Wood Sci Technol (2015) 49:319–330
DOI 10.1007/s00226-014-0691-7

ORIGINAL

XPS depth profile of plasma-activated surface of beech wood (*Fagus sylvatica*) and its impact on polyvinyl acetate tensile shear bond strength

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Received: 23 April 2014 / Published online: 5 December 2014
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Abstract High surface selectivity of atmospheric pressure plasma treatment was demonstrated experimentally by XPS depth profile measurement of plasma-activated beech wood. Wood surface activated by diffuse coplanar surface barrier discharge was sequentially sputtered by Ar^+ ion beam followed by immediate XPS analysis of freshly uncovered surface. According to the assessment, less than 330 nm of sputtered material was sufficient for complete removal of all plasma-formed functional groups. For the sake of practical implications of minimal vertical extent of plasma-mediated changes, the character of tensile shear bond strength improvement of polyvinyl acetate adhesive was examined with respect to its specific mass. A constant additive character of plasma activation to the bond strength was observed within the examined range of adhesive-specific mass.

Introduction

The growing popularity of polyvinyl acetate-based adhesives to bond indoor products is driven mainly by concerns about the health and environmental hazards associated with formaldehyde and volatile organic compounds (VOC) emission of competing adhesive formulations. Nevertheless, since polyvinyl acetate is a water-based adhesive, its use for bonding of generally nonpolar and hence non-wetting wooden surfaces suffers from inadequate contact of adhesive and adherent on atomic level. Only weak van der Walls interactions (such as London forces) instead of desired hydrogen bonds with wood fractions are formed. The situation is

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commonly addressed by improved formulations of adhesives and/or by employing the cold press to overcome the potential barrier of repulsive forces.

An alternative approach, which is nowadays routinely used in polymer printing industry, is the use of nonthermal plasma pretreatment (activation). High-voltage or high-frequency electromagnetic field is used to heat the ambient electrons above the ionizing energy of the surrounding gas. When some appropriate measures are taken to prevent an extensive heat transfer from the high-energy electrons to surrounding gas (such as placing a dielectric barrier between the power electrodes to constrain the lifetime of single plasma channel into the tens of ns), it is possible to design a plasma reactor operating close to room temperature that facilitates a considerable flux of gas-phase atomic and molecular radicals toward the solid surface to provide its desired chemical functionalization. The benefits of using the nonthermal plasma activation lay mainly in its applicability to heat sensitive materials and no need for additional drying steps, but also the possibility to avoid the complex formulation of polyvinyl acetate adhesives and even to affect positively the polyvinyl acetate curing kinetics (Avramidis et al. 2011a) and finally its generally straightforward implementation into the production line.

The work of several research teams within the last two decades demonstrates convincingly the plasma-assisted improvement of wood surface wettability. For plasma treatment done at atmospheric pressure (which offers far better technical feasibility and economy compared to that of low-pressure systems), three distinct types of plasma system configurations were mainly studied: volume dielectric barrier discharge (DBD), surface DBD and plasma jets. Plasma jets offer an excellent efficiency requiring plasma treatment times of only units of seconds (Busnel et al. 2010; Potocnáková et al. 2013). The jet systems suffer from limited cross-section diameter of plasma plume, which makes the treatment of large size areas time-consuming. Volume DBD, sometimes incorrectly called industrial corona, is a standard electrode configuration employed for decades to improve the surface wettability in polymer printing industry. Consequently, this configuration was the first to be tested for activating the wood surfaces. Material is either fed through the discharge gap formed by a pair of plane parallel electrodes (Podgorski et al. 2000; Avramidis et al. 2011b) or the material itself works as a return electrode for pulsed discharge current (Rehn et al. 2003). Treatment times of units of seconds are needed to achieve complete wettability over substantially wider areas compared to plasma jets. For sufficiently flat material surfaces, it is possible to address the intrinsic shortcomings of volume DBD, which are an excessively high-voltage requirement and resistive power losses in material bulk, by employing the configuration of surface DBD. Here, both polarity electrodes are placed underneath the dielectric panel in a way that allows formation of nonthermal plasma on its opposite side. Dielectric panel covered by thin (<0.5 mm) plasma sheath is consequently brought into contact with treated wood surface. There are virtually no limits to the thickness or electrical conductivity of the wood piece. The work by Odrášková et al. (2008) on so-called diffuse coplanar surface barrier discharge (DCSBD), which is a particular type of surface DBD being developed by the authors' team and which will be further discussed in this paper, showed almost a threefold improvement in energy efficiency compared to the volume DBD. A more recent study by Lux et al. (2013) on local temperature increase in 1 mm depth below the plasma-treated beech wood surface reports only 10 °C increase upon 9 s plasma treatment and 14 °C increase upon 18 s treatment. Pair comparison between the lateral spreading of sessile droplet and the water absorption to the wooden body reveals a pronounced effect of DCSBD plasma treatment on the former, but almost none (i.e., below a statistical significance) on the latter. This observation supports the hypothesis that the plasma-affected material is located mostly on uppermost wood surfaces. Further supporting evidence for this hypothesis can be drawn from the pair comparison of ATR–FTIR spectra of treated and untreated surfaces by Lux et al. (2013). Only minute spectroscopic changes were recorded, despite the fact of dramatic macroscopic differences of both surfaces. This may be explained by substantially lower volume occupied by plasma-modified material compared to that of evanescence field extending from the infrared beam ATR crystal (i.e., 0.5–5 µm). In this work, X-ray photoelectron spectroscopy (XPS) data of chemical depth profile of plasma-activated beech wood (*Fagus sylvatica*) are presented to provide direct experimental proof of the above-stated hypothesis of low depth of plasma modification.

Besides a pure scientific interest on the mechanism of plasma–wood surface interactions, the proven low extent of plasma-affected volume might have some interesting cost-saving implications for the wood adhesive bonding. The failure of adhesive joints is caused either by rupture within the adhesive itself or at the adhesive–wood interface or within the bulk of wood (cohesive failure). Standard methods of chemical or mechanical surface pretreatment result in the formation of a so-called weak boundary layer (WBL) that contributes to cohesion failure (Stehr and Johansson 2000). At the same time, thermoplastic adhesives (such as polyvinyl acetate) exposed to shear load are able to compensate to some extent the imposed external stress by their elastic deformation. As a consequence, joints prepared with larger amounts of adhesive exhibit higher tensile shear bond strength. To counteract the effect of WBL formation, larger amounts of adhesive must be used. The introduction of atmospheric pressure plasma activation of wood surfaces is reported to improve the bondability or paint adhesion by up to 30 % (Sakata et al. 1993; Rehn et al. 2003; Wolkenhauer et al. 2008; Busnel et al. 2010; Acda et al. 2012). This improvement is generally attributed to the enhanced interactions on adhesive–wood interface. With plasma activation resulting in a minimized formation of WBL, the adhesion strength improvement may be directly converted to the lesser amount of adhesive needed, since less elastic deformation of adhesive is needed to compensate for the weakened cohesive strength of WBL.

To validate this cost-saving hypothesis, tensile shear bond strength of polyvinyl acetate adhesive of various amounts was measured on plasma-activated surfaces.

Materials and methods

Substrate and adhesive

Twelve beech wood (*F. sylvatica*) straight-grained boards of 86 × 7 × 800 mm^3 with a moisture content of 10 ± 1 % were used for testing of bond shear strength.

Specimens were stored under conditions of 24 °C and 65 % relative humidity in the conditioning chamber Sanyo MTH 2400. Not more than 24 h before the treatment, board surfaces were sanded with 100-grit sandpaper and all dust was carefully removed by air blast. For XPS measurements, specimens were cut to $10 \times 7 \times 10$ mm^3. Polyvinyl acetate adhesive Duvilax LS 50 (Duslo a.s. Slovakia) of durability class D2 (according to EN 204:1991) was used for evaluation of tensile shear strength.

Plasma treatment

Plasma activation was done at atmospheric pressure air on in-house build DCSBD equipped with driving pulley to facilitate the movement of wooden boards at adjustable speed in close contact with the layer of generated plasma (Fig. 1). The DCSBD electrode element consisted of 16 equidistantly spaced conductive electrode strips of alternating polarity, screen-printed on one side of 96 % Al_2O_3 dielectric plate of $228 \times 92 \times 0.6$ mm^3 size. The width of a single conductive strip was 3 mm, and mutual distance of adjacent strips was 1.5 mm. The width of 3 mm was found to provide better treatment uniformity to that of 1.5 mm width used in the past (Odrášková et al. 2008). The screen-printed side of electrode was electrically insulated by circulating transformer oil, which also acted as a cooling medium to dissipate the plasma-generated heat. The system was powered by sinusoidal voltage of 10 kV$_{RMS}$ at 15 kHz and 450 W input power, supplied by Lifetech VF1500 power generator. The speed of the driving pulley was adjusted to a plasma treatment time of 30 s. The choice of this particular treatment time came from previous results of contact angle measurements. It is the double of the time needed to achieve complete wetting by water sessile droplet. In practice, such estimated treatment time is used as a starting point for further treatment time optimization procedure. Small specimens for XPS were treated manually for 30 s. Plasma layer generated above the free ceramic surface takes the form of numerous microfilaments of sub-millimeter diameter and 3 mm in length that are rapidly moving, which gives an illusion of diffuse plasma appearance to the naked eye. Bringing the electrically nonconductive wood specimen into close contact with the ceramic surface converts the shape of plasma microfilaments into plasma strips of real spatial uniformity

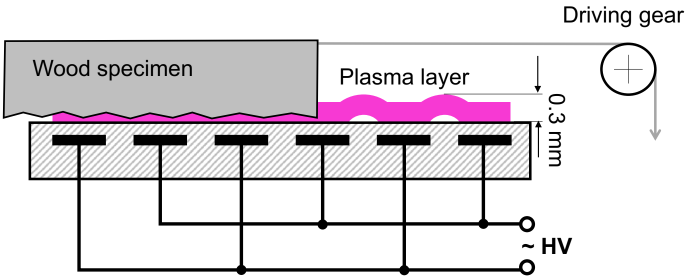

Fig. 1 Schematic of DCSBD plasma treatment reactor (not to scale). Intrinsic roughness of wood surface provides sufficient space for discharge plasma to be formed

following the pattern of power feed electrodes. The visual appearance of the phenomenon can be observed when treating transparent materials such as glass (Homola et al. 2013).

Contact angle measurement

Screening of free surface energy γ_s of initial and plasma-activated wood surface was done by means of the sessile drop technique using the SEE System E (Advex Instruments) device. Surface contact angle of 15 droplets of 2 µl volume was measured for each testing liquid, i.e., water, glycerol and diiodomethane. Assessment of dispersive and polar components of free surface energy was done by regressive Owens–Wendt method—OWRK (Owens and Wendt 1969) using the SEE System software library.

X-ray photoelectron spectroscopy

XPS measurements were done on the ESCALAB 250Xi (ThermoFisher Scientific). The system is equipped with 500 mm Rowland circle monochromator with microfocused Al Kα X-ray source. An X-ray beam with 200 W power and 650 µm spot size diameter was used. This X-ray spot size was found to be sufficient to compensate the natural chemical structure inhomogeneity of wooden surface, so that the signal from various sites of the investigated surface was the same within the statistical error. Positive charge accumulated on the surface was neutralized by electron flood gun. The survey spectra were acquired with pass energy of 50 eV and resolution of 1 eV. High-resolution scans were acquired with pass energy of 20 eV and resolution of 0.05 eV. In order to obtain depth profile, a series of 10 s bursts of Ar^+ ion beam of 200 eV energy were scanned over an area of 3.25×3.25 mm^2. Spectra were referenced to the hydrocarbon type C 1s component set at a binding energy of 284.8 eV. The spectra calibration, processing and fitting routines were done using Avantage software.

Tensile shear strength measurement

Tensile shear strength was determined according to EN 205:2003. The adhesive of known mass (80, 110 and 160 g/m^2) was applied to single-bonded surface within the 3 min from plasma activation and brought immediately into contact with the other plasma-activated board. Similar to that, reference specimens without plasma activation were prepared. Afterward, specimens were pressed in ITALPRESSE SCF/6-S for 24 h under 0.5 MPa at a temperature of 25 °C. After curing, specimens were conditioned at 24 °C and 65 % relative humidity for 6 weeks.

Actual tensile shear strength measurements were done on test specimens of $20 \times 14 \times 100$ mm^3 cut from bonded boards (14 from each board) on which pair of grooves separated by 10 mm was made in compliance with EN 205:2003. Universal tensile strength testing machine ZDM 10/90 (VEB TIW Rauenstein) equipped with Mini MFA 2 (MF GmbH) extensometers was employed. A loading speed of 5 mm/min resulted in bond rupture within the time range of 30–50 s. The tensile shear strength was determined as a ratio between the maximum applied force and 400 mm^2 area of bonded surface.

Results and discussion

Contact angle measurement

Results are summarized in Table 1. Untreated beech wood exhibits poor water wettability with a sessile droplet contact angle of (70 ± 4)°. Calculation from OWRK model suggests that this is due to the small polar component of free surface energy, which is only 8 $\mathrm{mJ/m}^2$. Plasma activation lasting 30 s was able to achieve complete wettability of beech surface by water, i.e., below the contact angle measurement detection limit. The calculation from remaining two test liquids resulted in a value of 21 $\mathrm{mJ/m}^2$ of polar component of surface free energy, which is almost a threefold increase of the initial state. The total free surface energy increased by 24 $\mathrm{mJ/m}^2$.

X-ray photoelectron spectroscopy

Figure 2 shows typical XPS spectra for untreated (A) and plasma-activated (B) beech wood specimens. Corresponding calculated O/C ratios are listed in the first column of Table 2. The C and O occurring at 285.2 and 532.9 eV, respectively, are the predominant species. Each specimen contains traces of same impurities, arising most probably from contamination during specimen preparation. The O/C ratio was found to be 0.29 for untreated specimen. This value is in good agreement with that estimated from the literature data (Inari et al. 2011). Exposure to plasma resulted in more than twofold increase of O/C ratio. The increase of oxygen containing groups is in good agreement with similar XPS studies done on wood (Tóth et al. 2007; Avramidis et al. 2012) as well as with above-presented data on increased polar part of free surface energy. To specify the changes in more detail, peak fitting routines were done. High-resolution scans of C 1s peaks were decomposed into four components for all specimens: C–C/C–H at 285 eV, C–O at 286.6 eV, C=O/O–C–O at 287.9 eV and O–C=O at 289.2 eV. Differences in peak areas indicating the surface changes are shown in Fig. 3 and in the remaining columns of Table 2.

Table 1 Contact angle and free surface energy of specimens

Specimen	Water	Glycerol	Diiodomethane	γ_s ($\mathrm{mJ/m}^2$)	$\gamma_s^{\mathrm{disperse}}$ ($\mathrm{mJ/m}^2$)	$\gamma_s^{\mathrm{polar}}$ ($\mathrm{mJ/m}^2$)
Untreated	(70 ± 4)°	(80 ± 4)°	(51 ± 8)°	36 ± 3	28	8
Plasma-treated (30 s)	–	(22 ± 3)°	(41 ± 5)°	60 ± 3	39	21

Free surface energies of liquids used in calculation ($\mathrm{mJ/m}^2$)—water: $\gamma = 72.8$, $\gamma^d = 21.8$, $\gamma^p = 51.0$; glycerol: $\gamma = 64.0$, $\gamma^d = 34.0$, $\gamma^p = 30.0$; diiodomethane: $\gamma = 50.8$, $\gamma^d = 50.8$, $\gamma^p = 0.0$

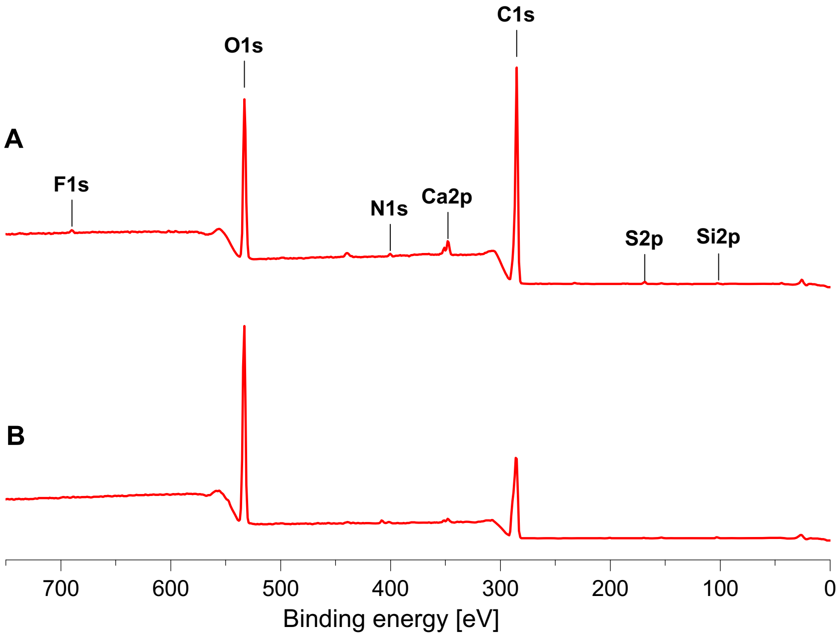

Fig. 2 XPS survey spectra of **a** control specimen; **b** plasma-treated specimen—*uppermost layer*

Table 2 Analysis of XPS spectra

Specimen	Atomic ratio	Relative area of chemical bonds (%)			
	O/C	C–C/C–H	C–O	C=O	O–C=O
Untreated					
Top layer	0.29	68	24	5	3
Plasma-treated specimen					
Top layer	0.62	42	32	19	7
Last (30th) layer	0.29	65	26	5	4

The most significant changes in beech wood resulting from plasma treatment are the decrease of C–C/C–H component and increase of O–C=O chemical bond. For these components, relative areas are changed by a factor of 1.6 and 2, respectively. For C=O only component, the effect of enhancement becomes even stronger: a plasma-treated process enhanced relative area of C=O by nearly a factor of four. The component C–O at 286.6 eV is assumed to be derived mainly from cellulose because of its high stability. The relative area corresponding to this component is practically resistant to plasma treatment. All of these functional groups represent polar bonds which correspond very well with the observed improved wettability by polar liquids such as water.

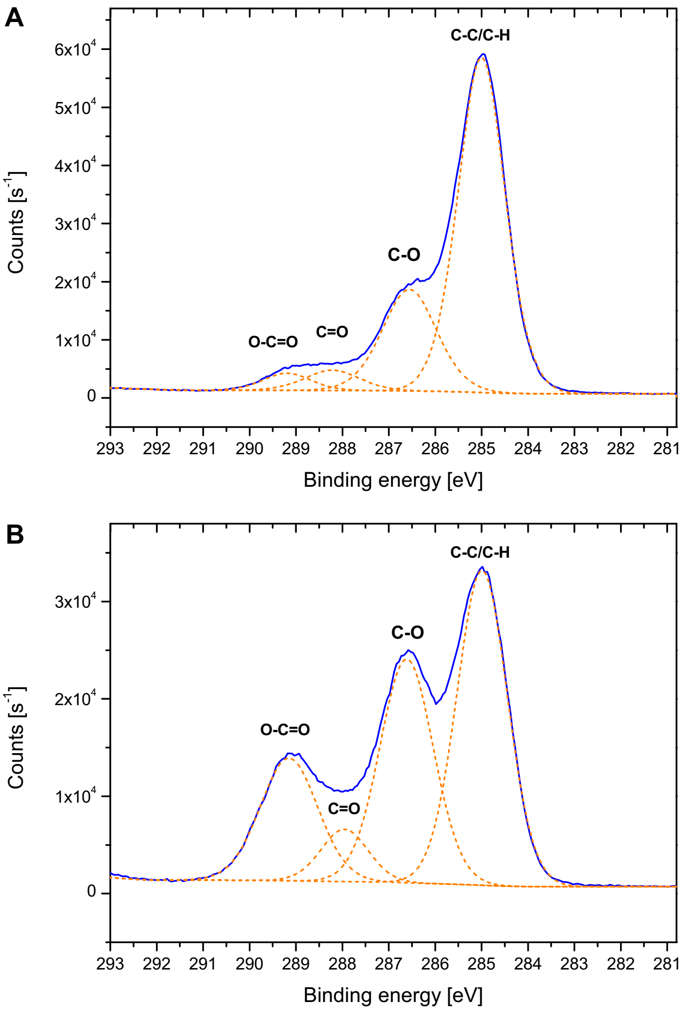

Fig. 3 Deconvoluted C 1s peaks for **a** control specimen; **b** plasma-treated specimen—*uppermost layer*

For depth profile analysis, an Ar^+ ion beam integrated into the present XPS system was employed for serial sputtering steps gently removing already XPS analyzed uppermost wood surface layers to allow an XPS scan of the layer underneath. It was important to choose carefully the ion gun operating parameters, especially the ion beam energy. It has to be as low as possible to minimize the wood internal changes induced by ion collisions cascade, but high enough to provide a reasonable sputtering yield. After 30 sputtering steps, the XPS signal converged to that of plasma untreated (reference) specimen, see Table 2. Further sputtering led to no significant changes in spectrum. The changes in the gradual depth profile of principal chemical bonds are shown by deconvoluted C 1s peak in Fig. 4.

The plot shows that progressive number of sputtering steps results in surface with higher C–C/C–H bond and decay of O–C=O and C=O bond coverage. The concentration of C–O remained virtually unchanged. For the sake of evaluating the effect of ion beam itself on wood compounds' chemical structure stability, the same sputtering experiment was made on plasma untreated specimens. No significant changes in depth profile spectra were observed. The changes in all C 1s components were within the range of ±2 %. In particular, the preferential sputtering of oxygen atoms led to the reduction of C=O component (the most pronounced indicator of plasma activation) from 5 to 3 % after 30 sputtering steps. Hence, ion beam does not interfere with the presented measurement via creating C=O functional groups of its own.

Attempts to determine the depth of sputtered crater by direct experimental observation failed, chiefly due to the intrinsic high surface roughness of wood material, but also due to the intentionally low sputtering yield of Ar^+ ion bombardment. Neither by confocal optical microscopy nor by scanning electron, it was possible to extract unambiguous information on topographic changes from the background surface roughness. Nevertheless, some rough estimation of maximum sputtered depth can be made from below trivial formula for vertical sputtering rate [m/s]:

$$\frac{z}{t} = \frac{M}{\rho N_A e} YIS \qquad (1)$$

where M and ρ stand for molar mass and density of sputtered substance, respectively, N_A for Avogadro constant, I for ion current, S for sputtered area, Y for sputtering yield and e for elementary charge.

According to literature (Yu et al. 2002), sputtering yield of cellulose bombarded by 30 keV Ar^+ beam is $Y = 9.7$ atoms/ion. This relatively high number is mainly

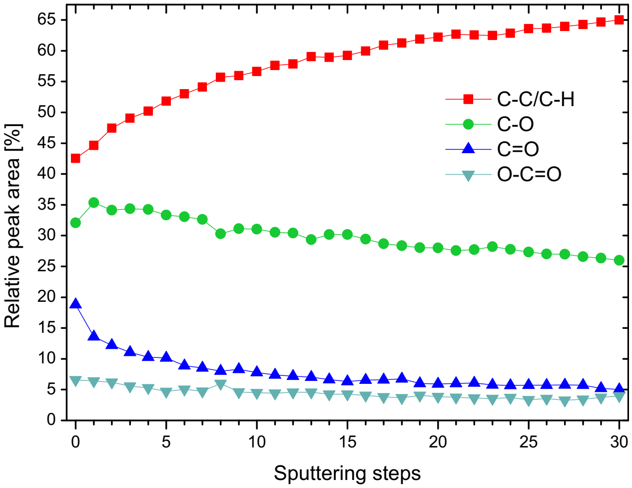

Fig. 4 XPS chemical depth profile of carbon components for plasma-treated beech wood

Table 3 Tensile shear bond strength of polyvinyl acetate joint

	160 g/m^2		110 g/m^2		80 g/m^2	
	Untreated	Plasma	Untreated	Plasma	Untreated	Plasma
Shear bond strength (N/mm^2)	8.2 ± 2.3	9.3 ± 1.1	5.8 ± 2.4	7.0 ± 2.2	2.9 ± 1.3	3.7 ± 1.5
Difference (N/mm^2)	+1.1		+1.2		+0.8	
p value	0.067		0.056		0.32	
Number of valid specimens	19	13	31	26	7	9

due to the high sputtering yield of oxygen atoms with low surface binding energy of 0.075 eV, which are therefore sputtered preferentially. To the authors' knowledge, there are no experimental data on sputtering yield of cellulose for lower energy ions (i.e., 200 eV in this experiment), but in general it should be lower. The magnitude of this reduction can be estimated from the known data of polyethylene terephthalate (which has a close stoichiometry to cellulose) to be at least of factor five (Kormunda and Pavlik 2010). Inserting values of $Y = 2$, $I = 5$ mA, $\rho = 1.6$ g/cm^3 for cellulose $M = 16$ g/mol for oxygen into formula (1) gives the vertical sputtering rate of 1.1 nm/s that corresponds to 330-nm-deep crater after the whole sputtering sequence. It should be emphasized however that this estimation should be used only as an upper limit value. In reality, owing to the intrinsically high wood surface roughness, the depth sputtered profile must be lower owing to the differences in local angels of incidence. In addition to this, low-energy ion fluence would be affected by deposited electrical charge on nonconductive wood surface, and finally, the sputtering yield would become continuously lower as oxygen atoms are being depleted from the surface. Nevertheless, this upper boundary estimation of 330 nm is smaller than the cell wall thickness of *F. sylvatica* wood (Koch and Kleist 2001). The authors therefore dare to conclude that the effect of plasma activation is indeed localized only on the uppermost layer of wood surface structure.

Tensile shear bond strength measurement

Measurement data of tensile shear bond strength are summarized in Table 3. In line with expectations, higher quantity of adhesive resulted in higher value of shear bond strength. Plasma activation in all three investigated cases increased the average shear bond strength by a surprisingly constant additive value of 1 N/mm^2. Since all collected experimental data were of normal distribution, it was possible to calculate the *p* value of Student's *t* test to validate the statistical significance of observed difference. For adhesive quantities of 160 and 110 g/m^2, it may be said that at a significance level of 0.1, the plasma activation improves the strength of adhesive joint. A hypothesis that the low penetration depth obtained by the current XPS study is caused by an insufficient plasma activation dose can be rejected at this very same significance level, since in the opposite case this would fail to provide an adhesion improvement. In terms of process economy, the shear bond strength increase of 1 N/mm^2 corresponds roughly to adhesive saving of 20 g/m^2.

Owing to the large dispersion of measured data, the difference is not of statistical significance for specimens of 80 g/m^2. It should be noted, however, that the quantity of 80 g/m^2 is substantially below the minimum quantity of 120 g/m^2 recommended by adhesive manufacturer. Insufficient quantity of adhesive results in only partial filling of interfacial voids and hence, in substantial uncertainty of actual surface bonding area. An interesting consequence comes from the constant additive character of plasma activation effect. An eager researcher wanting to maximize the relative improvement in tensile shear strength test is advised to evaluate adhesive joints prepared by minimum amount of adhesive given by the manufacturer specification sheet.

Conclusion

This XPS study confirms the formation of polar functional groups on the wood surface exposed to atmospheric pressure plasma. The result provides an elucidative microscopic insight into the observed macroscopic effect of improved wettability of plasma-activated surface. Sputtering experiment with low-energy Ar^+ ions revealed that the plasma activation is a highly surface-specific phenomenon, which for given operation conditions forms a negligible WBL. The absence of a WBL was cross-checked with the precise measurements of tensile shear bond strength of polyvinyl acetate adhesive. Observed increase of bond strength proves a constructive role of XPS-identified functional groups, despite its high surface-specific occurrence. These results suggest a constant addition character of plasma activation to the bond strength. Although the presented results are obtained only for DCSBD plasma source at atmospheric pressure air, for other geometry variants of DBD or nonthermal plasma jet, similar results should be expected, providing that a similar temperature of working gas is kept, so that the effect of thermal degradation of wood material can be neglected.

Acknowledgments This research was supported in part by the Project ‘R&D Centre for Low-Cost Plasma and Nanotechnology Surface Modifications’—CZ.1.05/2.1.00/03.0086 funded by European Development Fund, the European Social Fund and the state budget of the Czech Republic, Project ‘The Establishment of an International Research Team for the Development of New Wood-based Materials’ Reg. No. CZ.1.07/2.3.00/20.0269 and Internal Grant Agency (IGA) of the Faculty of Forestry and Wood Technology, Mendel University in Brno (IGA-79/2013) as well as by the Program of ‘Employment of Newly Graduated Doctors of Science for Scientific Excellence’ (Grant number CZ.1.07/2.3.00/30.009) co-financed from European Social Fund and the state budget of the Czech Republic.

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