Holzforschung, Vol. 60, pp. 698–701, 2006 • Copyright © by Walter de Gruyter • Berlin • New York. DOI 10.1515/HF.2006.118

# Pilot investigation of the mechanical properties of wood flooring paint films by *in situ* imaging nanoindentation

**Zehui Jiang[1], Yan Yu[2,*], Daochun Qin[2], Ge Wang[2], Bo Zhang[1] and Yuejin Fu[1]**

[1] Chinese Academy of Forestry, Beijing, China

[2] International Center for Bamboo and Rattan, Beijing, China

*Corresponding author.
International Center for Bamboo and Rattan, 8 Futong Eastern Street, Wangjing, Chaoyang,
Beijing 100102, China
E-mail: yuyan@icbr.ac.cn

## Abstract

*In situ* imaging nanoindentation was used to characterize the mechanical properties of pure paint films on eight types of plywood-based flooring with different wear resistance. Correlation between the mechanical properties of paint films and the wear resistance of wood-based floorings was investigated. The average modulus of elasticity (MOE) and hardness of the paint film tested were 3.33–5.77 and 0.19–0.34 GPa, respectively. A strong linear relationship ($r^2=0.87$) between the MOE and hardness of the paint film was found. Neither of these properties was directly correlated to the macro wear resistance of the floorings. Moreover, the composite index $H^{1.5}$ $E^{-1}$ – frequently used as an indicator of wear resistance in materials science – was not useful either in evaluating the wear resistance of wood floorings. However, *in situ* imaging nanoindentation was suitable for characterization of the mechanical properties of paint films, although this technique was not suited for direct evaluation of the wear resistance of wood floorings.

**Keywords:** *in situ* imaging nanoindentation; mechanical properties; paint film; wear resistance; wood flooring.

## Introduction

Thin film technology has been widely applied in many industrial fields. The most typical applications are for improvement of weathering, wear, chemical resistance, and mechanical properties. This technology has been used in the wood processing industry for decades, with paint films widely used on the surface of various wood products (furniture and flooring). The wear resistance of paint films on wood flooring has long been recognized as one of the most important properties, but the modulus of elasticity (MOE) and hardness are seldom investigated. This is primarily because of a lack of proper methods for measurement.

Techniques for thin film characterization in the field of materials sciences have been summarized by Chen et al. (2001). Among these, the nanoindentation technique has rapidly developed and represents a very powerful tool for investigating the correlation among the process technology, microstructure and mechanical performance of thin films on a nano scale (Catledge et al. 2002). The most important and unique advantage of nanoindentation is the capability to directly measure the mechanical properties of thin films without the need for peeling it off from the substrate material in advance. The concept of nanoindentation was introduced into wood science only a few years ago. Its application so far is limited to evaluation of the mechanical properties of softwood tracheid cell walls (Wimmer et al. 1997; Wimmer and Lucas 1997; Gindl and Gupta 2002; Gindl et al. 2004; Jiang et al. 2004). The present research focused on the feasibility of nanoindentation to characterize the mechanical properties of paint films and on the relation of the mechanical parameters obtained to the macro wear resistance of wood flooring.

## Materials and methods

### Materials

Eight types of plywood-based flooring with different wear resistance were supplied by the National Wood-based Panels Inspection and Supervision Center of China (NWPISC). The paint films on these floorings are formed of unsaturated polyester cured by UV radiation. The wear values of the wood floorings measured by NWPISC according to the China national standard for solid wood flooring (GB/T 15036.2-2001) are listed in Tables 2 and 3. The wear value is the weight loss of a paint film after being abraded 100 times with a table-type abrader (JM-4, Shanghai Modern Environment Engineering Technique Co., Ltd). A higher wear value indicates poorer capability of resisting wear.

### Nanoindentation test

A triboindenter produced by Hysitron Inc. (USA) was chosen to perform nanoindentation tests because of its outstanding *in situ* imaging function. A Berkovich diamond tip with a radius of less than 100 nm was adopted for indentation and scanning. The set peak load and loading-unloading rate were 250 μN and 50 μN s$^{-1}$, respectively. The depth at this peak load was in the range 160–230 nm for all samples, which ensured that the test result was not affected by the wood substrate.

Because the paint film on the wood floorings was very smooth, no special sample preparation procedure was required. Samples (15 mm×15 mm×flooring thickness) were directly adhered to metal stubs with a fast-cure adhesive and then fixed to a motorized sample stage using magnetic force. A very small white region was first selected under a light microscope attached to the instrument, and then scanned with a diamond tip to obtain a clear image at high magnification (similar to atomic force microscopy). Intact, smooth resin micro zones could be precisely selected from the image for indentation with the same

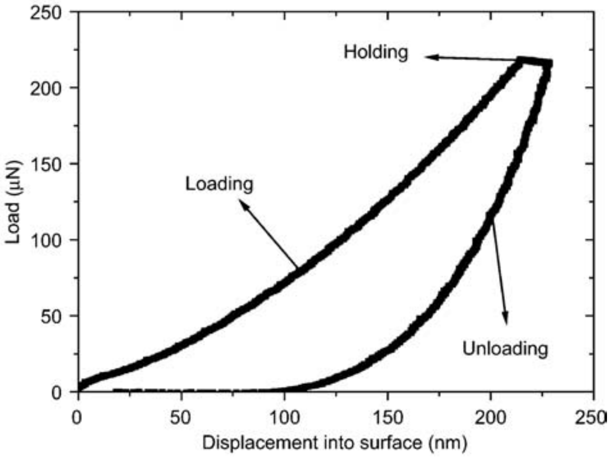


**Figure 1** Typical load-displacement curve for an indentation test on a paint film.

tip. After indentation, the scanning was repeated, and the actual positions of the indentations were determined to evaluate the test reliability. During indentation of the diamond tip into the paint film, the load and displacement were continuously recorded from loading to unloading (Figure 1). The MOE and hardness of the paint film were calculated from the curve according to the method of Oliver and Pharr (1992). More detailed information on nanoindentation can be found in Fischer-Cripps et al. (2004).

## Results and discussion

### Topography of indentations on the paint film

Figure 1 shows a typical load-displacement curve for the paint film. A significant creep effect can be observed from the holding segment of the curves. Plastic deformation accounts for nearly 40% of the total displacement, implying that the paint film is made of polymers with average viscoelasticity.

Traditional nanoindenters rely only on light microscopic images to locate and choose indentation areas. The left-hand image in Figure 2 reveals that many impurities or holes are densely distributed through the paint film. This means that light microscopy does not ensure that indentations would not take place on impurities or even in small holes. The *in situ* imaging nanoindentation technique used in this study allows for more precise selection of a clean and intact resin zone. The right-hand image in Figure 2 confirms that nearly all the indentations are located in the assigned area without any impurities and holes. Roughness analysis of 16 images with a scan size of 12 μm×12 μm indicated that the root mean square roughness (RMS) was less than 50 nm, with a mean value of only ~18.3 nm (Table 1). Both the high accuracy in locating indentations and the low roughness of the indentation area ensure the reliability of the tests.

**Table 1** Results for root mean square (RMS) roughness analysis of the surface of the wood flooring.

| | Max (nm) | Min (nm) | Mean (nm) | SD (nm) | CV (%) | n |
|---|---|---|---|---|---|---|
| RMS | 47.6 | 8.6 | 18.3 | 8.5 | 47 | 16 |

### Mechanical properties of a paint film

Data for the MOE and hardness of paint films for samples 1–8 are listed in Tables 2 and 3, respectively. The coefficient of variation (CV) for MOE and hardness for most of the paint films was less than 10%. Accordingly, the mechanical properties of the pure paint films tested were rather uniform. In general, the CV for MOE was less than that for the hardness. This indicates that the stiffness of the paint films was more stable than their hardness. The average MOE and hardness of the paint films tested were 3.33–5.77 and 0.19–0.34 GPa, respectively. Tables 2 and 3 reveal that neither the MOE nor the hardness of the pure paint films was directly correlated with the macro wear resistance of the painted wood flooring. Although the values for wear decrease with decreasing MOE and hardness for samples 1–4, this trend is not evident for samples 5–8. In particular, sample 8 showed the highest wear resistance, but its paint film had the lowest MOE and hardness values. In materials science, the hardness$^{1.5}$/MOE ratio is frequently used to indicate the wear

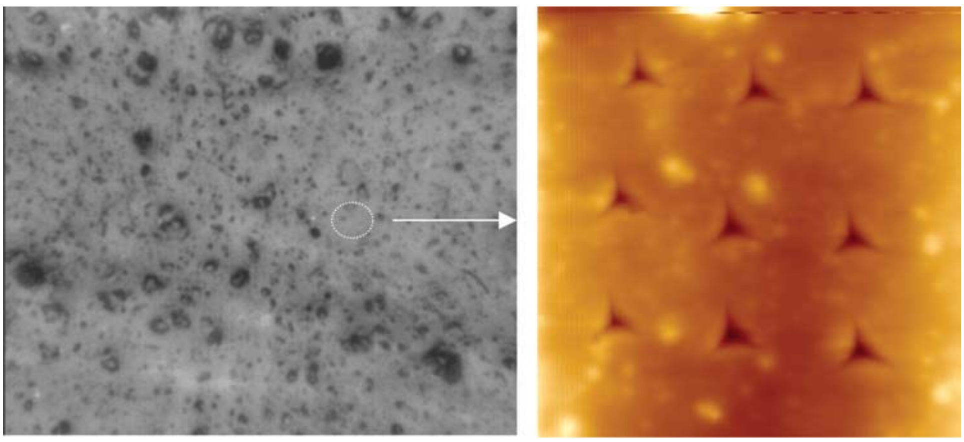

**Figure 2** Topography of indentations on a paint film on wood flooring. The left-hand image is a light micrograph of a paint film (253×). The right-hand image was obtained by scanning the zone enclosed by the white circle, with the same tip used for indentation (scan size 12 μm×12 μm).

**Table 2** MOE of paint films on wood flooring with different wear values.

| No. | Wear (g) | MOE (GPa) | | | | CV (%) | n |
|---|---|---|---|---|---|---|---|
| | | Max | Min | Mean | SD | | |
| 1 | 0.16 | 5.66 | 4.41 | 4.85 | 0.29 | 5.93 | 28 |
| 2 | 0.13 | 5.92 | 4.73 | 5.21 | 0.19 | 3.63 | 90 |
| 3 | 0.12 | 7.42 | 4.83 | 5.49 | 0.35 | 6.32 | 44 |
| 4 | 0.11 | 6.85 | 5.11 | 5.71 | 0.42 | 7.36 | 67 |
| 5 | 0.1 | 5.87 | 4.30 | 5.07 | 0.26 | 5.18 | 69 |
| 6 | 0.08 | 6.20 | 5.13 | 5.51 | 0.25 | 4.46 | 64 |
| 7 | 0.08 | 7.36 | 4.68 | 5.19 | 0.53 | 10.18 | 39 |
| 8 | 0.05 | 3.73 | 3.09 | 3.33 | 0.17 | 5.01 | 59 |

**Table 3** Hardness of paint films on wood flooring with different wear values.

| No. | Wear (g) | Hardness (GPa) | | | | CV (%) | n |
|---|---|---|---|---|---|---|---|
| | | Max | Min | Mean | SD | | |
| 1 | 0.16 | 0.34 | 0.24 | 0.27 | 0.03 | 9.31 | 28 |
| 2 | 0.13 | 0.35 | 0.29 | 0.32 | 0.02 | 4.92 | 90 |
| 3 | 0.12 | 0.45 | 0.27 | 0.34 | 0.02 | 7.25 | 44 |
| 4 | 0.11 | 0.40 | 0.29 | 0.33 | 0.03 | 8.02 | 67 |
| 5 | 0.10 | 0.32 | 0.25 | 0.27 | 0.02 | 6.08 | 69 |
| 6 | 0.08 | 0.37 | 0.30 | 0.33 | 0.02 | 4.93 | 64 |
| 7 | 0.08 | 0.50 | 0.23 | 0.29 | 0.05 | 18.23 | 39 |
| 8 | 0.05 | 0.22 | 0.17 | 0.19 | 0.01 | 6.16 | 59 |

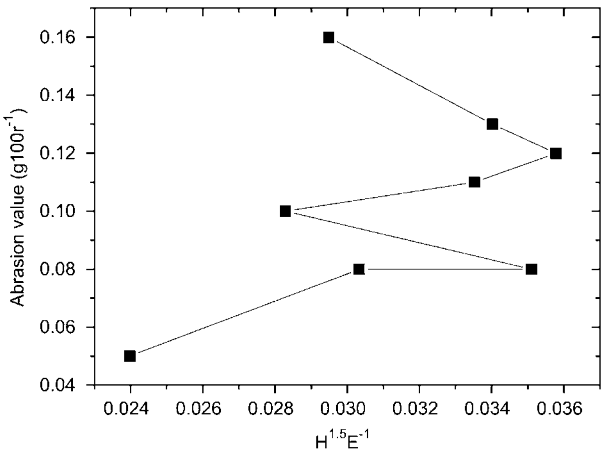


**Figure 3** Correlation between wear value and the index $H^{1.5} E^{-1}$.

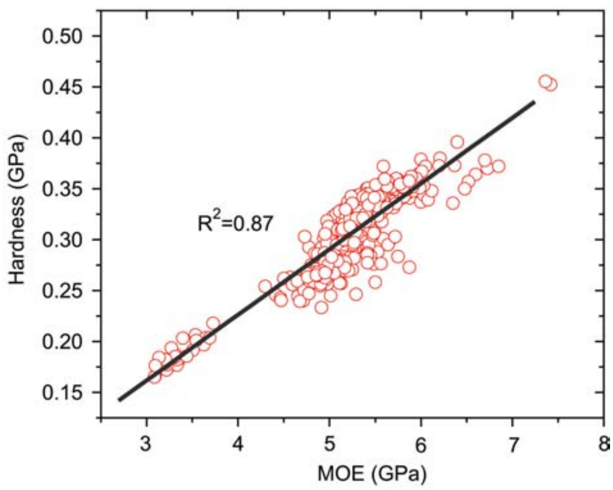


**Figure 4** Relationship between MOE and hardness of the paint film.

resistance of materials (Bhushan 1999). Normally, the higher the ratio, the higher is the wear resistance. However, this rule is not valid for wood flooring (Figure 3). The above discussion indicates that the wear resistance of wood flooring is not determined by only the mechanical properties of the pure paint film. Other factors, such as the type and content of dye filler and the size and quantity of small holes in the paint film, may also significantly affect the wear resistance. Figure 4 shows strong linear correlation ($r^2=0.87$) between the MOE and hardness for all paint films tested. A systematic investigation is needed to quantify the contribution of these factors. However, this basic study shows the great potential of nanoindentation as a possible new standard method for characterization of paint films on wood products.

## Conclusions

*In situ* imaging nanoindentation was successfully used to characterize the mechanical properties of paint films of plywood-based flooring. The technique is very useful for accurate location of indentation zones where the surface is smooth, pure and intact. The average MOE and hardness of the paint film tested were 3.33–5.77 and 0.19–0.34 GPa, respectively. There was a linear correlation between the MOE and hardness of paint films, but neither the MOE nor the hardness was directly correlated to the macro wear resistance of the wood flooring. The wear resistance was influenced by properties not yet investigated.

## Acknowledgments

The financial support of the National Natural and Science Foundation of China (project numbers 30400337 and 30371125) and the “948” project of State Forestry Administration (2002-45) is gratefully acknowledged. Dr. Leslie H. Groom, Dr. Chung-Y. Hse (Southern Research Station, USDA) and Dr. Todd F. Shupe (Louisiana State University, USA) are greatly appreciated for revision of the manuscript.

## References

Bhushan, B. Principles and Applications of Tribology. John Wiley & Sons, New York, 1999.

Catledge, S.A., Borham, J., Vohra, Y.K. (2002) Nanoindentation hardness and adhesion investigations of vapor deposited nanostructured diamond films. J. Appl. Phys. 91:5347–5352.

Chen, L.Q., Zhao, M.H., Zhang, T.Y. (2001) [The testing method of mechanical properties of thin films]. J. Mech. Strength 23:413–429 (in Chinese).

Fischer-Cripps, A.C. Nanoindentation, 2nd ed. Springer-Verlag, New York, 2004.

Gindl, W., Gupta, H.S. (2002) Lignification of spruce tracheids secondary cell wall related to longitudinal hardness and modulus of elasticity using nanoindentation. Can. J. Bot. 80: 1029–1033.

Gindl, W., Gupta, H.S., Schöberl, T., Lichtenegger, H.C., Fratzl, P. (2004) Mechanical properties of spruce wood cell walls by nanoindentation. Appl. Phys. A 79:2069–2073.

Jiang, Z.H., Yu, Y., Fei, B.H., Zhang, T.H. (2004) [Using nanoindentation technique to determine the elastic modulus and hardness of tracheids secondary wall]. Sci. Silv. Sin. 40: 113–118 (in Chinese).

Oliver, W.C., Pharr, G.M. (1992) An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments. J. Mater. Res. 7:1564–1583.

Wimmer, R., Lucas, B.N. (1997) Comparing mechanical properties of secondary wall and cell corner middle lamella in spruce wood. IAWA 18:77–88.

Wimmer, R., Lucas, B.N., Tsui, T.Y., Oliver, W.C. (1997a) Longitudinal hardness and Young’s modulus of spruce tracheid secondary walls using nanoindentation technique. Wood Sci. Technol. 31:131–141.

Received January 27, 2006. Accepted August 16, 2006.