Mechanics Transport Communications
Academic journal

ISSN 1312-3823
volume 11, issue 3, 2013
article № 0849
http://www.mtc-aj.com

MICROINDENTATION INVESTIGATION OF BALSA WOOD

Valentin Gaydarov[1], Krasimira Soukupova[2], Galina Zamfirova[1]
v_gaydarov@yahoo.com, gzamfirova@mail.bg

[1] Todor Kableshkov University of Transport, Sofia, BULGARIA
[2] Inst. of Chemical Process Fundamentals of the ASCR, CZECH REPUBLIC

Key words: *balsa wood, microindentation, total microhardness*

Abstract: *The samples tested were of raw wood and such treated by swelling in water and dried in various temperature regimes. Measurements were performed on a microhardness tester mhp-160 kit to a NU-2 microscope and on a dynamic ultra micro hardness equipment DUH-211S. Therefore we used a methodology for determining the so-called total microhardness (MHT), enabling measurement of the imprint in loaded state that was developed by our research group. The modern depth-sensing indentation method (DSI), consists of obtaining the data from load-penetration (indentation) curves. However, these results showed a large dispersion because of the high structural heterogeneity of the material but permit a direct detection of cavities which are often in the dimensions range of the penetrating pyramid. It has been found that the treated samples have lower total resistance to penetration and that increasing the penetration depth increases the microhardness. The latter is associated with a local material lute in the process of penetration. A hypothesis based on changes in the hydrogen bonds between cellulose molecules during the treatment has been proposed for the explanation of the experimental results.*

INTRODUCTION

Wood is a complex, natural product. Trees are classified into two groups as hardwoods and softwoods. The difference is in the cellular structure of the wood which is very complicated. Balsa wood is classified as hardwoods timber, because of its structure not because of its real hardness.

The balsa tree is the fastest growing tree in the world. Its lumber is very soft and light, with a open grain. The density of dry balsa wood ranges about (160±100) kg/m³. The light weight of the wood is because after water removing large porous remain.

The microindentation experiment we have developed was first of all for metals then for polymers and composite materials. Nevertheless this work aims at demonstrating the applicability of the method to the characterization of wood materials as well.

MATERIAL AND SAMPLE PREPARATION

Samples of balsa wood were subjected to investigation. Its structure characterizes by different cell types: small wood fibres, large cells which function is as pipes that move sap up the tree and cells used to store food (Fig.1) [1]. Fig.2 shows the structure of the cell and how the cellulose molecules are organized and incorporated into the cell wall.

Cellulose microfibrils have disordered (amorphous) regions and well uncluttered (crystalline) regions. The presence of many hydrogen bonds within and between cellulose molecules is characteristic for the crystalline region. They play very important role for the mechanical properties [2].

Four balsa wood samples in a disk shape were investigated. The disks with dimensions 8mm in diameter and thickness 2mm were cut perpendicular to the fiber direction.

- Sample 1 was an original untreated sample;
- Sample 2 was at first swollen in water and then dried slowly at 60 ^{o}C-102 ^{o}C for six days;
- Sample 3 was at first swollen in water and then fast dried at 102 ^{o}C for one day;
- Sample 4 was swollen and dried several times.

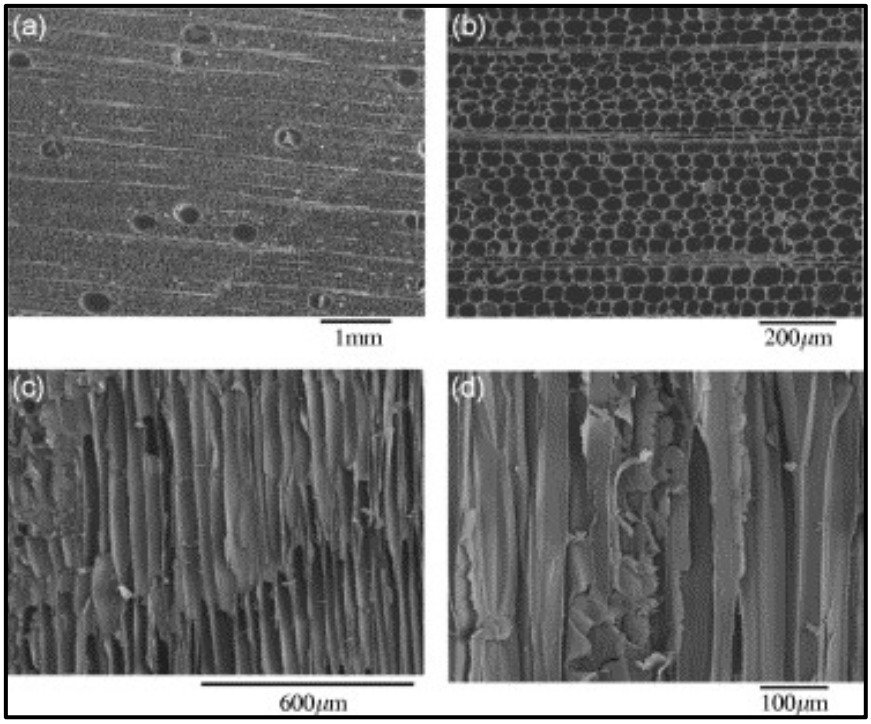


Fig.1 SEM micrographs showing the typical (a, b) across the grain and (c, d) along the grain cross-sections of balsa wood [3]

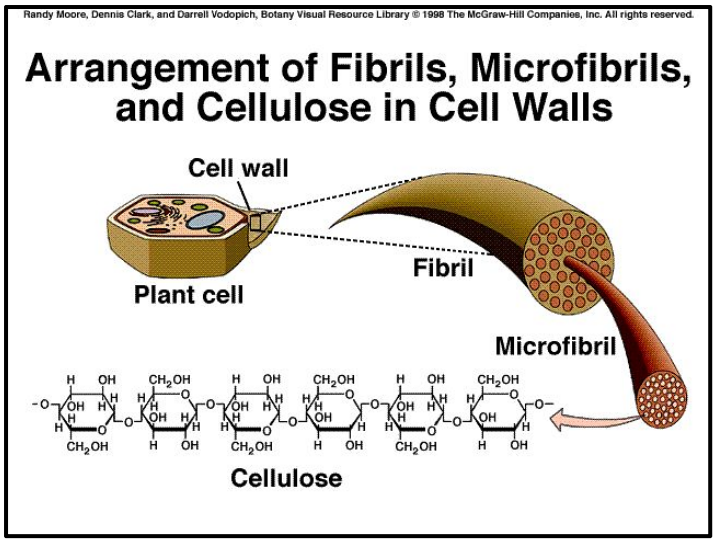


Fig.2 Structure of the cell walls (according Randy Moore and all)

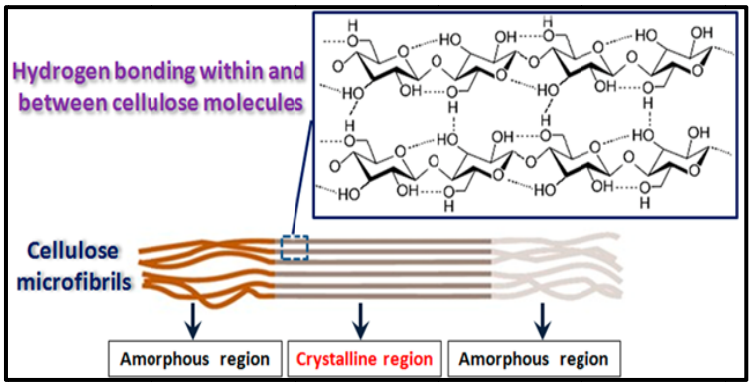


Fig.3 Molecular and supramolecular structutre of cellulose microfibriles

INVESTIGATION METHODS

Two methods and devises were used for microindentation test:

1. A Vickers microhardness device (mhp-160 for a UN–2 microscope) was used. The indenter was a regular square diamond pyramid, with top angle 136º. This devise was construed for providing classical, conventional indentation tests based on direct measurements of the residual imprint left on the sample surface after the load removal. In this case because of very high elasticity of the sample and impossibility to determine the imprint sizes standard Vickers microhardnes was not measured.

We applied microindentation tests developed by our scientific group consisting of the following [4]. The device construction allowed measurements of the indentation depth in loaded state using some constant of the device. The load was hanging and loading process was carried out by raising the table of the microscope. Determination of the so called total microhardness (MHT) analogically to Vickers microhardness was determined according the formula:

(1) $MHT = kP/49h^2$,

where (P) is the applied load, (h) indentation depth in loaded state and (k) is a constant dependent on the geometry of the pyramid.

The magnitude thus defined can be considered as a measure for the local total material resistance against penetration and is related to the total deformation, including elastic, plastic and viscoelastic components. Two loads were applied-100 mN and 200 mN. The measurements for every sample at constant load were carried out and the average value was taken.

2. Dynamic ultra micro hardness tester DUH-211S with applied load in the range of 0.2 mN to 2000 mN uses indentation methods based on the measurement of load–displacement curves at constant loading speed. The method is known as a depth-sensing indentation (DSI) or instrumented indentation testing (IIT). Typical trend of the load–displacement curves involves loading and unloading parts. The measurements were performed at a maximal load of 10 mN, loading speed 0.488 mN/s and at room temperature.

RESULTS AND DISCUSSIONS

The diagram on Fig.4 presents the result for the total microhardness obtained at applied load 100 mN and 200 mN and measured on the device mhp –160.

As seen, the increase in the applied loads, respectively indentation depth, leads to an increase in the total microhardness. It is a typical trend for this characteristic and results from its physical nature. All microhardness characteristics exhibit the mechanical behavior not only of the layer till which the indenter has reached, but the average properties of the whole

material trough which indenter passes. Furthermore, MHT includes not only the resistance against plastic deformation, but also the resistance against elastic deformation. So, the increasing the depth indentation gives rise to an effect similar to the resistance of the compressed spring - the more the compression the stronger is the resistance.

All treated samples have a lower MHT value, i.e. lower total resistance to deformation. This effect could be assigned to the complicated processes taking place during swelling and drying. Since the wood cells, consisting predominately of cellulose, are hydrophilic, wood is heavily affected by water and moisture. Water in wood exists in two forms - free water and bound water. Free water exists as a liquid and vapor in the cell cavities. Bound water is a part of the cell wall materials. The cell and cell walls fill with water which causes expanding of the material. Some of the hydrogen bonds between polymer chains in the crystalline areas of the cellulose microfibrils can break. As water polar molecules are small they form hydrogen bonds with the polymer chains and can get in between the cellulose chains. This softens the cellulose microfibrils as they are no longer so strongly bonded to each other [1]. As a consequence a deterioration of all mechanical properties including the rigidity of wood takes place.

When wet wood dries, free water leaves the cell cavities first and the wood does not shrink. After all free water is gone and only the bound water remains, the cell has reached its fiber saturation point (fsp) [5]. At this point, no water is present in the cell cavities but the cell wall is completely saturated. This process takes place at a cell level. At any given time some cells in a wood product may be in the fsp state while others are not. As wood is dried further, the bound water leaves the cell wall, and cells start to lose moisture below the fsp. When water leaves the microfibrils get closer to each other, and the material shrinks.

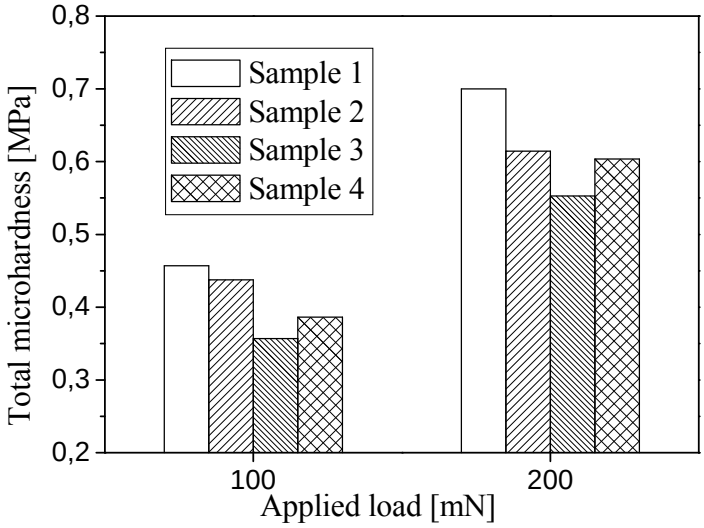
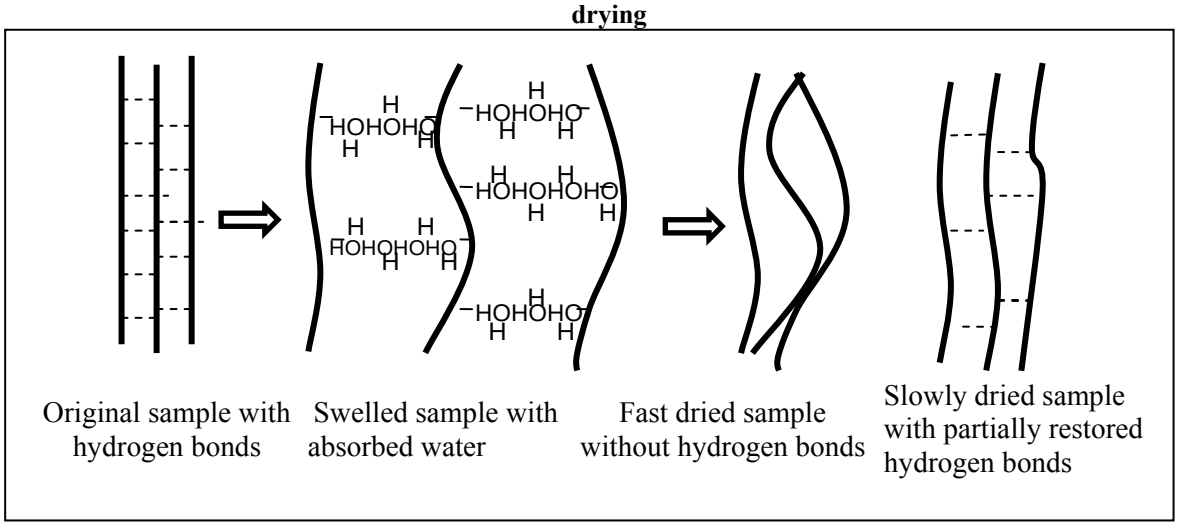


Fig.4 Total microhardnes for the samples measured applying 100 mN and 200 mN

In our case the sample fast dried has lowest hardness. It could be supposed that after swelling the hydrogen bonds between the cellulose molecules are destroyed and the subsequent fast drying at high temperature does not allow restoring of the hydrogen bonds between the polymer molecules. Consequently the crystalline areas are reduced drastically. When drying is slow there is time enough to recover partially the original structure. Fig.5 shows schematically the above described structural changes in cellulose after swelling and drying at a fast and slow regime. The sample which is swollen and dried several times occupies the medium position what concerns to his structural changes and hardness.

Samples measured by DUH-211S give very large dissipation of the results and differences in the shape of indentation curves for one and the same sample. It is because this device allows a maximal indentation depth up to 12 μm. For soft materials as balsa wood the maximal applied load is about one order of magnitude lower and the indentation size smaller than the measured by hardness tester mhp-160, respectively. So, the dimensions of the indentation imprints are in the same range as the structural inhomogeneity of the wood and the measurement is sensitive to it. When the indentation curve has no monotonic increase in its loading part, this is an indicator for inhomogeneity of the sample. A steeper section shows that the indenter passes through a more compact zone, and a slanting section- in cavities or areas of low density. Even the depth and the dimension of the cavity in the penetration area could be determined. As marked with a solid line for the example in the case illustrated in Fig.6 there is some cavity situated in the depth around 3 μm under the surface and with dimensions around 4 μm in direction perpendicular to surface.

Fig.5 Scheme of the structural changes in cellulose after swelling and drying



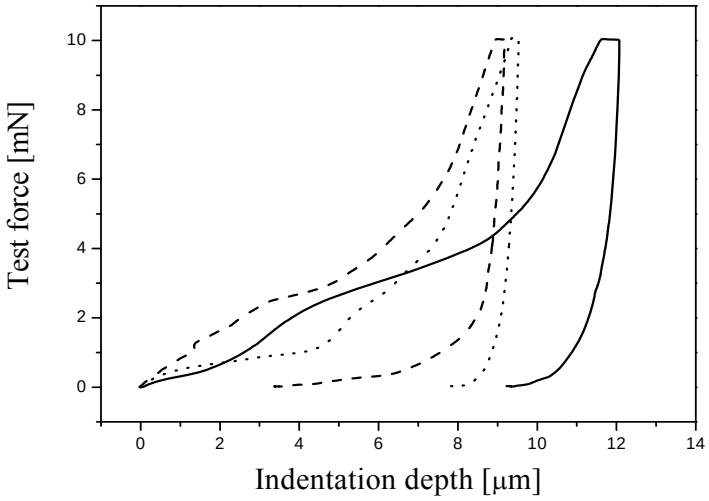


Fig.6 Indentation curves for sample 1

CONCLUSIONS

1. Two nonstandard approaches to studying nonhomogeneous material were suggested:
   - first, based on measurements with a standard Vickers microhardness tester and applying a method for total microhardness determination previously developed by our scientific team.
   - second, based on obtaining the data from indentation curves (depth-sensing indentation (DSI))

   The advantages, possibilities and limits of application of the approaches were discussed.
2. It was established that the samples treated by swelling and drying exhibit poorer mechanic characteristics. We suppose that it could be attributed to distortions of the hydrogen bonds between cellulose molecules during the swelling and non-recuperation or partially recuperation after drying.
3. Depth-sensing indentation is not an appropriate method for studying mechanical properties of micro-nonhomogeneous materials but it allows obtaining approximate information about the dimensions of cavities in the material.

REFERENCES:

[1]. Драганова Р., Химия на дървесината, ДИ „Техника”, София, 1976

[2]. Bhuiyan Md., Hirai N. and Sobue N., Changes of crystallinity in wood cellulose by heat treatment under dried and moist conditions, J. of Wood Science, Vol. 46, Issue 6, pp 431-436, 2000

[3]. Ravichandran G. and Vural M., Failure mode transition and energy dissipation in naturally occurring composites, Composites Part B: Engineering, Volume 35, Issues 6–8, pp 639–646, 2004

[4]. Zamfirova G. and Dimitrova A., Some Methodological Contributions to the Vickers Microhardness Technique, Polymer Testing, 19, pp 533-542, 2000

[5]. Ahlren P., Goring D. and Wood J., The fiber saturation point of various morphological subdivisions of Douglas-fir and aspen wood, Wood Sci. and Technology, Vol. 6(2), pp 81-84, 1972

МИКРОИНДЕНТАЦИОННО ИЗСЛЕДВАНЕ НА ДЪРВЕСИНА ОТ БАЛСОВО ДЪРВО

В. Гайдаров[1], К. Скоупова[2], Г. Замфирова[1]
v_gaydarov@yahoo.com, gzamfirova@mail.bg

[1]ВТУ „Тодор Каблешков", София,
БЪЛГАРИЯ
[2]Институт по фундаментални химични процеси, Прага,
ЧЕШКА РЕПУБЛИКА

Ключови думи: *балсово дърво, микроиндентация, тотална микротвърдост*

Резюме: *Изследвани са образци от необработена дървесина и такива третирани чрез набъбване във вода и изсушени при различни температурни режими. Измерванията са проведени на микротвърдомер mhp-160, окомплектовка към микроскоп NU-2 и ултрамикротвърдомер DUH-211S. Използвана е разработена от нас методика за определяне на т.н. тотална микротвърдост (МНТ), даваща възможност за измерване в натоварено състояние. Съвременният метод DSI (Depth sensing indentation), състоящ се в построяване на индентационни криви натоварване-проникване, показва голямо разсейване на резултатите, поради високата структурна неоднородност на материала, но позволява пряко констатиране на кухините, които понякога са от порядъка на размерите на проникващата пирамида. Установено е, че третираните образци имат по-малко общо съпротивление срещу деформация и че с увеличаване дълбочината на проникване микротвърдостта нараства. Последното е свързано с локално уплътняване в процеса на пенетрация. За обяснение на експеримента са предложени хипотези за структурните промени в дървесината, основаващи се на факта, че между целулозните молекули има водородни връзки, които се променят при третиране.*