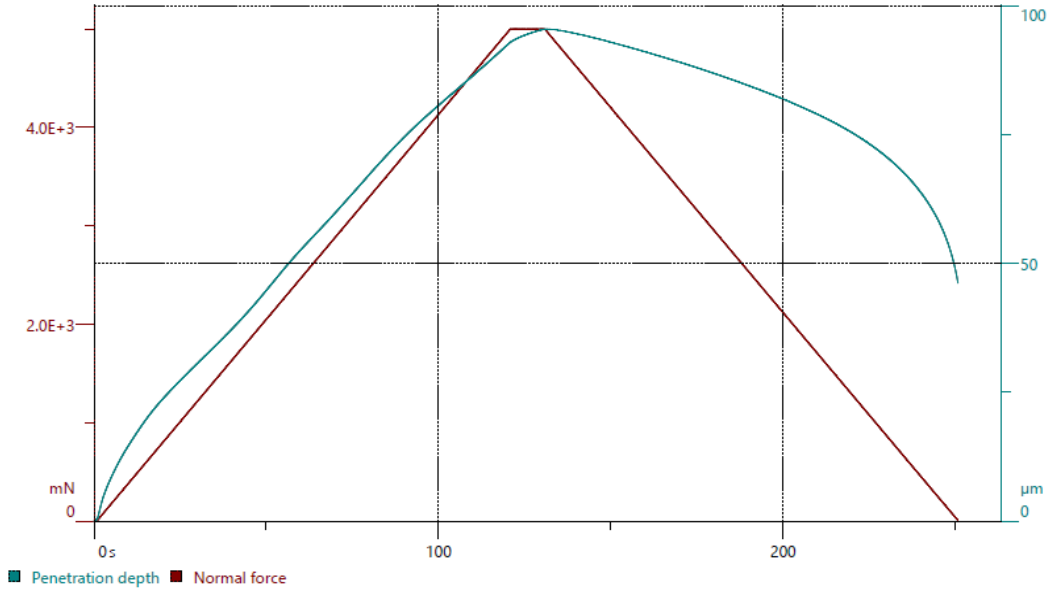


Fig 2. *The curve of force-time and indentation depth-time for the test shown in Figure 1*

In Figure 3, "h" is the displacement with respect to the initial surface and "P" is the loading force. The curves are characteristic of an elastic–plastic substance. h_m and P_m are the maximum depth and loading, respectively, and p_{ave} is the average contact pressure, where A_m is the real projected contact area

measured at h_m. P_m/A_m can be considered as the hardness (H). The slope of the upper part of the unloading curve during the initial phase of unloading defines the elastic unloading stiffness, S = dP/dh. At complete unloading (zero load), the residual depth after complete unloading is h_r. The areas under the load curve and the unload curve are W_t - the total load work, and W_e, the work, released by elastic unloading. Thus, the area bounded by the unloading curve and the loading curve is $W_p = W_t - W_e$, i.e. the plastic work done by the indentation process [9].

As far as instrumented indentation is concerned, nanoindentation experiments are most frequently reported nowadays. This is the result of the development of atomic force microscopy. Nevertheless, instrumented micro-indentation has also proven very useful in the study of wood, surface-treated wood, wood coatings, wood composites and various related lignocellulose materials.

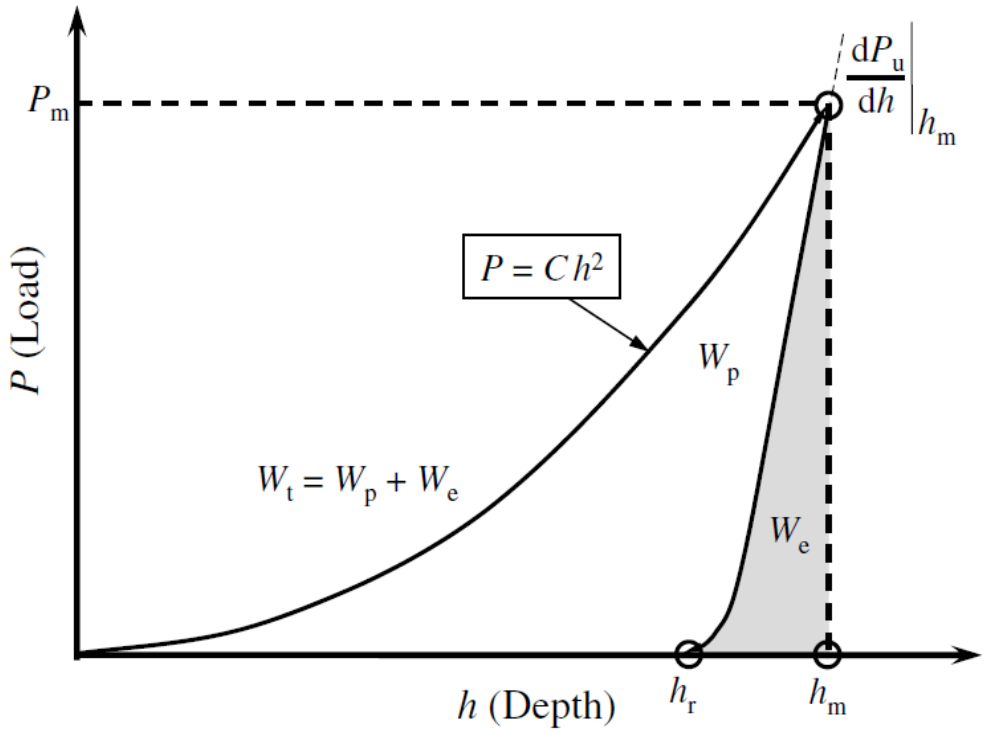


Fig. 3. *The explanation of some important parameters in the indentation force–displacement test [9]*

In this section, some selected examples of the application of depth-sensing micro-indentation testing on wood and wood-based materials are presented. Barletta [2] performed depth-sensing micro-indentation tests to evaluate the strength properties of wood. The correlations obtained were evaluated by a statistical method, and it was also found that the film on the wood can affect the consistency of the hardness values in the case of the Brinell method. Using micro-indentation, the hardness of surface densified wood was studied by Scharf et

al. [26]. They found that the interaction between the density profile and the test parameters caused high variability of the hardness values obtained. Nevertheless, they concluded that the performance of surface-densified wood can be evaluated based on the density profile. Fortino et al. [7] performed scratch tests with a microindenter on small-scale samples of wood impregnated with polyethylene glycol. They investigated the elastic and plastic deformation of the wood and determined the influence of wood structure on its performance. Lykidis et al. [18] found that instead of visual measurements of the indentation diameter, Brinell hardness values of wood can be determined by instrumented indentation measurements, resulting in a better correlation between hardness values and density. Finally, studies of wood pests are also extremely important in the field of wood science and technology, and it has been shown that micro-indentation can be a useful tool in the study of structures built by termites [20].

In what follows, some micro-indentation studies of coated wood and wood coatings are presented. Lu and Shinozaki [17] studied the shear strength between a coating and a substrate. Their micro-indentation studies concluded that the interface failure was most likely initiated on the basis of the mutual influence of the opening orthogonal stress and of the shear stress. The creep and elastic recovery of polyurethane films were studied using micro-indentation by Chagnon et al. [3]. Tendela and Kaufmann [29] combined a micro-indentation test with a radial speckle interferometer to investigate the adhesion of a coating. Hermann et al. [12] investigated the scratch behaviour, wear performance, and penetration depth of various UV-cured wood coatings using the microindenter. The investigation focused on the composition of a coating and as expected, hard and brittle coatings exhibited more defects at lower loads than ductile and soft coatings. UV curable self-healing acrylate coatings were investigated by Paquet et al. [24], who used the tester as well to produce controlled-depth scratches. Self-healing coatings with microcapsules were studied by Schreiner et al. [27], who determined the load required to destroy the capsules based on the force-indentation depth curves. Micro-indentation also found its application in the field of bio-based wood coatings [23]. The depth-sensing micro-indentation tests were used in the study of the influence of the integration of camphoric and isophthalic acids in UV-curing polyester resins for coatings on the mechanical performance of the UV-cured specimens. The potential of functionalized ceramic particles in laminate coatings to improve scratch resistance was investigated by Rusu et al. [25]. The tests were performed using a combined micro-scratch and indentation tester. They concluded that the use of functionalized ceramic particles reduced surface damage from scratches and improved the recovery properties of the surface layer.

1.3. Surface Treatment of Wood with Nanoparticles

There are several descriptions of the treatment of wood to obtain micro- or nanoparticle deposits of ZnO or SiO_2 on its surface. For example, Devi et al. [5] gave an overview of the preparation of

wood-polymer nanocomposites using ZnO nanoparticles and studied their properties, including mechanical properties, UV stability, water resistance, dimensional stability, and thermal stability. They also highlighted the uniform distribution of ZnO nanoparticles in the polymer matrix within the wood structure [22]. Low-temperature chemical growth processes have shown promise in the preparation of ZnO nanostructures, often using hexamethylenetetramine (HMTA). However, this study by Osman et al. [22] departs from this view by using inexpensive, commercially available zinc acetate dihydrate and sodium hydroxide precursors for hydrothermal synthesis. Characterization involved X-ray diffraction to determine crystal structure and grain size, while the morphology of the ZnO nanopowders was studied by scanning electron microscopy (SEM). The results confirmed the effectiveness of this hydrothermal method in obtaining high-quality ZnO nanoparticles [11]. The impregnation of SiO_2 depends on the deposition time which increases the quality of the composite material, and the thermal and mechanical properties are significantly improved. According to Han et al., impregnation of modified wood with itaconic acid (IA) significantly increases the cell wall thickness, reduces moisture absorption, and greatly improves the dimensional stability of the wood [31]. In another work, the preparation of a wood-SiO_2 composite by vacuum/pressure impregnation was described, improving the microstructure, the thermal/mechanical properties, and hydrophobicity. SEM, FTIR, XRD, TG, and water contact angle measurements were used to evaluate the microstructural and physicomechanical changes of the composite [31]. Several other similar research reports involving SiO_2 can be found in the literature: Zhang and co-authors improved various properties of heat-treated rubber wood by impregnation with a SiO_2 precursor [31], and Dong et al. [6] combined nano-SiO_2 treatment with furfurylisation.

2. Materials and Methods

2.1. Materials

Raw wood samples of pine (*Pinus sylvestris*) with dimensions of 5 mm x 20 mm x 50 mm were obtained from the Czech Republic. The surfaces examined were radial or semi-radial. The chemicals used to treat the specimens, namely zinc acetate dihydrate and sodium hydroxide, were obtained from Lach-Ner. Tetraethoxysilane (TEOS) and ammonium hydroxide (ACS reagent, 28-30% solution in water) were obtained from Thermo Scientific Chemicals. Ethanol was purchased from MERCI, s.r.o. All the chemicals were used without any additional purification.

2.2. Treatment of Wood to obtain ZnO and SiO_2 Particles on its Surfaces

First, the pine wood samples were subjected to thorough cleaning with deionized water to remove impurities. Then, the cleaned samples were dried in a drying chamber at a temperature of 60˚C.

The next phase involved the synthesis of zinc oxide (ZnO) nanoparticles following the methodology described in the work of Osman et al. [22]. For this purpose, the dry wood samples were immersed in a solution prepared at room temperature in a shaker for 5 hours. After

this immersion period, the specimens were dried again, this time at 60˚C overnight, followed by curing at 105˚C for 1 hour.

For the synthesis of silica (SiO_2), the procedure described by Adamopoulos et al. [1] was used. Then, the wood samples were impregnated with the SiO_2 solution following the same procedure as for ZnO.

Hereinafter, the control samples will be referred to as "Pine", the samples with ZnO particles as "PineZ", and those with SiO_2 as "PineS".

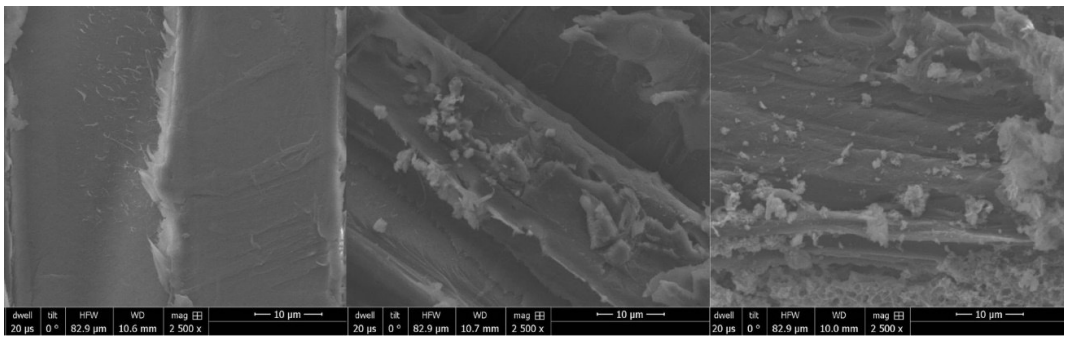
2.3. Micro-indentation Measurements

Single-loading micro-indentation tests to investigate the differences between early and late pine (Pine, PineS, and PineZ) wood were performed using the MCT3 microindenter (Anton Paar) with 100 CR6 steel balls (d = 6 mm). A single-load indentation was applied at the determined maximal load of 5 N. At least five indentations were made on each specimen. To investigate the mechanical properties of pine earlywood (Pine, PineS, and PineZ) as a function of indentation depth, the indentation depths were set to be 10, 20, 40, 80, and 120 µm and the number of indentations ranged from 5 to 11.

3. Results and Discussion

3.1. SEM Images

The surface morphology of the wood samples is visually depicted in Figure 4, with each image offering unique insights. In Figure 4a, the control wood sample shows a relatively clear and unaltered surface. From Figures 4b and 4c, it is noticeable that both the PineZ and PineS samples were modified with nanoparticles. This modification is clearly visible in the form of small crystalline clusters within the lumen cell walls on the longitudinal surface (Figure 4b). Finally, in Figure 4c, the (PineS) samples show a distinct modification, with the longitudinal surface of the lumen cell walls with crystalline deposits of SiO_2. It is noteworthy that all images have the same magnification range, allowing a direct and accurate comparison of the morphological surface changes due to the different treatments.



a. b. c.

Fig. 4. *Comparison of scanning electron microscope (SEM) micrographs of: a. untreated wood surface, b. modified wood surface with ZnO, and c. modified wood surface with SiO_2*

3.2. Micro-indentation into Early and Late Pine Wood

First of all, the standard deviations of the results presented in Table 1 are generally relatively high, indicating a relatively large dispersion of the values obtained. The reason for this is probably a high variability of wood properties, even within a single sample. Nevertheless, the hardness and stiffness of the latewood are greater than those of the earlywood. For example, in the untreated wood, the estimated Vickers hardness of the untreated pine latewood is actually about 4 times that of the earlywood. Similarly, the estimated Vickers hardness of the late pine wood (PineS and PineZ) is about 2 times and as much as 7 times higher, respectively. Similarly, the nanoindentation experiments of Golovin et al. [8] revealed up to 4 times higher hardness of late Scots pine wood compared to earlywood [2]. The mechanical properties of blue-stained and uninfected control Norway spruce wood were studied by Künniger and et al. [15]. They determined Brinell hardness using a standard Brinell hardness test method on tangential and radial surfaces but did not find clear differences. However, they reported a high scatter of the results, which also occurred in our experiments.

Table 1 also shows that the PineS and PineZ samples were less stiff and softer than the untreated wood samples, both in the case of the earlywood and the latewood. Explanation for this phenomenon has not been confirmed. However, it is anticipated that the sodium hydroxide used in the treatment procedure, as described in Adamopoulos et al. [1] and Han et al. [11], affected the material properties of the wood. Sodium hydroxide is well-known to swell cellulose at a certain concentration or even dissolve it at high concentrations. Soda hydrates cause dissolution and can penetrate the amorphous region of cellulose and break up the adjacent crystalline regions [28]. Therefore, treatment of wood with suitable chemicals can be used in the production of flexible wood such as flexible wood membranes. For example, a versatile, biodegradable 3D porous membrane from natural wood was prepared. Its high flexibility results from structural and chemical changes, including a wavy pattern formed by the removal of lignin/hemicelluloses [13].

Differences in mechanical properties between early and latewood (standard deviations in parentheses) Table 1

Sample	Earlywood					Latewood				
	N	E* [GPa]	E_{IT} [GPa]	H_{IT} [MPa]	HV_{IT} [Vickers]	N	E* [GPa]	E_{IT} [GPa]	H_{IT} [MPa]	HV_{IT} [Vickers]
Pine	12	0.88 (0.08)	0.66 (0.06)	49.50 (9.10)	5.05 (0.93)	10	2.10 (0.38)	1.57 (0.29)	250.03 (58.31)	25.49 (5.94)
PineS	9	0.63 (0.08)	0.55 (0.07)	23.62 (3.82)	2.41 (0.39)	9	1.11 (0.34)	1.01 (0.31)	43.89 (26.43)	4.47 (2.69)
PineZ	5	0.49 (0.21)	0.45 (0.19)	11.74 (4.90)	1.20 (0.50)	10	1.24 (0.19)	1.13 (0.17)	84.88 (22.62)	8.65 (2.31)

Mechanical properties of pine earlywood (PineS and PineZ) as a function of indentation depth (standard deviations in parentheses) Table 2

Indentation depth [µm]	PineS					PineZ				
	N	E* [GPa]	E_{IT} [GPa]	H_{IT} [MPa]	HV_{IT} [Vickers]	N	E* [GPa]	E_{IT} [GPa]	H_{IT} [MPa]	HV_{IT} [Vickers]
10	6	0.58 (0.27)	0.53 (0.24)	10.22 (3.17)	1.04 (0.32)	5	0.49 (0.21)	0.45 (0.19)	11.74 (4.90)	1.20 (0.50)
20	6	0.60 (0.19)	0.55 (0.17)	17.52 (4.64)	1.79 (0.47)	10	0.52 (0.25)	0.47 (0.23)	11.55 (5.24)	1.18 (0.53)
40	6	0.83 (0.15)	0.75 (0.13)	33.11 (6.35)	3.38 (0.65)	11	0.82 (0.16)	0.75 (0.14)	25.27 (8.18)	2.58 (0.83)
80	6	0.69 (0.05)	0.63 (0.05)	27.97 (3.11)	2.85 (0.32)	8	0.59 (0.12)	0.54 (0.11)	16.54 (4.01)	1.69 (0.41)
120	6	0.72 (0.04)	0.66 (0.04)	37.93 (5.08)	3.87 (0.52)	10	0.58 (0.07)	0.53 (0.06)	16.78 (4.09)	1.71 (0.42)

Table 3 presents the explanations of the symbols from Tables 1 and 2 [10].

3.3. Indentations to Different Depths in Wood Treated with the Particles

This set of measurements was designed differently and had a different purpose from the set with the results shown in Table 1. In the first case (Table 1), the indentations were performed with a pre-set indentation load of 5 N, and indentation depths at this maximal force reached values up to 40-60 µm, depending on the measurement site. The aim of this set of measurements was to prove if differences in the mechanical properties of earlywood and latewood can be determined by the micro-indentation technique. On the other hand, the second set of measurements aimed at seeing the potential mechanical differences as a function of the presence of nanoparticles on a surface layer of wood. In this case, the maximal penetration depths were pre-set to be between 10 and 120 µm and the indentation force was automatically determined/controlled by the device. Therefore, the direct comparison between the results in Tables 1 and 2 could not be made.

Table 2 shows the results of our micro-indentation experiments to determine the potential variation in mechanical properties as a function of indentation depth. In other words, to see if the surface layers of wood are harder due to the presence of nanoparticles on a wood surface. It was hypothesised that such variation could be the result of higher concentrations of ZnO or SiO_2 particles on the surface and in the wood layers near the surface. Unfortunately, we could not find any clear differences with respect to the indentation depth, especially if we consider the high variability (high standard deviation) of the results.

Explanation of the symbols in Tables 1 and 2 [10] Table 3

N	Number of measurements	
E^*	Plane strain modulus	A measure of the stiffness of a material in a specific loading condition, calculated from equation (1): $$E^* = \frac{1}{\frac{1}{E_r} - \frac{1 - v_i^2}{E_i}} \quad (1)$$ where: E_i is the elastic modulus of the indenter [GPa]; v_i – the Poisson's ratio of the indenter; E_r – the reduced modulus (a combination of the sample material and indenter elastic deformations, [GPa]).
E_{IT}	Instrumented indentation modulus	Calculated from equation (2): $$E_{IT} = E^* \cdot \left[1 - v_s^2\right] \quad (2)$$ where; v_s is the Poisson's ratio (0.3).
H_{IT}	Indentation hardness	H_{IT} is a measure of the resistance to permanent deformation or damage, obtained in equation (3): $$H_{IT} = \frac{F_{max}}{A_p} \quad (3)$$ where: F_{max} is the maximum test force [N]; A_p – the projected contact area [cm^2].
HV_{IT}	Vickers hardness calculated from H_{IT}	HV_{IT} is estimated from H_{IT} by equation (4): $$H_{IT}[MPA] = \frac{9.81}{\sin\alpha} \cdot H_{IT}[Vickers] \quad (4)$$

In Table 2, only the results of the micro-indentation tests for earlywood treated with nanoparticles are shown, but not for latewood. This is because the objective of

this preliminary study at this stage was to evaluate the applicability of the micro-indentation technique in the case of nanoparticle treated wood. We wanted to see if it was possible to detect hardness differences between wood with and without nanoparticles. However, in continuation of our research, there is a clear need for micro-indentation experiments on the latewood of nanoparticle treated wood, and these experiments are planned for the future. We will also investigate a different indentation technique with multiple loading cycles on the same sites.

4. Conclusions

Based on an extensive literature review, it was demonstrated that micro-indentation is a highly useful and versatile technique with the potential to study the mechanical properties of surface-treated wood. In addition, micro-indentation studies were conducted on untreated Scots pine wood and Scots pine wood with surface deposits of SiO_2 or ZnO. The differences between the mechanical properties of early and latewood can be clearly seen by the micro-indentation technique and it was shown that the treated wood is less stiff and softer. On the other hand, we could not find any influence of ZnO and SiO_2 particles on the mechanical properties as a function of the indentation depth.

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