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Deposition of conductivity films on wood-based materials using plasma-enhanced PVD

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ABSTRACT

***Key words**: Plasma treatment, wood-based materials, PVD technology, magnetron sputtering, conductivity layer, conductive laminate board*

In a feasibility study, experiments were performed using the PVD (physical vapor deposition) technology for the plasma-enhanced deposition of a thin metal layer on wood-based materials. The aim of the study was to generate a conductivity layer on the substrates with the application focus on the integration of electronic features in wood-based furniture components. Using the magnetron sputtering process, a molybdenum layer of about 3 to 5 microns was successfully deposited on fiberboards, wood plastic composites, European Oak and paper samples. Prosperous results were achieved for conductivity measurements and examination of effectiveness of conductivity using a microcontroller board with touch and capacitive proximity switching function. In resistive tests, high conductivity (i.e. low resistance values) was achieved from the initial values of the uncoated substrates between 3 x 10^9 and 8 x 10^{12} ohm to a significantly lower level between 1 x 10^2 to 1 x 10^3 ohm after coating. Furthermore, the high conductivity of the deposited layers enables their application in a switching circuit. For all tested materials, the switching circuit was operating both in the touch and in the capacitive proximity function. Further experiments implied the prototyping of a laminate board with an integrated conductivity layer made by Plasma PVD.

1 INTRODUCTION

Plasma treatment is a state-of-the-art technology for the activation and coating of different materials, mostly plastics or metals. The plasma effect enables, depending on its application, a micro cleaning, a plasma activation and etching (e.g. to increase the free surface energy) or a functionalization through plasma coating using PVD (physical vapor deposition) and CVD (chemical vapor deposition) technologies. In these coating processes, thin films in the range from nanometers to microns are deposited on the substrate to obtain scratch- and wear-resistant, easy-to-clean, conductive or antibacterial/antimicrobial surfaces (Gerullis et al. 2018, Jocham et al. 2013, Rehn et al. 2003, Sinic et al. 2013).

Modern PVD process technologies are used to deposit high quality thin films, with different, functional properties, for innovative applications. The main basic PVD process technologies are evaporation (plasma and/or ion assisted), magnetron sputtering (dc or dc pulsed) and arc source deposition (dc or dc-pulsed). These processes use in most cases plasma and ion enhancement to control and optimise the deposition process to improve the properties of the obtained films.

Plasma and ion assisted PVD process technologies are widely used in the different application areas of thin film deposition. It is an essential element to achieve for example good film adhesion, high deposition rate, high film density, high hardness, desired mechanical stress and low surface roughness, beside other film and surface properties (Bunshah 1994, Mattox 1998, Pulker 1999, Strauss et al. 2003, Strauss et al. 2008).

In this study, it was evaluated if PVD processes are applicable for the deposition of thin, metal layers on wood and wood-based materials. The aim of the study was therefore to generate a conductivity layer on the substrates with focus on the integration of electronic features, like heating, sensors, conducting paths, etc. in wood-based furniture components.

2 MATERIALS AND METHODS

2.1 Materials

Commercially available medium density fibreboards (MDF; type *Egger MB*, board thickness 19mm), European Oak, wood plastic composites (WPC; consisting of 70% wood flour and 30% polypropylene, thickness 6mm), and paper samples (*Mondi Advantage Speed natural brown 80g/m²*) were used in this study. Before PVD coating, the materials were sawed to smaller samples (dimension 5 x 5cm), the paper samples were cut into slices of 10 x 5cm.

2.2 Thin film deposition using PVD techniques

Magnetron sputter deposition is a non-thermal vaporization process where surface atoms and molecules of the target (metals, alloys, compound materials) are physically ejected from a solid surface by momentum transfer via an energetic bombardment of particles which are usually gaseous ions (working gas: argon) from a plasma (Bewilogua 2013, Figure 1).

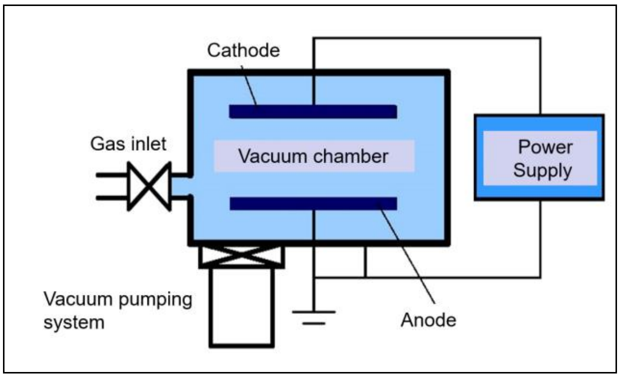

Figure 1. Principle configuration of magnetron sputtering (Bewilogua 2013)

Magnetron sputtering processes run usually from 10^{-4} to 10^{-2} mbar gas pressure in a vacuum magnetron sputter deposition unit as presented in Figure 2. In the case of depositing metal films, metal targets like molybdenum, tungsten, titanium and silver for the deposition material and argon for the working gas are used.

In the study, the process parameters in the magnetron sputtering depositing thin metal layers on wood-based materials were:

- Target material: molybdenum
- Total pressure: in the range of 10^{-2} to 10^{-1} mbar
- Target power density: 3W/cm²
- Voltages: in the range of 200V to 400V
- Temperature: below 50°C
- Bias: no biasing of substrates
- Frequency: dc mode

Figure 2. Vacuum magnetron sputter deposition unit (*Auto 306 Vacuum Coater from Edwards*) with power supplies and measurement components (*MCT – Material Center Tyrol, University of Innsbruck*)

2.3 Characterization

Cross cut tests, PVD layer thickness measurements, electrical resistance measurements and investigations of conductivity using a microcontroller board were performed on PVD-coated specimens.

2.3.1 Cross cut tests

For examination of adhesion properties of the PVD layers on the different substrates, cross cut tests were performed according to *ISO 2409:2007*. Due to the limited size of the coated samples of approximately 5 x 5cm and the small thickness of the layer in a micron scale, the cross cut tests were performed manually using an utility knife. Only two cuts were made in longitudinal and cross section. A minimum of three cross cut measurements were made on each specimen. The optical examination of the cross cuts was carried out using a stereo microscope *Zeiss Stemi 2000-C, KL 1500 LCD*.

2.3.2 Determination of layer thickness

The determination of PVD layer thickness was carried out by the cross-sectioning method according to *EN ISO 2808:2007-05* using a microscope (*Olympus BX51*). Before the measurement, the PVD-coated samples were cut into 1 x 0,5cm specimens. After that, the cross section was investigated with 50 and 100 times magnification of the microscope and images were taken. For calculation of layer thickness, the software *Color View Soft Imaging Systems* was used.

2.3.3 Electrical resistance measurements and investigation of conductivity

To characterize electrical resistance of the uncoated and PVD-coated samples, two different measurement devices were used. The electrical resistance measurements of the uncoated samples were performed according to DIN EN 61340-2-3:2015-04; VDE 0300-2-3:2015-04, using a tera-ohmmeter (*Tera-Ohmmeter TO-3 from H.-P. Fischer Elektronik GmbH & Co*). A guard ring electrode was positioned on the substrates and the surface electrical resistance was determined (absolute value). The test voltage was 250 V.

The PVD-coated samples showed very low electrical resistance values, that could not be measured with the tera-ohmmeter. Therefore, the electrical resistance measurements of the PVD-coated samples were carried out using a multimeter (*FLUKE 175 True RMS*).

Another analytical method for the examination of effectiveness of the conductivity of the coated thin layers implied the application in a switching circuit. Using a microcontroller board (*Bare Conductive, TouchBoard*) with touch and capacitive proximity switching function and connected loudspeaker, the conductivity of the PVD-coated materials was tested.

2.4 Prototyping of a conductive laminate board

Finally, a prototype of a wood-based laminate board with an integrated conductive layer was produced. Hence, the molybdenum plasma-coated paper was interlaminated during the production of a veneered particle board. The following materials were prepared for the laminate board:

- Commercially available particle board (substrate thickness 19mm);
- Molybdenum plasma-coated paper (functional intermediate layer);
- Commercially available common walnut veneer (top layer thickness 0,6mm).

To bond the individual laminate layers, a 2-component epoxy system with hardener from *Time Out Composites* was used. The resin (*SICOMIN SR1280*) and the hardener (SICOMIN SZ8525) were mixed in a gravimetric mixing ratio of 100:24. The resin system (amount of 140g/m^2) was applied directly to the materials to be bonded and was spread evenly by a squeegee, removing excess resin. The laminate was built up from the particle board as the base plate, the plasma-coated paper as an intermediate layer, to the veneer as a top layer. The epoxy resin-bonded laminate was then subsequently pressed in a laboratory press (*SUT-PUK 1600 from Svoboda*) at a temperature of 100°C for 10 minutes at a specific pressure of 3 bar.

3 RESULTS AND DISCUSSION

3.1 PVD plasma coating on wood-based materials

The magnetron plasma sputtering was used for the deposition of a thin metal layer on wood-based materials. For all the tested substrates, a homogenous molybdenum layer was successfully deposited on WPC, MDF, oak timber and paper specimens. Images of the uncoated and coated samples are presented in Figure 3.

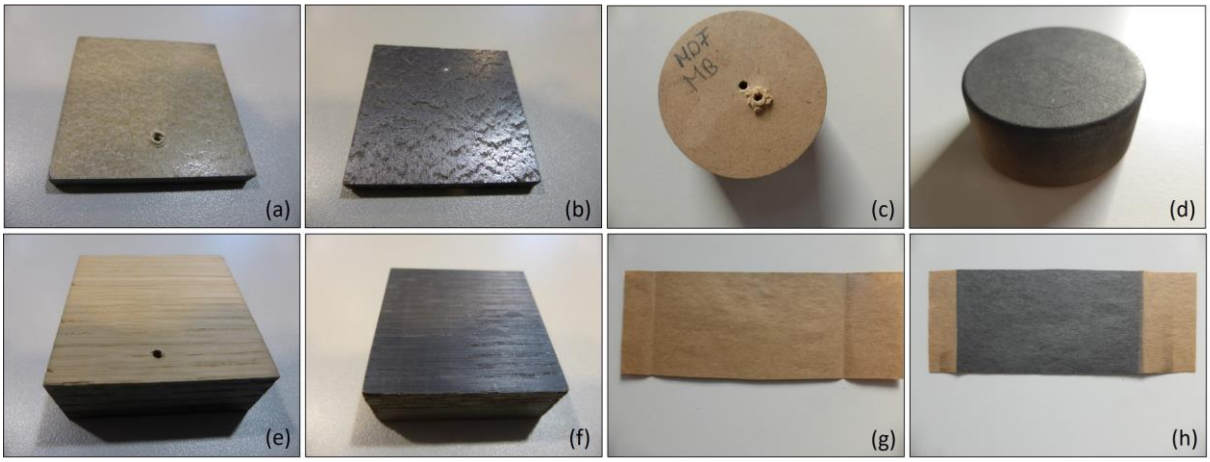

Figure 3. Images of the uncoated and coated specimens (a) WPC uncoated and (b) WPC coated with molybdenum, (c) MDF uncoated and (d) MDF coated with molybdenum, (e) oak timber uncoated and (f) oak timber with molybdenum, (g) paper samples uncoated and (h) paper samples with molybdenum

On all tested materials, a homogenous molybdenum layer was deposited on the surfaces. The individual surface topographies of each sample was maintained. The coated MDF and paper samples presented a smooth surface, whereas the wood species oak had "open-pores". This wood type was characterized by small holes in its surface that resulted in a three-dimensionally textured surface. The irregularities of the WPC surface, which originated in the extrusion process, were maintained after plasma coating as well, indicating that a very thin layer was applied by PVD.

For examination of adhesion properties of the molybdenum layer on the different substrates, cross cuts tests were performed and analyzed by microscope. Representative images of the cross cuts are shown in Figure 4. In the case of the MDF sample, no significant abnormalities in the cross cut features were found. The adhesion between the molybdenum layer and the MDF substrate was good and no delamination was detected. Similar results were obtained for the paper sample, which showed good adhesion between layer and substrate. The adhesion of the PVD coating to the substrate was also satisfactory for the WPC sample and the oak specimen.

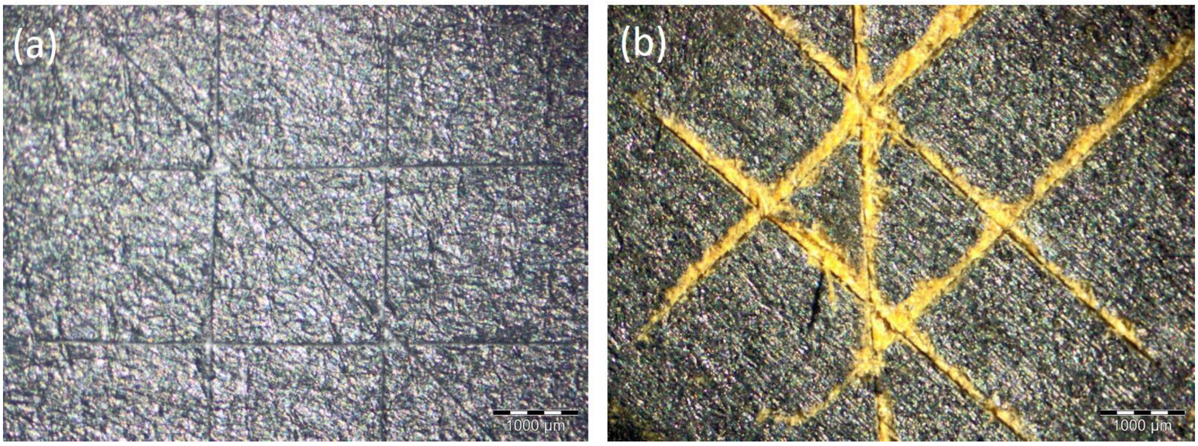

Figure 4. Images of the cross cuts with 20 times magnification of the microscope (a) in the paper sample and (b) in the MDF specimen

The determination of PVD layer thickness was carried out by the cross-sectioning method using a microscope. Repeatable values were measured for the thickness of the molybdenum layer on the oak specimen (3,3 ± 0,3 microns), MDF substrate (3,9 ± 1,0 microns) and WPC samples (4,5 ± 0,4 microns). Difficulties occurred in the determination of the layer thickness on the paper sample. Due to the instability of the paper, the profile of the specimen could not be positioned exactly in the focus of the microscope. Hence, too high values for the layer thickness were determined caused falsely by a three-dimensional effect due to the flection of the specimen.

3.2 Conductivity of the thin layer

The conductivity respectively the resistance value of the thin layer was measured by ohmmeter. Table 1 presents the results of the resistance measurements on different wood-based substrates using the tera-ohmmeter *TO-03* before coating and by means of multimeter *FLUKE 175* after coating. The electrical resistance values of the uncoated materials showed usual high values of about 3×10^9 ohm for oak timber to about 8×10^{12} ohm for the plastic bonded WPC material. Due to the plasma-deposited, conductive molybdenum layer, the resistance values were reduced significantly to a lower level between about 1×10^2 and 1×10^3 ohm after coating.

Table 1. Resistance values of the untreated and molybdenum-coated specimens

No.	Materials	Resistance value before coating [Ω]	Resistance value after coating [Ω]
1	WPC	8,00 x E+12	227 ± 21
2	MDF	5,00 x E+11	1010 ± 113
3	Oak	3,00 x E+09	1200 ± 173
4	Paper	2,00 x E+11	320 ± 17

Furthermore, the reason for the difference in the resistance values after coating could be either the surface topography of the substrates ("non-porous", "open-porous", "diffuse-porous", etc.) or even the deposited layer thickness and its distribution, which may vary widely between different substrates and could cause measurable differences in conductivity, which still has to be verified.

In addition, effectiveness of the conductivity of the thin layers was tested using a microcontroller board with touch and capacitive proximity switching function. For all tested coated materials WPC, MDF, oak and paper samples, the switching circuit was operating both in the touch and the capacitive proximity function. Hence, the conductive layers enabled an operating electrical circuit which worked via touch as well as through contact-free approach that was signalized by a LED lamp optically and by an acoustic signal via loudspeaker. As a representative example, the switching functions of the molybdenum-coated paper sample are demonstrated in Figure 5.

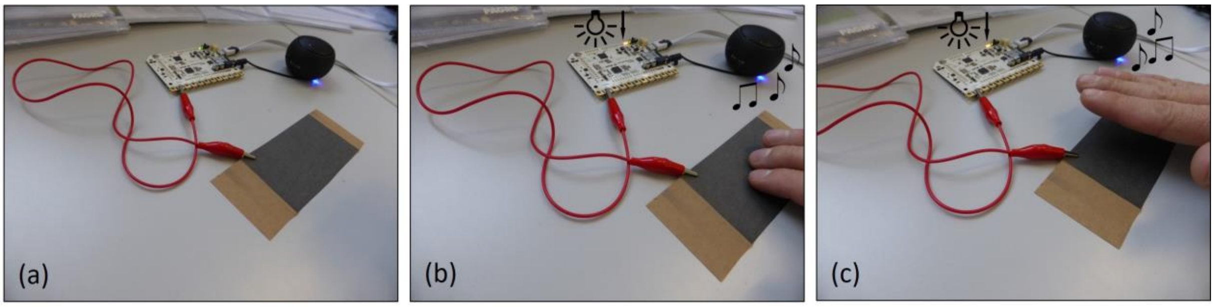

Figure 5. Switching circuit due to the conductive molybdenum layer on the paper sample (a) in standby-mode, (b) operating via touch function and (c) via capacitive proximity function. The switching function is indicated by an orange LED lamp lighting up and by an acoustic signal transmitted via loudspeaker.

Additionally, the maintenance of the switching functions was tested using a wooden panel positioned on the conductive paper. In spite of the insulating material, the switching circuit was operating both in touch and proximity mode. Hence, further experiments were considered including the integration of a PVD conductive layer in a laminate board.

3.3 Prototyping of a conductive wood-based laminate board

A prototype of a conductive laminate 19 mm particleboard with an integrated plasma-coated paper with mo-lybdenum was successfully produced (Figure 6). The use of epoxy resin as adhesive led to a compact laminate structure with good adhesion properties. After the pressing process, slight excess of resin was detected on the surface and the edges of the board that in further experiments could be avoided due to an adjustment of the pressing parameters or through modification of the resin viscosity.

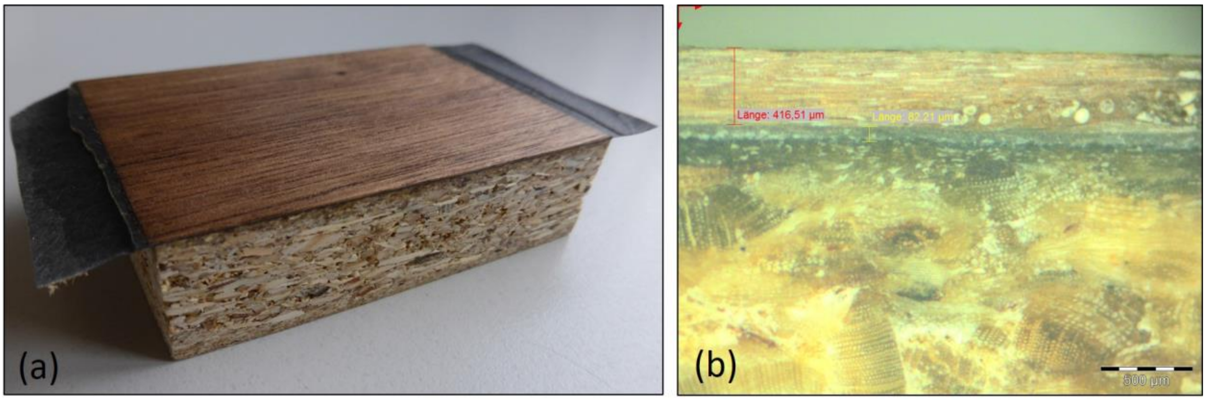

Figure 6. (a) Prototype of a laminate 19 mm particleboard with an integrated conductive molybdenum layer on a paper specimen made by Plasma PVD and (b) image of the layer composition of the laminate board made by microscope (top layer - veneer with a thickness of 416 microns; intermediate layer – molybdenum-coated paper with a thickness of 82 microns; base plate – 19 mm particleboard)

After lamination, electrical resistance measurements and investigations of conductivity were carried out. As a result, no significant increase of the electrical resistance values of the molybdenum layer were determined after interlamination. Hence, the influence of the bonding and pressing process on the conductivity was negligible but should be confirmed in further experiments on large-scale specimens. Furthermore, the switching functions of the molybdenum layer remained after lamination and the conductive laminate board worked reliably both in the touch and in the capacitive proximity function which was tested by the microcontroller board.

4 CONCLUSION AND OUTLOOK

In the present study, it was investigated if PVD processes are applicable for the deposition of thin, metal layers on wood and wood-based materials. The aim of the research was to generate conductivity coatings on these substrates with focus on the integration of electronic features in wood-based components.

In the first test series, molybdenum layers were successfully deposited on different wood-based materials using PVD Magnetron-Sputtering. The thin layers with thicknesses of about 3 to 5 microns exhibit a good adhesion to all substrates, a homogenous surface topography and also distinguished conductivity properties measured by means of ohmmeter and microcontroller board. Furthermore, a prototype of a conductive laminate board with an integrated molybdenum layer was successfully produced.

Further experiments will imply the development and production of conductive laminates in full-scale components by an appropriate adaption of the PVD process. Hence, different laminate structures with interlamination of a conductivity coating according to the customer demands of the wood-working and furniture industry will be developed and tested.

An objective of further studies could be the decorative surface coating using these PVD techniques. Therefore, various metal-based layers in different colors, for instance silver, gold, bronze, black and prismatic color, will be deposited on wood and wood-based materials preserving the characteristics of the particular surface topography.

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