MODIFICATION OF FUNCTIONAL PROPERTIES OF GLASS FIBER FILTERS SURFACES USING PDV MAGNETRON SPUTTERING

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Abstract

The glass microfibre filters (GF/A) are designed to monitor water and air pollution, wastewater filtration, algae and bacteria culture filtration, protein filtration, and food analysis. Filters are characterized by low flow resistance and allow for a high workload. Thin layers of CuAg, Cu, Ti, Zr, Cu_xAg_yO, Cu_xO, TiO_x, and ZrO_x were deposited on the surface of glass fiber filters using DC magnetron sputtering (PVD). The study indicated that Cu_xAg_yO and Cu_xO films exhibited thicknesses similar to those created by non-reactive deposition. In contrast, TiO_x and ZrO_x films required three times longer deposition durations to achieve similar thicknesses to pure metal films. The research explores modification of the surface of glass fiber filters using CuAg, Cu, Ti, Zr, Cu_xAg_yO, Cu_xO, TiO_x, and ZrO_x thin films deposited by DC magnetron sputtering (PVD). Thin films containing oxygen demonstrated improved tribological properties with reduced static and dynamic friction coefficients.
Keywords: PVD Magnetron Sputtering, CuAg, Cu, Ti, Zr, Cu_xAg_yO, Cu_xO, TiO_x and ZrO_x Thin Films, Glass Fibers Filters, Functional Properties

1. INTRODUCTION

Filtration is a separation process in which particles from a suspension are separated from a liquid or gas using a porous medium. This medium can be filters or various types of filter media. Filters and filter media are divided into several aspects, depending on type, application, or material. [1] Filtration parameters can be divided into parameters of the filter material, parameters of the filtered particles, and parameters of the filtration process. The main properties of filters are efficiency, durability, resistance to external influences, breathability, and porosity. Inorganic and organic substances (natural and synthetic) are used. Inorganic substances include minerals, carbon, glass, metals, metal oxides, and ceramics. [2] Glass is resistant to acids and has satisfactory resistance to alkalis but is not resistant to abrasion. The use of filters made of glass and glass fibers is limited by poor mechanical resistance. [2, 3]

Magnetron sputtering is a modification of sputtering technology, where a planar magnetron is added under the target to make the process more efficient. A glow discharge is created in the plasma; positive ions of the inert gas hit the target (cathode) and sputter its atoms. A sputtering of a specific material occurs on the substrate. [4, 5] Magnetron sputtering is one of the most commonly used technologies for depositing thin layers. It is used in a wide range of applications, such as for large-area parts, flat-panel displays, or thin-film solar cells. This technology can be used to coat materials that are very sensitive to temperature, as there is a low thermal load on the substrate. The use of thin film formation can be seen in the food industry, filtration systems, medical devices, and the textile industry due to the antibacterial properties provided by some types of metal layers. [5, 6, 7]

The presented research aims to propose a suitable modification of filtration systems with thin layers to improve their functional properties and ensure easier cleaning of their surfaces. Experiments are focused on forming thin layers to modify materials for filtration applications and monitoring their structural changes, porosity, utility properties, and mechanical stability.

2. MATERIALS AND EVALUATION METHODS USED

2.1 Materials used

The experiment was performed on the base material, filter material (substrate): a glass microfiber filter (designation GF/A) from the manufacturer Whatman (100 % borosilicate glass without fillers). The glass microfiber filter (diameter 47 mm; thickness 260 μm and retention size 1.6 μm) is intended for monitoring water and air pollution and filtering wastewater, filtering algae and bacteria cultures, protein filtration, and food analysis. Furthermore, it is used for radioimmunoassay of weak β emitters and gravimetric determination of particles in the air. It is characterized by low flow resistance and allows for high loading. [8, 9]

2.2 Designed thin layers and their deposition

Thin layers applicable to glass fiber filters were designed according to a literature search. It was found that copper, silver, and their mixture, as well as titanium and zirconium, are used for the above-mentioned applications. [7, 10] The layers used for deposition on the surface of glass microfiber filters were created from targets made of zirconium, copper, titanium, and copper-silver alloys. A copper-silver alloy with an atomic weight ratio of 30:70 (Cu:Ag) was used. The deposition of thin layers was carried out in a coating device NP 70 from KWS CZ s.r.o. [11]

The working gas argon with a flow rate of 30 sccm was used to form thin layers. The deposition was carried out by rotating the stage in the chamber at a speed of 10°/sec (0.1745 rad/sec) with a total number of 3 revolutions. A low primary pressure, $P_{prim} \leq 0.001$ Pa, was used to achieve high purity of the deposition process. The working pressure used was 0.3 Pa at a source power of 1000 V. Thin layers of metals and their alloys were formed from the targets mentioned above, where two samples were made for each metal with a different specified source power (**Table 1**).

Table 1 Designation of samples of thin layers of metals and metal oxides

Target	Sample	Working pressure (Pa)	Entered power source (kW)	Sample	Working pressure (Pa)	Entered power source (kW)
	Thin metal layers			*Thin metal oxide layers*		
CuAg	**C2.1**	0.3	0.9	**K2.1**	0.13	0.9
CuAg	**C2.2**	0.3	0.4	**K2.2**	0.13	0.4
Cu	**C3.1**	0.3	0.9	**K3.1**	0.13	0.9
Cu	**C3.2**	0.3	0.4	**K3.2**	0.13	0.4
Ti	**C4.1**	0.3	0.9	**K4.1**	0.13	0.9
Ti	**C4.2**	0.3	0.4	**K4.2**	0.13	0.4
Zr	**C5.1**	0.3	0.9	**K5.1**	0.13	0.9
Zr	**C5.2**	0.3	0.4	**K5.2**	0.13	0.4

Deposited metals and their alloys tend to oxidize over time, which can lead to changes in their properties. Therefore, thin layers of selected metal oxides (based on copper, titanium, zirconium, and copper-silver alloys) were designed. Two working gases were used in the coating process – argon (flow rate 30 sccm) and oxygen (flow rate 3.3 sccm) in a ratio of 10:1. The number of table revolutions, i.e., the deposition time for layers from titanium and zirconium targets, was increased to 9 revolutions at the same speed due to the longer time required to form these layers (**Table 1**).

2.3 Evaluation methods

Surface roughness

The change in surface roughness of glass fiber filters was evaluated on a non-contact optical 3D profilometer S Neox from SENSOFAR according to ISO 21920-2:2021 Geometrical product specifications (GPS — Surface texture: Profile Part 2: Terms, definitions, and surface texture parameters). The measurement was performed at an optical magnification of 20x for all thin layers formed on glass substrates. For a suitable interpretation of the results, the surface of each scan was leveled in the Software.

Material resistance to deformation

The stiffness test of flat textiles was carried out according to the standard ČSN 80 0858 on a TH-5 stiffness tester. The samples were prepared based on the standards ČSN 80 0069 and ČSN 80 0072 with dimensions of 2.5 cm×4.7 cm. The bending stress of the material was carried out when the device's jaws were deflected by an angle of 60°. For each type of layer, three measurements were made, with the layer facing away from the device and towards it. The maximum force values were obtained and converted into the bending moment, an equivalent quantity for stiffness.

3. RESULTS AND DISCUSSION

The measured data were processed using Microsoft Excel and OriginPro. Additionally, hypotheses were tested to compare samples of deposited thin layers with the substrate and to analyze the effects of different specified source power levels on the same substrate. A two-sample t-test was conducted at a 95% significance level for these comparisons.

According to the evaluation of the structure of the samples from the SEM analysis images, it was found that the substrate has an anisotropic structure, the glass fibers are uniform, and impurities are recorded on them (**Figure 1a**). The SEM analysis of thin layers on glass fiber (GF) filters indicated the following: a perfectly smooth surface was observed for layers C2.1, C2.2, C3.2, C4.1, K2.2, K5.1 and K5.2 (**Figure 1b**); a smooth surface was observed for samples C3.1, C4.2, C5.1, C5.2 and K4.1, on which a smaller amount of impurities was visually visible compared to the substrate (**Figure 1c**); a more significant amount of impurities is visible on the fibers of sample K4.2, its surface is similar to the substrate (**Figure 1d**).

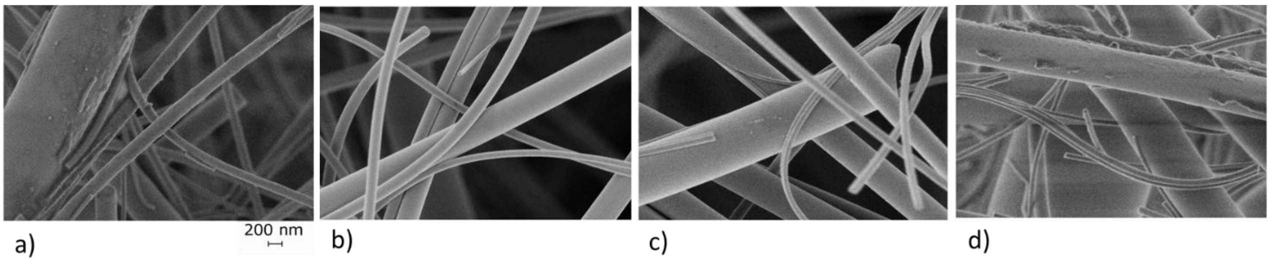


Figure 1 Glass fiber substrate without thin film a) GF; thin layers on a glass substrate: b) C2.1, c) C3.1, and d) K4.2

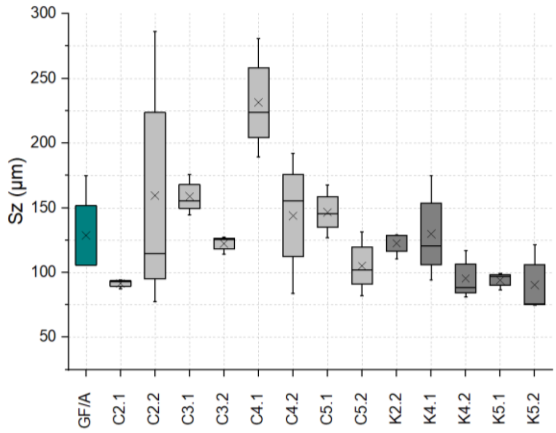
Based on the chemical element analysis (**Table 2**), it was detected that the GF filter does not contain an organic proportion of elements; the most represented elements are oxygen (60.3 at. %) and silicon (25.7 at.%). From the calculated ratios of elements Cu:Ag, a higher proportion of silver was found in the Cu_xAg_yO and CuAg layers than copper. According to the determined ratios of elements to oxygen in the oxygen layers, a higher proportion of metal elements from the target was found in the Cu_xAg_yO and Cu_xO layers compared to the amount of oxygen from the reactive gas. In the ZrO_x layers, a higher proportion of oxygen is found compared to zirconium.

Table 2 Representation of elements in thin layers in (at. %)

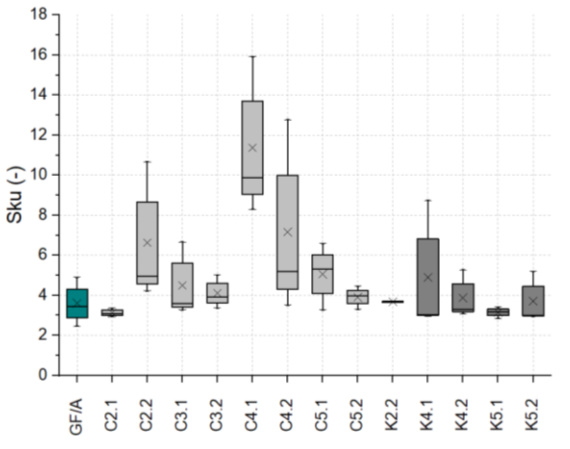
Sample	C2.1	C2.2	K2.2 (Si-wafer)	K2.2 (Si-wafer)	K4.1 (Si-wafer)	K4.2 (Si-wafer)	K5.1 (Si-wafer)	K5.2 (Si-wafer)
Elements (at%)	Cu:Ag 38:62	Ag: Cu 47:53	Ag: Cu 36:64	Ag: Cu:O 33:59:8	N/A	N/A	Zr:O 32:68	Zr:O 8:92

** The chemical composition of thin layers K2.1, K3.1, and K3.2 was not evaluated due to their exclusion from further testing.*

The height parameters of the surface roughness were monitored – the maximum height of the surface Sz (**Figure 2a**) and the kurtosis of the surface Sku (**Figure 2b**). The Sz values for C2.1, C3.2, K2.2 and K5.1 were measured in a small range of values (**Figure 2a**). All measured values of sample C4.1 were higher than the substrate, according to the hypothesis, a statistically significant difference was found. When testing the hypothesis of the difference between the individual samples, a statistically significant difference was found for C3.1-C3.2, whose values do not overlap. The Sku values for samples C2.2, C4.1, C4.2, and K4.1 had a wide range (**Figure 2b**). According to the hypothesis, a statistically significant difference was found for sample C4.1 compared to the GF substrate. No statistically significant differences were found between samples differing in the specified power of the source. They were higher for samples C2.2 and C4.1. Based on the Sku values, it was found that the GF substrate has a pointed surface structure, with the exception of the values for samples C2.1 and K5.1, which oscillate around a normal distribution.



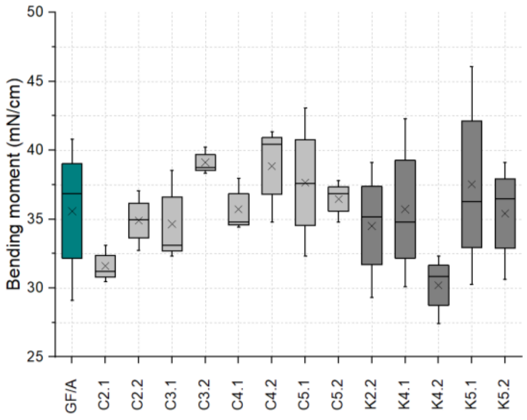
a)



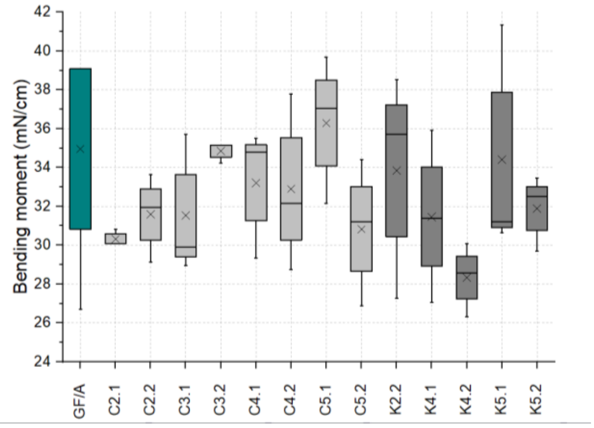
b)

Figure 2 Surface roughness parameters a) parameter Sz b) parameter Sku

Evaluation of resistance to deformation: the obtained force values were converted into bending moment. For the GF substrate, a negatively skewed distribution was observed for values both away from the device (35.6 ± 4.8 mN/cm) and towards the device (35.0 ± 5.8 mN/cm), i.e., values higher than the average prevail (**Figure 3**). The measured values for most samples for the GF substrate, except for C2.1 and C3.2, occurred in a wide range. Based on the hypothesis testing of the samples with deposited thin layers versus the substrate, no statistically significant difference was found. In the hypothesis testing of the samples tested from the device and to the device, a statistically significant difference was found for the C3.2 layer.



a)



b)

Figure 3 The bending moment of the samples during thin layer testing: a) away from the device, b) towards the device

According to the measured spectra, elemental analysis of samples with thin layers is influenced by the substrate due to the very small thickness of the deposited layers. A wide range of element ratio values was found for the Cu_xAg_yO and Cu_xO layers. The measurement was performed only once for each sample. In the article [7], a passivation layer was detected in deposited thin Cu layers. This phenomenon could not be proven or disproved using the chosen method; the detections were influenced by oxygen from the substrate. From the determined ratios of elements to oxygen in a series of oxygen layers, a higher proportion of elements from the target compared to oxygen was observed in the Cu_xAg_yO and Cu_xO layers, and a higher proportion of oxygen compared to zirconium in the Zr_xO layers. This is due to the amount of reactive gas flow used or the power of the source. This factor is related to the layers' thickness and particle dedusting speed. For the Cu_xAg_yO and Cu_xO layers, a low oxygen flow or high source power was used, resulting in low target oxidation and so-called poisoning. On the contrary, for the Zr_xO layers, a high oxygen flow or a relatively low source power was used, resulting in high target poisoning. This is related to forming different surface states after reactive sputtering – fully metallic, fully oxidized, and partially oxidized. All of the above factors are described in the article [12]. In the case of Cu_xAg_yO layers, the reaction of oxygen is likely only with copper due to the low reactivity of silver with oxygen, i.e., the formation of thin layers of Ag, Cu, and Cu_xO, which is described in the article [13].

The measured values of the resistance of the layers to deformation for most samples for the GF substrate occurred in a wide range, probably due to the anisotropic distribution of glass fibers in the substrate itself. In the hypothesis for comparing samples tested away from the device and towards the device, a statistically significant difference was found for layer C3.2. This may be due to the measured values for these samples being in a small interval, i.e., having a narrow quartile range.

4. CONCLUSION

The presented research evaluates thin layers of CuAg, Cu, Zr, and Ti targets (layers of pure metals and alloys and oxygen layers) deposited on glass fiber filters. The examined thin layers on glass fiber substrates have sufficient adhesion, and there was no delamination of the layers during various tests. They do not significantly change the samples' resistance to deformation and do not prevent the filters' permeability. In layers deposited on glass fiber substrates, the appearance of the layer structure was found to depend on the specified power of the source.

Research needs to be focused on a series of oxygen layers, where we can focus on changing the reactive gas flow rate or source power. This would allow the properties of the layers to be monitored based on changes in their stoichiometry. Another option is to create thin layers where the deposition time will be varied while using the same source power to monitor the change in structure as a function of layer thickness when the deposition time is changed.

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