Original Article

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Hardness of surface-densified wood. Part 1: material or product property?

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Abstract: The established methods for testing the hardness of wood are of questionable value for assessing the performance of surface-densified wood, since the density profile beneath the densified surface is an important property that needs to be considered. The purpose of this study was to evaluate the influence of the density profile of surface-densified wood and the hardness test parameters, such as indenter geometry and applied load on the measured hardness. The influence of the density profile varied considerably depending on the hardness test parameters. This can make a comparison of hardness values of surface-densified wood prone to misinterpretation. The selection of hardness test parameters should either be product-specific, or the density profile itself should be used to evaluate the hardness of surface-densified wood. A strong influence of the density profile on the indentation depth development during the hardness tests indicates the possibility of predicting the density profile based on the hardness test methods.

Keywords: Brinell hardness; density profiles; ionic liquids; Janka hardness; wood compression.

1 Introduction

Density is one of the most important characteristics of wood as it correlates with most mechanical properties (Kollmann 1936). High-density wood is preferable to low-density wood

in applications where high strength, hardness, or resistance against wear are of importance. Increasing the density by densification, e.g. by a transverse compression of the wood material, is therefore seen as an attractive approach to improve the competitiveness of low-density wood species (Navi and Sandberg 2012). Bulk densification, i.e. the compression of the entire volume of wood, normally leads to a reduction in wood volume of about 50–70%, whereas surface densification limits the density increase to the surface region. This results in only a slight decrease in the total wood volume, whilst achieving a similar resistance to mechanical stresses in the surface region. Surface densification of low-density wood species for use in products such as flooring and table-tops – where a hard and wear-resistant surface is of primary interest – has a potential economic advantage over the use of bulk densification or high-density wood species. Large-scale industrial implementation has not yet been achieved, although recent developments have been promising (Neyses 2019).

Hardness is a measure of the resistance of a material to penetration by a body of a harder material, and it is commonly used to predict the performance of a wood product. In wood densification, hardness is often used to assess the effectiveness of the densification procedure. From a product perspective, the goal of densification is a high hardness rather than a high density, rendering the use of hardness as a legitimate property. Established methods for hardness testing are designed to locate the resulting stresses close to the contact area between the indenter and the material and thus, contrary to a compression test, hardness describes a surface property. In theory, the hardness value of a homogenous material should be independent of load and indentation depth. However, in practice, test parameters such as the indenter geometry, indenter size, load, and loading regime influence the hardness value (Broitman 2017; Tabor 1951). Furthermore, the hardness tests themselves mechanically alter the material and thus the results are dependent on the characteristics of the test material. Standards are supposed to provide a foundation to obtain comparable hardness measurements, a condition that does not apply for methods of measuring the hardness of wood samples.

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The most common hardness testing methods for wood are the Brinell and Janka methods (Niemz and Stübi 2000). The Brinell method (Brinell 1900), originally developed for metals, involves a steel sphere (indenter) being pressed into the test specimen with a defined load. After removing the load, the diameter of the indentation is measured. The Janka method (Janka 1906) which was developed specifically for wood, involves a steel sphere being pressed into the wood until its equator (diameter) is reached and the required force to reach this indentation depth is recorded. The general difference between the two methods is that the Brinell method is a force-controlled test, whilst the Janka method is an indentation-depth controlled test (Figure 1). The measurements with these two methods may test different aspects of the material because the depth-controlled methods do not take into account any elastic spring-back of the material after releasing the indenter load. Since there is a clear relationship between the indentation diameter and the indentation depth for all indenter geometries, and because the indentation depth is fairly easy to measure during a hardness test, indentation depth has become commonly used to describe indentations in hardness testing.

The Brinell hardness (HB) is the ratio between the applied target load and the residual contact area of the indentation after removal of the load. For wood, the HB measured according to EN 1534 (CEN 2020) defines a load of 1000 N applied with a spherical indenter of 10 mm in diameter. In depth-controlled tests, such as the Janka method (ASTM 2020) and the Japanese standard for surface

hardness (JSA 2009), the approach is turned around and the indentation depth is predefined. The contact area between the indenter and the material is fixed in depth-controlled tests, and the hardness is expressed as the required force to reach the target depth. The Janka method has been criticised because of the friction and rupture caused by the large indentation depth (Kollmann 1951). The Japanese standard targets a shallow indentation of 0.32 mm in depth and thus only a narrow volume at the wood surface is tested. Several other test procedures (Doyle and Walker 1985; Monnin 1919; Möhrath 1932; Pallay 1938) and methods of calculating hardness (Meyer 1908) exist, but they are less frequently used for testing wood hardness.

Testing the hardness of wood is highly affected by the unique material properties of wood. Wood is a porous material, and under compression the bulk volume deforms plastically due to collapse of the cells and, thereafter, the elastic compression of the cell walls (Gibson and Ashby 1988). Plastic deformation of compacted wood cells, i.e., densification of the cell wall itself, requires high stresses. The initial density of the wood and the used test force have a large influence by how much the wood deforms elastically and plastically during a hardness test (Doyle 1980). As observed by Schwab (1990), an increased test force will increase the HB. The test force does not only affect the share of elastic and plastic deformation, but also determines the indentation depth in a force-controlled test. This affects the amount of wood volume under stress, and depending on the indenter geometry, it determines the types of mechanical stresses such as compression, tension

Figure 1: Principle of (A) a force-controlled hardness test and, (B) a depth-controlled hardness test. In force-controlled hardness tests the force is gradually increased until the target force is reached and held to an equilibrium, after which the force is released, and hardness is calculated based on the dimension/depth of the indentation. In depth-controlled hardness tests the strain is gradually increased until the target indentation depth is reached. Hardness is expressed as the required force to reach the target indentation depth.

and shear (Fischer-Cripps 2000). At a low indentation depth, the material directly under the tip of a spherical indenter is stressed in tension and compression, but with increasing indentation depth, more and more friction and shear stresses arise around the sides of the indenter. Indenter geometries such as wedges or cones do not change the geometrical shape of the indentation with increasing indentation depth. Therefore, similar types of mechanical stresses act in the material, independent of indentation depth (Doyle 1980).

The highly non-uniform density distribution of surface-densified wood makes it difficult to obtain a hardness value from a standard hardness test that accurately represents the properties of the surface (Rautkari, Kamke, and Hughes 2011a). The density profile (DP) has a significant influence on the hardness value (Laine et al. 2013a). The shape of the DP depends mainly on the parameters of the densification process, such as press temperature, pressing speed, compression ratio, moisture content and species (Rautkari et al. 2011b; Laine et al. 2013a) and it should be possible to produce a certain DP based on these parameters as it was shown for particleboards (Hänsel et al. 1988). Neyses et al. (2020) observed that surface-densified wood with a thin (1.2 mm) layer of high-density beneath the surface of the material exhibited lower HB than expected based on the density increase, and concluded the distance between the DP peak and the densified surface influences the HB value. Rautkari et al. (2011a) showed a strong influence of the DP and test load on the HB for wood composites similar to densified wood. For depth-controlled hardness tests, the choice of the target indentation depth determined the tested depth of the material and hence the influence of the DP. In both force-controlled and depth-controlled hardness testing, inhomogeneous materials with high resistance to indentation close to the test surface will benefit from small target indentation depths or target loads, while a deeper target indentation or higher target load might lead to a penetration of the densified layer and the hardness value will be influenced by the material beneath the surface layer. In addition, densified wood reacts differently to the indenter-induced stresses than the undensified wood in terms of elastic and plastic strain. An important issue for surface densified wood is to get the location and shape of the DP to achieve the desired surface properties such as hardness (Laine, Rautkari, and Hughes 2013b) for a specific load situation of a product, and to find a hardness-testing method representing these surface properties. The standard methods currently used for hardness testing are not adopted for testing of surface densified wood and care

should be taken when evaluating different densification procedures or density profiles.

The described behaviour of existing hardness test methods raises the question if there is a hardness test method available that can provide appropriate hardness values for surface-densified wood. Thus, the purpose of this study was to evaluate how the current test methods are affected by the interaction of test parameters and the DP, and suggest improvements of the hardness test procedure for surface-densified wood.

2 Materials and methods

2.1 Specimen preparation

480 defect-free Scots pine (*Pinus sylvestris* L.) specimens $50 \times 21/18.5 \times 50$ mm (tangential $\times$ radial $\times$ longitudinal) in size were used. The two different initial thicknesses were prepared to allow densification at two different compression ratios (CRs). The maximum growth-ring angle relative to the tangential surface to be densified was 20° . The dry density of the specimens ranged from 425 kg m^{-3} to 578 kg m^{-3} . The specimens were conditioned to equilibrium moisture content (MC) at 20°C and 65% relative humidity and then randomly distributed into 10 groups of 48 specimens for treatment as shown in Table 1.

2.2 Chemical pre-treatment

Wood can be treated with an ionic liquid (IL) prior to densification, resulting in a change of the location and shape of the DP, e.g. to get a densified peak-region immediately beneath its surface (Neyses et al. 2020). An IL is a salt, commonly with a melting temperature below 100°C , and some type of ILs have been used to dissolve crystalline cellulose by the cleavage of intermolecular bonds between cellulose chains (Hanabusa et al. 2018). This may lead to plastic deformation of the crystalline cellulose in wood under compression, resulting in a DP considerably different from the achievable DP without IL pre-treatment. Due to the highly inhomogeneous DP of IL pre-treated specimens, a higher sensitivity to the hardness test parameters is expected.

The specimens in the IL1 and IL2 groups were oven-dried at 103°C to 0% MC and 2.5 g of an aqueous solution (25 wt%) of 1-butyl-3-methylimidazoliumchloride (BmimCl of 98% purity, Acros Organics, Fair Lawn, NJ, USA) were applied with a pipette to the tangential surface to be densified (ca. 2.5 ml per specimen). These specimens were then dried to 0% MC at 70°C .

2.3 Surface densification

The specimens were uniaxially compressed in a laboratory hot press (Fjellman Press AB, Mariestad, Sweden) with one heated platen. The process parameters were chosen based on previous research (Laine et al. 2013a; Neyses et al. 2020). Two CRs of 8.1 and 19% were obtained by compressing specimens with two different initial thicknesses to the same target thickness of 17 mm. The specimens were

Table 1: Material and process parameters in the test.

Group ID	BmimCl pre-treatment	Mean density $\pm$ st. dev (kg m ⁻³) at (13% MC)	MC at pressing (%)	CR (%)	T (°C)	Preheating time (s)	Press speed (mm/min)	Press closing time (s)
D1	No	574 $\pm$ 48	13	8.1	150	10	2.7	33
D2	No	574 $\pm$ 51	13	8.1	200	10	2.7	33
D3	No	571 $\pm$ 40	13	8.1	150	10	1.0	90
D4	No	573 $\pm$ 49	13	8.1	200	10	1.0	90
D5	No	574 $\pm$ 48	13	19.0	150	10	2.7	90
D6	No	558 $\pm$ 44	13	19.0	200	10	2.7	90
D7	No	574 $\pm$ 50	13	19.0	150	10	1.0	240
D8	No	573 $\pm$ 52	13	19.0	200	10	1.0	240
IL1	Yes	574 $\pm$ 43	0	8.1	250	10	2.7	33
IL2	Yes	568 $\pm$ 49	0	19.0	250	10	2.7	90

The densification process is based on a full factorial design with three factors with two levels each (CR, temperature and press speed). Two treatment groups with IL pre-treatment complement the experimental design. Number of specimens per group = 48. BmimCl, 1-butyl-3-methylimidazoliumchloride; CR, compression ratio, see (Laine et al. 2013c); T, temperature; and MC = moisture content.

placed with the bark-side tangential surface towards the heated platen. First, a low contact pressure was applied and held for 10 s to raise the temperature of the surface and thus plasticise the wood. The pressure was then increased (3–5 MPa) resulting in a strain rate of 1 mm min⁻¹ or 2.7 mm min⁻¹ and compression was stopped at the target thickness by mechanical stops. The specimens were kept under pressure and heat for 240 s, after which the press was cooled by internal water circulation until its platen temperature reached 35 °C (4–6 min, depending on pressing temperature), after which the pressure was released. All the specimens were then wrapped tightly in plastic film to prevent moisture changes prior to further testing after ca. 14 days.

2.4 Density profiles

After surface densification but prior to hardness testing, the DP of each specimen was obtained by X-ray densitometry with a laboratory DP analyser (DENSE-LAB X, Electronic Wood System GmbH, Hameln, Germany) with a spatial resolution of the X-ray beam of 50 μ m. Measurements were undertaken with step intervals of 44 μ m in the radial direction. Key features of the DPs are the density peak, i.e., the highest local density achieved by the densification process, and the peak distance, i.e., the distance of the density peak to the densified surface. The “densified surface” is defined as the surface in contact with the heated platen during densification.

2.5 Hardness testing

Seven different hardness test procedures were used (Table 2), each test being carried out on a number of randomly chosen specimens from each treatment group. The hardness measurements were made in a ZwickRoell ZwickiLine 2.5 TS universal testing machine (ZwickRoell GmbH & Co. KG, Ulm, Germany) equipped with a 2.5 kN load cell. The applied force and indentation depth measured with the crosshead displacement (Niemz and Stübi 2000) were recorded every 0.1 s in all cases.

Hardened steel indenters were used in all the tests and were applied on the densified surface in the radial direction. The position of contact was fixed on the centre of the longitudinal axis of each specimen. A spherical indenter was chosen based on the standard EN 1534 standard (CEN 2020) and JIS Z 2101 (JSA 2009). The cylindrical indenter was chosen based on the “Chalais” hardness test (Monnin 1919). The wedge was chosen based on the principal of depth-independent indentation geometries (Doyle and Walker 1984). The loading regime of all the tests with the exception of the Brinell-Japan test was according to the EN 1534 standard, with slight modifications. The load was increased linearly to the target load over a period of 15 s and then held for 25 s. To study the degree of elastic indentation, a controlled unloading phase of 15 s was added to the loading scheme, where the elastic recovery was defined by the difference between the maximum indentation during the test and the residual indentation immediately after the test. The final loads of the wedge- and cylinder-based tests were chosen to achieve the same contact pressure at the average indentation depth determined in the Sphere-1000 N test. The Brinell hardness (HB) was calculated by

$$HB = \frac{F}{A_{\text{cont}}} \quad (1)$$

where F is the target load and A_{cont} is the curved area of the indentation. According to the EN 1534 standard, the area of the residual indentation (A_{cont}) is used to calculate HB (Figure 2B). Another commonly used approach is to calculate HB as the ratio of the force to the contact area at maximum indentation (d_{max}) as shown in Figure 2A (Neyses 2019; Niemz and Stübi 2000; Rautkari 2012). HB was calculated according to both approaches.

Elastic recovery ϵ was calculated by

$$\epsilon = \frac{d_{\text{max}} - d_{\text{residual}}}{d_{\text{max}}} \quad (2)$$

where d_{max} is the maximum measured indentation depth and d_{residual} is the indentation depth after release of the load.

With the Brinell-Japan test, a depth-controlled surface hardness test was carried out according to the JIS Z 2101 standard, where

Table 2: The geometries and test parameters of the different hardness test procedures.

Test method	Indenter geometry	Loading according to:	Target load (N)	No. of measurements per group	Total no. of measurements
Sphere-1000 N	sphere	EN 1534 + unloading	1000	32	320
Sphere-500 N		EN 1534 + unloading	500	32	320
Brinell-Japan		JIS Z 2101 + further loading	–	32	320
Cylinder-2500 N	cylinder	EN 1534 + unloading	2500	16	160
Cylinder-1250 N		EN 1534 + unloading	1250	16	160
Wedge-2500 N	wedge	EN 1534 + unloading	2500	16	160
Wedge-1250 N		EN 1534 + unloading	1250	16	160

Loading regimes were according to EN 1534 (CEN 2020) or JIS Z 2101:2009 (JSA 2009) standards.

Figure 2: Contact area A_{cont} (A) at maximum load and (B) after release of the load.

the indenter was pressed into the specimen at a constant speed of 0.5 mm/min until the target depth of 0.32 mm was reached. To study the effect of different target indentation depths, the loading was continued at a speed of 2 mm/min until an indentation depth of 4 mm was reached. Hardness of the Brinell-Japan test is expressed as the required force in N to indent to 0.32 mm.

3 Results and discussion

The results of the hardness tests are an interaction of material properties and test parameters. The properties of surface-densified wood on a cell level and its composition in the indentation direction of the hardness tests (i.e. the DP) represent the material properties in this study. The influence of target load in hardness testing, the way of

calculating the hardness number and the indenter geometry represent the hardness test parameters.

3.1 Material property: density profile

The mean DP for each treatment group is shown in Figure 3. The DPs of the specimens densified without chemical pre-treatment (D1–D8) are fairly similar in shape, location and density of the peak region, but there is a clear difference between these DPs and the DPs of the groups that have been pre-treated with ILs.

The DPs of the IL-treated specimens show a higher density peak closer to the densified surface than the other groups. This suggests that the hardness should be higher for these specimens (Neyses et al. 2020). The greater CR of

Figure 3: Average DPs of the different groups (D1–D8, IL1 and IL2 according to Table 1, $n = 48$) of surface densified wood with a compression ratio of (A) 8% and (B) 19%.

the groups in Figure 3B resulted in wider density peaks. The undensified wood had a density of about 575 kg m^{-3} (cf. Table 1).

3.2 General hardness test results

The data on elastic recovery, the HB based on the residual indentation and the HB based on the maximum indentation for all six force-controlled hardness tests and the required indentation forces for the depth-controlled test are available in Supplementary Tables S1–S4. General trends and observations are presented in the following text.

3.3 Material property: compressive strain in densified wood

The groups with highly-densified surface regions exhibited the highest elastic recovery as reported earlier by e.g. Rautkari et al. (2013). Elastic recovery ranged from 45% (Sphere-1000 N) for material containing its density peak further away from the densified surface to 91% (Cylinder-2500 N) for material in the group IL2 which exhibited a wide and strong density peak close to the densified surface.

Whilst testing densified Scots pine wood in radial compression, Blomberg et al. (2005) observed that the proportional limit of densified wood became diffuse and the modulus of elasticity became low compared to undensified wood. These findings and the high elastic

recovery of densified wood indicated that densified wood under transverse compressive stress behaves like undensified wood, with the important difference that the densification process itself incorporates the first two stages of the stress-strain curve plastically into the material (Figure 4). During the thermomechanical densification process, the glass-transition temperature of the wood material is exceeded, leading to elastic cell wall buckling in the plateau-stage (2). Subsequent reduction of the temperature, undercutting the glass transition temperature of the material, transforms the elastic deformation into a “plastic” deformation, i.e., most of the elastic recovery is interlocked. When loading the densified wood in transverse compression (Figure 4B), the cells have already collapsed, so that opposing cell walls touch each other and the initial elastic bending of cell walls (1 in Figure 4A) is physically blocked. At high degrees of densification, the linear elastic stage (1) and the following plateau of void volume reduction (2) should either be shorter or non-existent in the stress-strain curve. Thus, compressive loading of densified wood in the transverse directions mainly results in elastic deformation in the cell walls. Consequently, when testing the hardness of surface-densified wood, a material with high variation in its material properties is investigated. On a local level, the degree of previous thermomechanical compression of the wood defines by how much the wood reacts in an elastic-plastic (undensified wood cells) and in a non-linear elastic fashion (densified wood cells) to the indentation stress.

In force-controlled tests, the ratio of densified to undensified wood material and the order of these layers in

Figure 4: Idealised visualisation of stress-strain curves of (A) undensified and, (B) densified wood cells under compressive loading in the transverse direction. (1) Linear-elastic bending of the cell walls. (2) Plastic buckling of the cell walls and reduction of void volume. (3) Contact of opposing cell walls and steep increase of stress. (4) Full densification on a macroscopic level. Cell walls are visco-elastically compressed. On a macroscopic level stage 2 and 3 will happen simultaneously.

the indentation direction (i.e. the DP) will not only determine the indentation depth but also the degree of elastic recovery. Hardness that has been determined based on the residual indentation alone accounts for the plastic deformation caused by the indenter.

3.4 Test parameter: target load

An increase of target load in a force-controlled test will increase the amount of induced stress, which in turn increases

strain in the material and thus the maximum indentation depth reached. For a homogeneous wood specimen, it is already known that a doubling of the target load results in a disproportionally higher hardness (Schwab 1990). In hardness testing of surface-densified wood, the influence of the level of the target load depends on the shape and location of the DP (Rautkari et al. 2011b). This effect on the hardness is visualised in Figure 5 by the indentation-vs-time curves measured in two specimen during HB testing.

A similar hardness was determined for both specimens at 1000 N target load, but at a given time during the loading

Figure 5: The development of the indentation depth in two surface-densified specimens (different process conditions) during an HB test with a target load of 1000 N.

Case (A) represents a specimen with a densified peak region fairly deep beneath the densified surface, and case (B) represents a specimen with a densified peak region immediately beneath its surface.

Figure 6: HB based on the residual indentation area determined at 500 and 1000 N target load for specimens with different DPs.

regime, the indentation depth is greater in the low-density surface (case A) than in the high-density surface (case B). Ignoring the minor effect of holding the target load, hardness determined at 500 N load (indentation depth measured at 7.5 s in Figure 5) should be considerably lower in case A due to the greater indentation depth.

Laine et al. (2013a) showed that DP-characteristics such as the peak density and peak distance to the densified surface correlate with HB, but as shown in the present study, the correlation is dependent on the target load. The HB according to EN 1534, i.e. the ratio of the force to the contact area after releasing the force, is shown in Figure 6.

The results showed that inhomogeneous DPs made it difficult to judge the HB and that the target load influenced the measured hardness. Among the groups D2, D6 and IL1, D2 appeared to be the hardest when tested with the Sphere-1000 N method, while the same group exhibited the lowest hardness when tested with the Sphere-500 N method. IL1 is an extreme example of target load influence, where the hardness is higher at 500 N target force than at 1000 N. If

the peak is located close to the densified surface (IL1 and IL2) a low target force will not have high enough stresses to overcome the mechanical resistance of the high-density layer, and the hardness value represents the properties of that thin layer. By applying a higher force, stresses may overcome the resistance of the densified layer and be transferred to the undensified region further from the densified surface, such that the hardness value represents a mixture of the high-density surface layer and the underlying low-density material. Determining which way of hardness determination best represents the in-service behaviour of the material is an open question.

3.5 Test parameter: indenter geometry

The general influence of elastic-recovery and target load on HB was similar for all three types of indenter geometries. The indenter geometry led to the average indentation depths as shown in Table 3.

The relatively deep indentations at maximum load in the Cylinder-2500 N and the Wedge-2500 N indicated that stresses reached much further into the tested specimens than in the other methods. This was supported by the observation of a high degree of “sinking-in” of several specimens around the wedge-shaped and cylindrical indenters at 2500 N (Figure 7A). The indenter geometry and the applied test force led to deformation of the whole wood piece indicating that a large share of the wood volume was mechanically stressed. The Wedge-2500 N and Cylinder-2500 N methods came close to being a compression test of the whole specimen volume and were thus invalidated as a hardness test. The Wedge-1250 N (Figure 7B), Sphere-500 N (Figure 7C) and Cylinder-1250 N methods led to very shallow indentations.

The tips of the cylindrical and wedge-shaped indenter are blunt, which leads to a relatively large contact area at low indentations depth. The induced stress is hence distributed over a larger area and specimens with the densified layer close to the densified surface benefit from the elastic behaviour. HB was as high as 328 N mm^{-2} in case of the group IL2 tested with the Wedge-1250 N method.

Table 3: Average indentation depth of all specimen groups. Indentation depth $d_{\max}$ measured at maximum load (at 40 s test time) and d_{residual} after release of the load.

	Sphere-1000 N (mm)	Sphere-500 N (mm)	Cylinder-2500 N (mm)	Cylinder-1250 N (mm)	Wedge-2500 N (mm)	Wedge-1250 N (mm)
Average $d_{\text{residual}} \pm \text{st. dev}$	0.91 ± 0.44	0.42 ± 0.18	1.16 ± 0.83	0.26 ± 0.14	0.79 ± 0.24	0.34 ± 0.17
Average $d_{\max} \pm \text{st. dev}$	1.52 ± 0.36	0.94 ± 0.25	2.54 ± 1.18	0.85 ± 0.24	2.66 ± 1.04	0.98 ± 0.28

Figure 7: (A) “Sinking-in” of a specimen around the cylindrical indenter. (B) Shallow indentation of the wedge-shaped indenter. (C) Shallow indentation of the spherical indenter. The image of the spherical indenter is staged. The indentation point was moved to the edge of the specimen, as the indentation process cannot be seen from the side during the hardness test.

Figure 8: HB determined at 500 and 1000 N target load at maximum indentation. The outlines show the HB based on the indentation area after release of the load (cf. Figure 6).

3.6 Test parameter: calculation method

It is evident that expressing hardness based on the ratio of applied load to the residual contact area is not an applicable concept when elastic deformation is the main mode of strain (Broitman 2017). By determining HB at maximum indentation, the plastic and elastic response to the indentation load is considered, but not the elastic recovery. Figure 8 shows the respective HB determined at maximum indentation.

The results are closely connected to the elastic recovery. A highly elastic material will show a relatively high HB determined at minimum indentation, but the positive effect of elasticity will not be exhibited when determining the hardness at maximum indentation. It also has to be considered that both ways of calculating the hardness do not take into account the occurrence of sinking-in or piling-up during the tests (Figure 9), with both phenomena having a large effect on the resulting hardness value (Norbury and Samuel 1928). Neither indentation depth measurements at maximum load nor measurements of the residual indentation area can account for this. Furthermore, three different types of residual indentations were observed in the material for all indenter geometries (Figure 10).

The appearance of the indentations and the phenomenon of sinking-in indicate that a different area than the contact or projected area contributes to the resistance of the material during a hardness test and hence calculating hardness based on these values may be misleading even when determining HB at maximum load. Defining an effective area of the wood which resists the indentation force to express hardness as a force over the resisting area is complex. The material properties and test parameter change the non-uniform stress distribution under the indenter. In theory, the true projected area of contact at the maximum load to account for sinking-in could be measured optically, but in practice it would be almost impossible as the measurement must be done at maximum load during the hardness test.

The failure behaviour also indicates another factor not considered by the hardness test. The observed brittle behaviour (Figure 10B), mainly in the IL1 group, suggests that the wood failed in a shear fashion at the fracture zone.

Figure 9: (A) Sinking-in around the indenter during loading. (B) Piling-up around the indenter during load application. (C) Residual indentation after the release of the load.

Figure 10: Appearance of (A) shallow indentations, (B) indentations where the material failed in a brittle manner and (C) deep indentations. Shallow indentations are common for highly densified specimens with a thick densified layer where elastic deformation is dominant. Surfaces which failed in a brittle manner are found in specimens with a thin surface-layer of high density. Deep indentations are typical for specimens where the densified area is located far from the test surface.

For a product such as flooring or furniture the impact of brittle failure is large and the failure mode should thus be an essential part of a hardness test result.

3.7 Test parameter: depth-controlled method

The influence of load can be negated by using a depth-controlled test, such as the Brinell-Japan test. The Brinell-Japan method determines the force needed to indent a sphere 10 mm in diameter to a depth of 0.32 mm. Since the densified region in the groups D1-D8 is located deeper in the specimens, the determined hardness for these groups is very similar (82–123 N). The IL-treated specimens, groups IL1 and IL2 with the densified region close to the

densified surface, showed a high hardness of 283 and 317 N respectively. Similar to HB determination at maximum load, the Brinell-Japan method does not account for elastic spring-back. The target indentation depth of 0.32 mm is predetermined by the standard and has a large influence on the stressed volume.

More information of the material can be acquired by further depth-controlled loading. Figure 11 shows the Brinell-Japan test, i.e. the force required to continuously press the spherical indenter to a given depth for the three representative test groups.

The slope of the individual curves provides a good picture of the DPs, where the forces required for indentation into the IL-treated specimens were similar to each other in the beginning of the test but where, with further indentation depth, the thinner densified region in the IL1 group (CR 8%)

Figure 11: The force-vs-indentation measured with the Brinell-Japan test method. The load cell of the hardness testing machine was limited to a test force of 2500 N and the Brinell-Japan tests stopped when this force was reached.

was overcome and the slope angle decreased, which was not the case in the IL2 group (CR 19%). The D2 group behaved differently because the densified region is located further beneath the specimen surface.

3.8 How to measure hardness in surface-densified wood

The problematic influence of the DP on the hardness measurements is difficult to overcome by using the standardised hardness tests. To secure comparable indentations, a specific range of indentation depth, or appropriate target forces need to be defined. Defining the “right” target force for surface-densified wood would be complex and constant adjustments of the target force depending on the DP would be required. Depth-controlled hardness tests lack the consideration of elastic spring-back.

As a general material property, hardness as the ratio of applied load over an indentation contact area does not provide very meaningful data for surface-densified wood. From a product perspective it is most reasonable to define expectable load case forces or maximum acceptable indentation depths. For example, a heavy dining table weighing 200 kg resting on four legs creates a rather low but long-term pressure on the flooring. A person weighing 100 kg in high heels creates a high and dynamic short-term pressure at a high frequency. Both cases and their frequencies should be considered. Furthermore, a differentiation between hardness based on residual indentation

depth and maximum indentation depth can be made to account for short-term and long-term exposure. It has been shown that some DPs perform better for certain load cases than others and depending on a certain product or product group an optimal DP should exist. Determining the DP of surface-densified wood is hence one way to assess the performance of surface-densified wood. X-ray densitometry is one option to measure the DP, but high acquisition costs and relatively small specimen dimensions limit the usability of the method. In the present study, a change of target load (cf. Figure 5) or target indentation (cf. Figure 11) showed that the DP is somehow reflected in the indentation-vs-time and indentation-vs-force curve of the hardness measurements. The continuous measurements during hardness tests may give valuable information about the DP, which could be used to evaluate the performance of surface-densified in a more general way than individual hardness tests, while still acquiring the traditional hardness number.

4 Conclusions

To improve the way in which the hardness of surface-densified wood is evaluated, several hardness test methods were applied and the influence of the material properties of surface-densified wood and changes of the hardness test parameters on the obtained hardness values were analysed. There is a strong influence of the hardness test parameters such as applied load and indenter geometry on the material behaviour during hardness testing, and the ambiguities found in the hardness measurements of undensified wood are enhanced by adding another dimension of variety, i.e. the changing material properties in the densification direction of surface-densified wood.

The interaction of the density profile and the test parameters resulted in a high variation of measured hardness values, regardless of which method was used. It is therefore questionable whether there exists a generally valid concept of hardness as a material property for surface-densified wood. However, the density profile itself may be used to assess the performance of surface-densified wood and its strong influence on the material behaviour during hardness testing is embedded in the continuous measurement data during hardness tests. This may allow the prediction of the density profile from measurements of the indentation in a hardness test.

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