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TEXTURE CHANGES IN THERMALLY MODIFIED WOOD

ZMIANY TEKSTURY W DREWNIE MODYFIKOWANYM TERMICZNIE

Thermal modification of wood is performed in order to improve properties of the material, e.g. the dimensional stability, mechanical hardness, biological resistance of wood etc. The process is considered as environmentally friendly as it is made without adding any toxic chemicals. The dimensional stability and biological resistance depend primary on hygroscopic behavior of wood, which is closely related to submicroscopic organization of cellulose. The microfibril angle (MFA) has been recognized as the most obvious parameter describing the organization. However, the traditional application of wide angle, small angle and ultra small angle X ray scattering methods did not allow to observe distinct changes in the MFA distribution of thermally modified wood. In our previous studies we have successfully applied the Orientation Distribution Function (ODF) for quantifying the crystallographic changes of the ultrastructure caused by air parameter variations. Therefore, the same concept is applied in the present study to describe the changes after heat treatment.

The analysis was performed for poplar and beech wood industrially modified at 195°C. The ODF was obtained from X ray diffraction measurements. Sets of selected pole figures of cellulose were registered by the Schulz back reflection technique. The experimental pole figures were the input data for texture analysis. The ADC method was used to calculate the 3D texture function, here the ODF. The rearrangement of the crystalline organization of the thermally modified wood was manifested by a decrease of the maximum values of texture components as well as texture index. The dominating texture components were also changed into (3 -1 1)[1 3 0] and (1 0 0)[0 -1 0] for beech and poplar wood, respectively. The identified changes of the texture are clearly visible in the inverse pole figures of the tangential and radial anatomical directions.

Modyfikację termiczną drewna prowadzi się w celu poprawy właściwości tego materiału, np. stabilności wymiarowej, twardości, odporności biologicznej itp. Proces ten uważany jest za przyjazny dla środowiska ze względu na to, że odbywa się on bez konieczności dodawania toksycznych związków chemicznych. Stabilność wymiarowa oraz odporność biologiczna zależą przede wszystkim od właściwości higroskopijnych drewna, które są ściśle związane z submikroskopowym uporządkowaniem celulozy. Średni kąt mikrofibryl został uznany za najbardziej oczywisty parametr opisujący uporządkowanie. Jednakże typowe zastosowanie metod szeroko-, nisko- i ultra-nisko-kątowego rozpraszania rentgenowskiego nie pozwala zaobserwować istotnych zmian w wartościach średniego kąta mikrofibryl po modyfikacji termicznej drewna. W naszych poprzednich pracach wykazaliśmy, że Funkcja Rozkładu Orientacji (FRO) może być wykorzystana do opisu ilościowego zmian krystalograficznych ultrastruktury drewna spowodowanej zmianami zmiennymi parametrami powietrza otoczenia. Tym samym ta sama metoda została wykorzystana w obecnej pracy do opisu zmian ultrastruktury po modyfikacji termicznej.

Badania prowadzono dla drewna topoli i buka, które modyfikowano w warunkach przemysłowych w temperaturze 195°C. FRO otrzymano po wykonaniu pomiarów dyfrakcyjnych promieniowania X. Zbiór wybranych figur biegunowych celulozy zmierzono wykorzystując metodę odbić wstecznych Schulza. Zarejestrowane figury biegunowe stanowiły dane wejściowe do analizy tekstury. W celu wyznaczenia trójwymiarowej funkcji tekstury (tj. FRO) wykorzystano metodę ADC. Zmiany uporządkowania krystalicznego celulozy po modyfikacji termicznej obserwowano w postaci zmniejszenia się maksymalnych wartości składowych oraz indeksu tekstury. Zmianie uległy również dominujące składowe tekstury na (3 -1 1)[1 3 0] i (1 0 0)[0 -1 0] odpowiednio dla drewna bukowego i topolowego. Zidentyfikowane zmiany tekstury są również jednoznacznie obserwowane na odwrotnych figurach biegunowych dla promieniowego oraz stycznego kierunku anatomicznego.

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1. Introduction

Thermal modification of wood has been considered as an ecological method improving wood properties, e.g. biological resistance and dimensional stability. The ecological character of the process is primary related to the fact that the desired improvement of the properties is not caused by wood impregnation with chemicals. The only factors influencing the changes are heat and steam. The process itself consists in heating the wood material to temperature of at least 180°C under steam atmosphere (ThermoWood® Handbook 2003). The application of steam during the thermal modification primary prevents the material from burning. The modification induces changes in the chemical composition of wood, i.e. the extractives content is reduced and hemicellulose is partially degraded (Andersson et al. 2005, Metsä-Kortelainen et al. 2006). During the thermal treatment also the highly anisotropic microstructure of wood is changed. The increase in the degree of crystallinity is reported, which may be related to additional crystallization in amorphous regions as well as reorganization of the already arranged areas (Bhuiyan et al. 2000, Andersson et al. 2005, Akgül et al. 2006). Moreover, there was observed increase in the width of crystallites without significant change of values of the mean microfible angle (MFA), which is defined as the angular deviation of the microfibril axes from the longitudinal direction of the cell.

The microstructure changes of thermally modified wood were not investigated in the 3-D domain. However, it was already proved that the Orientation Distribution Function (ODF) can be applied in the case of wood. The application of the ODF required modifications of the approach due to the specific features of the material (Bonarski and Olek 2006, Olek and Bonarski 2006). The qualitative and quantitative analyses of the texture function using the complete and inverse pole figures were also not reported. Also the misorientation parameters being sensitive measures of the wood anisotropic microstructure (Bonarski and Olek 2007) were not applied to determine the changes after thermal modification. The objective of the present study is to use the 3-D measures of the wood microstructure in order to determine the influence of thermal modification.

2. Material

The investigated material was obtained from thermally modified wood of the following species European beech (*Fagus sylvatica* L.) and poplar (*Populus* spp.). The modification was performed in industrial conditions at the temperature of 190°C. The material was equilibrated to the moisture content level of ca. 10% after the modification. The samples in the shape of rectangular prisms were cut from the equilibrated wood. The thickness of samples corresponded to the radial anatomical direction and was equal to ca. 6 mm. The radial plane of samples was exposed to X ray diffraction.

3. Experimental

The X ray diffraction pattern was used to select reflections 101, 020 and 004 for which the four sets of pole figures were measured for European beech and poplar wood before and after the thermal modification. Each data set consisted of the experimental pole figures of the following planes (101), (010) and (001) which were registered using the X-ray diffraction technique for the following ranges of angles – radial $\alpha = 0° \ldots 75°$ and azimuthal $\beta = 0° \ldots 360°$ with steps $\Delta\alpha = 5°$ and $\Delta\beta = 5°$, respectively. The measurements were made with the Philips X'Pert system equipped with the texture goniometer ATC-3. The filtered X ray radiation CoKα ($\lambda = 0.179026$ nm) was used.

The procedures of defocusing correction as well as normalization were applied to the sets of experimental pole figures in order to use them as the input data in the calculating procedure of the texture analysis. The ADC method introduced by Pawlik (1986) was used to calculate the 3D texture functions (here ODFs) for both wood species before and after the modification. The ODFs were used to compute sets of complete pole figures with the radial angle α ranging from 0 to 90° as well as inverse pole figures of sample directions related to the wood anatomical ones. The ADC method was supplemented by the modification taking into account the monoclinic lattice symmetry of the investigated material (Bonarski and Olek 2006). All numerical analyses were made with the software package LaboTex (LaboSoft 2003).

4. Results

The sets of complete pole figures obtained for European beech wood before and after thermal modification are presented in Figure 1. The differences in distributions of lattice planes for wood before and after the modification are clearly visible. In general, the pole figures of wood before the modification reveal much sharper preferred orientation. The maximum value identified in the (010) pole figure was 6.3 for the wood before the modification, while the maximum reached after the modification was only 3.2. The inverse pole figures of the longitudinal direction (L) were also calculated for European beech wood (Fig. 2) in order to present the texture

function in the space coordinates related to the wood anatomical directions, as well as to show the differences in the space arrangement of cellulose. The maximum intensity of the inverse pole figure of the longitudinal direction before the modification was 3.4 and higher than the intensity of the modified wood (the maximum of only 2.4).

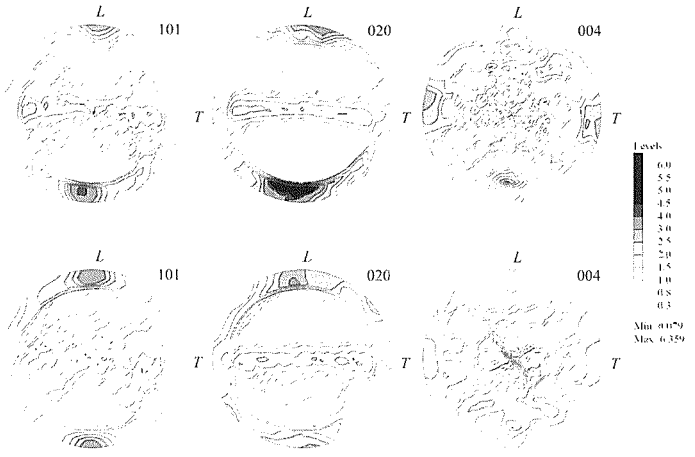

Fig. 1. Complete pole figures of European beech wood before (top figures) and after (bottom figures) thermal modification

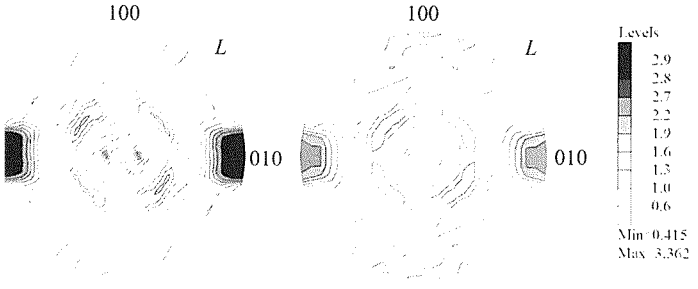

Fig. 2. Inverse pole figures of the longitudinal direction (*L*) of European beech wood before (left) and after (right) thermal modification. 100 and 010 are the Miller's indices of the crystallographic axes of the cellulose lattice

The same procedure was also applied for poplar wood. The results of texture analysis in the form of two sets of complete pole figures are presented in Fig. 3. The inverse pole figures of the *L* direction are shown in Fig. 4. It was found that the maximum values in the pole figure (010) before and after thermal modification were 10.1 and 8.3, respectively. Also the maximum values in the inverse pole figures of the *L* direction were identified as equal to 5.5 and 6.2 for unmodified and modified wood, respectively.

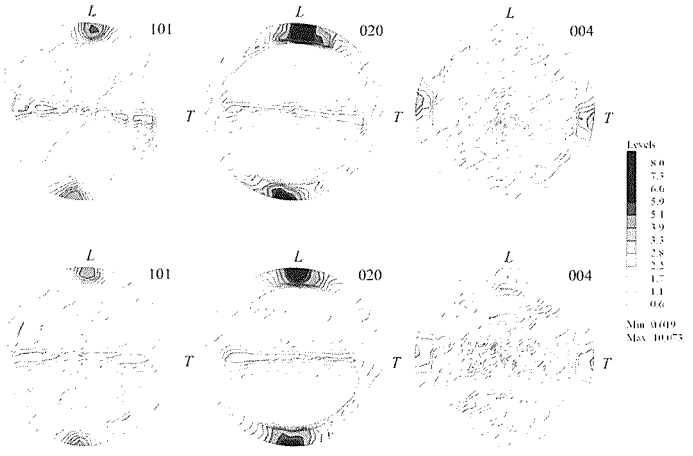

Fig. 3. Complete pole figures of poplar wood before (top figures) and after (bottom figures) thermal modification

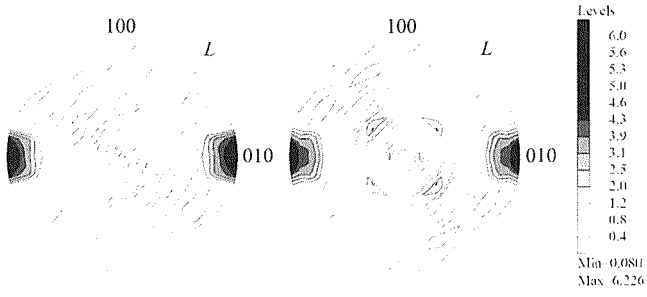

Fig. 4. Inverse pole figures of the longitudinal direction (*L*) of poplar wood before (left) and after (right) thermal modification. 100 and 010 are the Miller's indices of the crystallographic axes of the cellulose lattice

The maximum values of the ODF identified in the (010) and inverse pole figures were also related to the values of the texture index (*J*) obtained by the use of the discrete method ADC (LaboSoft 2003). The related values are presented in Table 1. The obtained results clearly show changes in wood microstructure of both wood species before and after thermal modification.

The dominating texture components also changed its volume fractions during the thermal modification (Table 2). In all investigated samples the dominating orientation was {1 0 0}<0 -4 1> for which high volume fractions were observed before the thermal modification. The other orientation component present in both species was {5 -1 2}<-1 -5 0>. The component had some tendency to increase its amount in poplar wood. The thermal modification caused changes in proportions of all components, however, the {1 0 0}<0 -4 1> component was still dominating.

TABLE 1

The maximum values of texture functions and texture index obtained for investigated wood species before and after thermal modification

Wood sample	Maximum ODF	Texture index (J)	Maximum value in	
			(010) pole figure	Inverse pole figure of the L direction
European beech before modification	23.4	2.46	6.3	3.4
European beech after modification	6.1	1.19	3.2	2.4
Poplar before modification	42.2	4.02	10.1	5.5
Poplar after modification	15.8	2.59	8.3	6.2

TABLE 2

Dominating texture components {hkl}<uvw> and their estimated volume fractions in wood before and after thermal modification

Wood sample	Volume fractions of {hkl}<uvw> orientations [%]	
	{1 0 0}<0 -4 1>	{5 -1 2}<-1 -5 0>
European beech before modification	39	1
European beech after modification	22	1
Poplar before modification	30	2
Poplar after modification	19	3

5. Discussion

The presented sets of the (010) pole figures as well as inverse pole figures of the L direction showed a tendency of the investigated wood to change its space arrangement after the thermal modification. The investigated microstructure tends to obtain more favorable state because of mechanical strength. The presented results revealed strong preferred orientation close to the (010) lattice direction in both wood species. However, the orientation had different extent. The investigated texture was essentially attenuated as the result of the thermal modification. It was clearly observed when compared the corresponding values of the ODF maximum and texture index (see Table 1 and Figures 1 to 4).

The reported changes of the microstructure of the investigated wood species are reflected also in quantitative relations between identified texture components. The dominating {1 0 0}<0 -4 1> orientation exhibits the distinct tendency to decrease during the modification process in the both wood species. The opposite trend was observed for other identified orientation, i.e. for {5 -1 2}<-1 -5 0> (Table 2). However, due to the relatively small amount of the component the changes can be credibly noticed for poplar wood only.

6. Conclusions

Regarding the character of changes of the density distribution of the (010) poles as well as numerical values of the texture function it is justified to state that the thermal modification essentially alters cellulose texture. The changes seem to lead to increase of elastic properties of the modified wood. Moreover, the altered texture can be related to improved hygroscopic properties of modified wood. Among the two investigated wood species, European beech is more susceptible for the microstructure changes as caused by the thermal modification.

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