THIN LAYERS ON THE FABRICS PREPARED BY THE SOL-GEL METHOD

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Abstract
This work deals with the properties of the thin films prepared by the so-gel method. Its resistance to the mechanical stress – the friction. The plasma was useed for the better grip of the layers on the fabrics.

Key words: sol-gel method, polymers, mechanical properties, plasma

1. Introduction

The thin layers of the silicon dioxide are known for their outstanding properties such as the hardness, the wear resistance, the chemical resistance and the excellent optical and dielectric properties, which are extensively used as the functional coatings for the various types of the materials. These purely inorganic layers are generally fragile, and their perfect compaction requires relatively the high temperatures [1]. These deficiencies can solve the inorganic-organic materials, which can be compared with the pure silica layers. This layers can give the new and often unique properties.

2. Material and methods

2.1 Materials and pre-treatment

The 100% polypropylene fabrics of 130 $g.m^{-2}$ (PP), 100% polyester fabrics of 175 $g.m^{-2}$ (PL) and the polyester-micro fabrics of 79 $g.m^{-2}$ (PLmicro) were used for all types of experiments.
The impurities were removed from the samples by the following procedure:
1 The washing in the bath containing 1 g^{-1} Syntapon ABA (mixed anionic surfactants), the baths ratio - 1:30 (to 1 g of the material to the 30 ml bath). The bath was prepared from distilled water and the samples held at boiling for the 15 minutes.
2. The samples were rinsed under the cold running water for the 5 minutes.
3.They were left between the filter paper until the completely drying.
4.The extraction was realized by Soxhlet extractor with chloroform (the 9 cycles).
5.The samples are left to dry on the filter paper after the extraction.

2.2 Plasma treatment

The plasma modification is used for surface modification of materials. It is to use to cleaning of the surface of the material. The surface is destroyed and the new chemical groups are established on the surface of the material.
The modification of the plasma reactor was used DCSBD (Diffuse Coplanar Surface Barrier Discharge). It is a device that is designed for working flat material in A4 size plasma at atmospheric pressure under different working gases.

Processing conditions for this experiment were as follows:
Power - 300 W, Feed Rate eighth - 10-3 $m.s^{-1}$, Working gas – air.

2.3. Composition of films and their application to textile

The thin layers were prepared by the sols AC2 and AC3 (table 1). Those baths were diluted in IPA 1:4 (sol:IPA) and AC2/Z AC3/Z were marked. The overview of the prepared samples submitt table 2.

Table 1: Composition of thin layers (TMSPM – the methacryloxypropyltrimethoxysilane, IPA – the isoprpylacohol, HCL – the hydrochloric acid, H_2O – the distilled water, BPO – the dibenzoylperoxide)

Sol	**AC2 [ml]**	**AC3 [ml]**
TMSPM	3	3
IPA	49	49
HCl 2 $mol.dm^{-3}$	0.2	0.2
H_2O	0.2	0.2
BPO	0.2	

The textile materials were coating by the sols, this proces was duing by the soaking and by the pulling. The hydrolysis in the laboratory environment was extended to 15 minutes and samples were placed on the folded aluminum foil. All materials with a layer of AC2/Z were heat treated at 80 ° C for 3 hours. The layer of AC3 / Z were heat treated at 150 ° C for 2 hours (without the PP material).

Table 2: Survey samples and their descriptions

the fabrics type /the treatment	the non-plasma treatment		the plasma treatment	
PP fabrics	AC2/Z	-	AC2/Z	-
PL fabrics	AC2/Z	AC3/Z	AC2/Z	AC3/Z
PLmicro-fabrics	AC2/Z	AC3/Z	AC2/Z	AC3/Z

3. Measurment methods

3.1 Scanning electrion microskopy (SEM)

The surface morfology of samples was observed under scanning electrion microskopy (SEM, Vega) at the diferent magnification.[2]

3.2 Determination of the bending moment

The method which gives information about the immediate stiffness fabrics, is static. The device TH5 (Czechoslovak patent)was designed for the measurig of this moment TH5 provides the bending stiffness of the measured resistance force against the bending of the fabric [3].
The TH5 device senses the strength which will use to the strip of fabric on the sensing element. The strip has the standard fixed length l and the width b. The method is illustrated in figure 1.

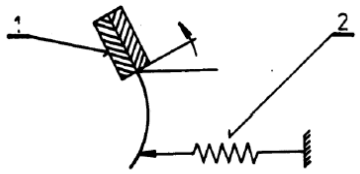

Figure 1: Chart of the device TH5 (1- clamping jaw, 2- sensing element)

The relationship [46]:was used for the calculating the bending moment:

$$M_O = F_1 . K$$

where: M_O ... the bending moment for the width of the sample 1 cm [mN.cm]
F_1 ... the maximum value of the force [N]
K .... the constant calculated from the relationship K = L / b (for the specific conditions of K = 0.604)
L .... the length of the sample measured at the 60 ° deviation from the edge of the jaw to the sensor equipment of L = 1.5 cm
b .... the working sample width b = 2.5 cm.

3.3 Determination of the friction coefficient

The friction surfaces deals with the interaction of bodies in mutual relative motion. By definition, the friction resistance to movement of a body along the surface of the second body (figure. 2). The friction is static or dynamic [4].

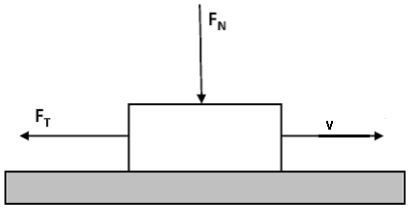

Figure 2: Device for measuring the friction (F_N – the normal force, F_T – the friction force, v – the velocity, the direction of the movement)

4.Results and discussion

4.1 SEM

The selected images of the textiles coated with the layers are displayed in the figure 3 to 8. In te figures 3 and 4 is documented the layer AC3/Z PL on the fabric. Outside of the places where the layer is damaged (figure 4) is practically visible the thin layer. The layer reproduces very well the surface of the fibers. The AC2/Z layers of fabric PLmicro are documeneted in the figures 5 and 6. The surface of the fibers and the bridges between the fibers are shown in the figures 5 and 6. The figures. 7 and 8 confirm that the film of AC2/Z have very well adhesion to the PP samples.

4.2 Determination of the bending moment

The prepared layers of fabrics are shown to the significantly change their stiffness in the bending. The calculated values of the bending moments are listed in table 3. It is evident, that the samples

coated with layers have the higher coeficeiet of the bending than the samples without the layers.The textile fabrics coated with a layer of AC3/Z have the greater bending moment than the fabric with the layer AC2/Z. This can be attributed to the higher degree of the polymerization and the bridges between the fibers

Table 3: The calculated values of the bending moments measured materials (P – the plasma treatment, M_O – the mean values of the bending moment; LD – the lower limit of the 95% confidence interval for average, LH – the upper limit of the 95% confidence interval for mean value).

type of materials	layer and treatment	M_o [mN.cm]	LD	LH
PES - weft	non	3.48	2.80	4.08
	AC2/Z	10.36	9.24	11.49
	P + AC2/Z	12.70	9.59	15.30
	AC3/Z	27.30	23.23	31.15
	P + AC3/Z	61.14	23.79	38.06
PES - warp	non	5.36	4.06	6.37
	AC2/Z	17.38	15.57	19.40
	P + AC2/Z	24.34	21.07	27.49
	AC3/Z	50.86	45.07	55.50
	P + AC3/Z	60.38	56.49	63.53
PESmicro - weft	non	0.49	0.39	0.57
	AC2/Z	1.69	1.29	2.09
	P + AC2/Z	2.69	2.26	3.27
	AC3/Z	5.20	5.20	8.02
	P + AC3/Z	7.69	7.69	8.82
PESmicro - warp	non	1.33	0.99	1.68
	AC2/Z	3.06	2.62	3.58
	P + AC2/Z	2.61	2.13	3.08
	AC3/Z	6.46	5.39	7.58
	P + AC3/Z	11.54	10.52	12.38
PP- weft	non	2.38	2.05	2.88
	AC2/Z	7.97	6.11	9.41
	P + AC2/Z	13.00	11.93	13.98
PP- warp	non	2.49	2.04	3.04
	AC2/Z	8.35	6.80	9.57
	P + AC2/Z	11.40	8.44	14.51

4.3 Determination of the friction coefficient

The calculated values of the friction coefficients of fabric coated with layers (table 4) are surrounded by the considerable uncertainty due to the small number of the measurements and the considerable scattering of the values

The representative static coefficient of friction appears μ_{statP} (calculated from the initial value F_{maxP}) and the dynamic coefficient of friction μ_{dyn} (calculated from the median value Fmed). The

static coefficient of the friction μ_{stat} (calculated from the measured maximum force value F_{max}) is burdened with the considerable scattering of the values.

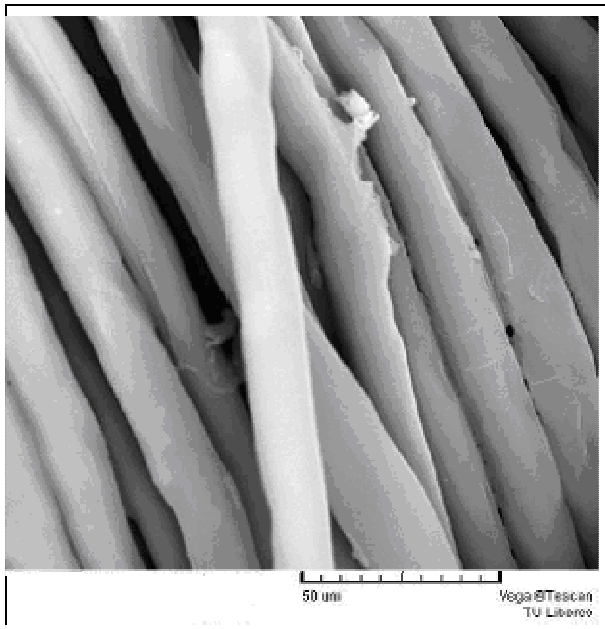

Figure 3: The PP sample with layer AC2/2Z. (Electron scanning microscope VEGA)

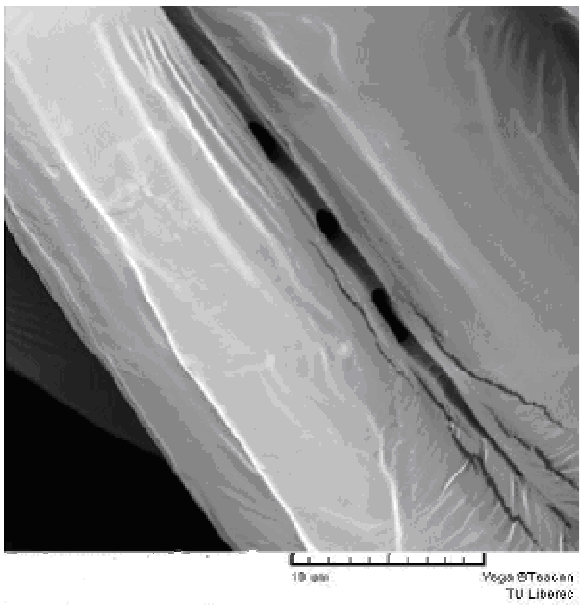

Figure 4: The PP sample with layer AC2/2Z, detail. (Electron scanning microscope VEGA)

Table 4: Summary of calculated coefficients of friction (LD - lower limit of the 95% confidence interval for the mean value; LH – upper limit of 95% confidence interval for-the mean value).

	μ_{statP}			μ_{stat}			μ_{dyn}		
type of materials / treatment	average	LD	LH	average	LD	LH	average	LD	LH
PES-non-layer	0.14	0.11	0.19	0.21	0.16	0.26	0.10	0.07	0.13
PES AC2/Z	0.23	0.19	0.28	0.37	0.27	0.48	0.20	0.12	0.27
PES P + AC2/Z	0.24	0.05	0.42	0.32	0.15	0.50	0.18	0.18	0.19
PES AC3/Z	0.36	0.15	0.57	0.48	0.30	0.63	0.30	0.23	0.37
PES P + AC3/Z	0.26	0.09	0.43	0.43	0.24	0.61	0.29	0.20	0.38
PESmicro-non-layer	0.11	0.07	0.15	0.21	0.15	0.28	0.08	0.04	0.13
PESmicro AC2/Z	0.20	0.09	0.30	0.26	0.19	0.33	0.13	0.11	0.16
PESmicro P + AC2/Z	0.25	0.19	0.30	0.29	0.19	0.40	0.10	0.06	0.14

PESmicro AC3/Z	0.27	0.20	0.34	0.33	0.22	0.44	0.13	0.06	0.19
PESmicro P + AC3/Z	0.35	0.30	0.38	0.49	0.36	0.63	0.25	0.17	0.33
PP-non-layer	0.10	0.02	0.22	0.22	0.08	0.37	0.10	0.04	0.17
PP AC2/Z	0.12	0.06	0.17	0.22	0.16	0.28	0.09	0.06	0.13
PP P + AC2/Z	0.12	0.06	0.18	0.18	0,10	0.26	0.09	0.06	0.11

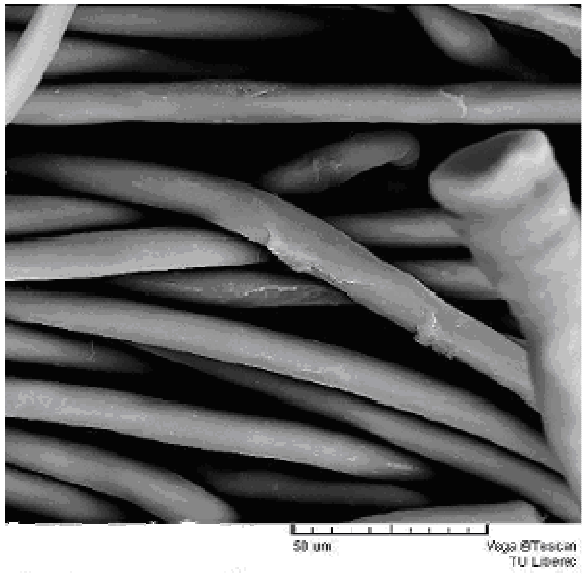

Figure5: The PL sample with layer AC3/2Z. (Electron scanning microscope VEGA)

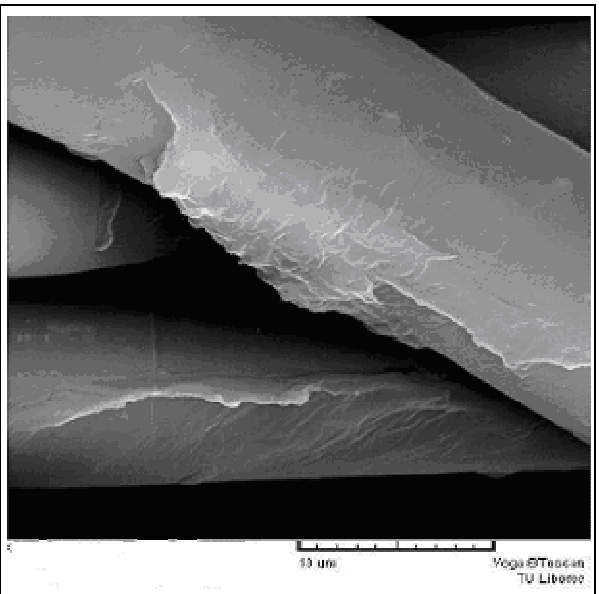

Figure 6: The PL sample with layer AC3/2Z, detail. (Electron scanning microscope VEGA)

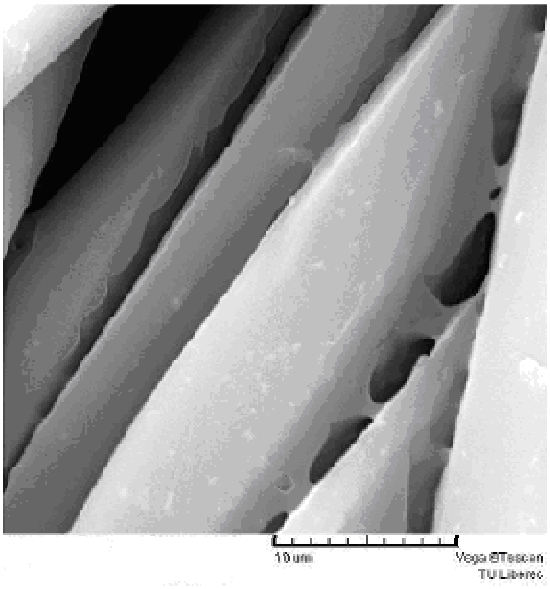

Figure 7: The PLmicro sample with a layer AC2/2Z, detail. (Electron scanning microscope VEGA)

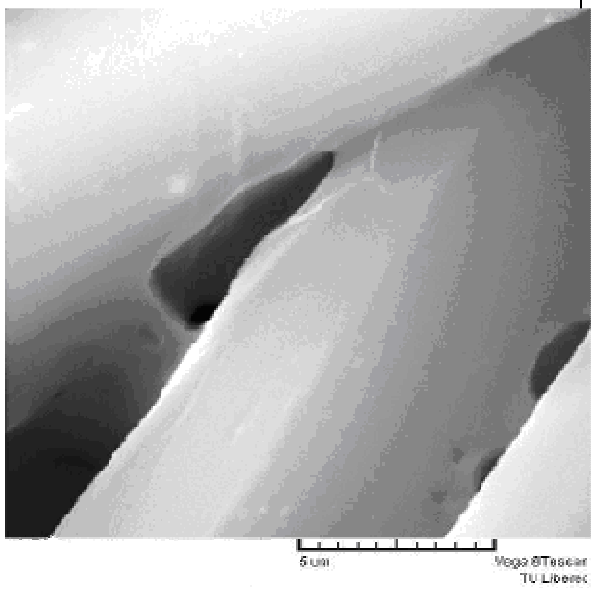

Figure 8: The PLmicro sample with a layer AC2/2Z, detail of crossing fibers. (Electron scanning microscope VEGA)

4.Conclusion

The layers on textile materials (PL, PL micro and PP) were made by soaking. The layers were prepared by the radical polymerisation (BPO at 80 ° C) and by the thermal polymerization (150°C). The thin layers on PP were prepared only by the radical polymerisation due to the low thermal resistance PP.

The scanning electron microscopy confirmed that the deposited layers were of good quality and in some places inorganic-organic layers formed bridges between the fibers. The prepared layers of fabrics changed the stiffness in bending significantly. The fabrics with the layers preparted by the thermal polymerization at 150°C have the greater bending moment than the fabrics with layers prepared by radical polymerisation (BPO, 80°C). This can be attributed to the higher degree of polymerization and thus higher strength of the materials. The plasma treatment samples prepared with the thin layers have the higher the mean values of the bending moment than the samples without the plasma treatment.
Both polyester fabrics (PL and PLmicro) with the layers significantly increased the values of the coefficients of friction (static coefficient of friction μ_{statP} and dynamic coefficient of friction μ_{dyn}). The different betwen the PP plasma treatment samples and non-plasma treatment samples are not conclusive.

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References

1. Zhang, X. a kol.: *Surface Coatings Technol*. 201, 2007, s. 6051.
2. Kovačič, V., Militký, J., Rubnerová J.: *Rastrovací elektronový mikroskop jako prostředek moderní výuky v oboru textilním*. [cit. 2009-04-13]. Dostupný na WWW: <http://www.ft.vslib.cz/depart/ktm/?q=cs/rem>.
3. ČSN 80 0858: *Zkoušení tuhosti a pružnosti plošných textilií*, Praha, Český normalizační institut 1974, 6 s.
4. Kovačič, V.: *Tření*. [cit. 2008-10-4]. Dostupný na WWW: http://www.ft.vslib.cz/depart/ktm/files/20060106/zvt_p_6a.pdf