

The Influence of Thermal Load on Composite Coating PTFE

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This article deals with the thermal stability of the PTFE coating and the composite coating composed with the PTFE matrix and titanium dioxide particles. These coatings have application in production of aluminium moulds in the automotive industry, specifically in the vulcanization of rubber materials. The first type of coating (PTFE coating) is already applied in the production and thanks to its application on the Al mould surface, the maintenance interval was increased by up to 400% compared to the uncoated state. Vulcanization of rubber compounds usually take place at a temperature 150 – 170°C. This temperature can be increased after addition of additives to a temperature 220°C. For this reasons, there is the research focused on degradation of these coatings at various thermal loads. Thermal stability was tested on coatings - PTFE a PTFE + TiO₂ – applied on aluminium substrate (type Al – Si). Experimental samples were loaded at temperatures 150°C, 200°C, 250°C, 300°C, 350°C, 400°C a 500°C at a time 30 minutes and 60 minutes. The samples after thermal load were performed to tribometric analysis, and to the surface roughness measurement of selected parameters. The samples were also evaluated from the point of view of surface morphology and state of the surface with SEM and EDS analysis.

Keywords: Surface Pre-treatment, PTFE Coating, Composite Coating, Coefficient of Friction, Thermal Stability

1 Introduction

The research described in this article has been implemented to extend the lifetime of Al moulds used to manufacture motor vehicle tires. These moulds consist from 8 to 36 segment, which after the joining creates the unit in the shape of the resulting product – tire. This moulds were made of aluminium alloys such as Al – Si and Al – Mg. Each segments is cast by successive low pressure casting. After casting, the segments are machined to the required shape and surface quality. The mould production costs are in the range from hundreds to millions of crowns.

Thus prepared moulds are capable of operating from 2500 to 2700 cycles. After this interval is necessary maintenance, which consist of shutting down moulds out of operation and cleaning of the mould working surface. Cleaning is performed mechanically, either manually using steel sanders or with stronger trapping of the impurities by applying a dry ice layer to the surface and then manual cleaning. Another possible type of cleaning is blasting fine particles of sand.

During cleaning occurs to the financial losses. The first factor is the shutdown of the production and the second is the gradual reduction of the lifetime of the moulds caused by the above-mentioned cleaning. Due to the desired savings, the teflon coating (PTFE) was applied to the moulds working surface, which after the testing and function verification caused an increase of the maintenance intervals 200 - 400% (depending on the mould type and the produced product). Using different types of micro and nano-particles into the PTFE coating should be to fur-

ther increase the interval between maintenance and increase the lifetime of Al moulds. [1]

Vulcanization of rubber mixtures takes place at temperatures of about 220°C. The previously used PTFE coating without the addition of TiO₂ particles has been tested for temperatures up to 170°C. For this reason, it is necessary to verify the thermal stability of the coatings (PTFE and PTFE + TiO₂ particles). In this experiment, abrasion resistance testing was performed on the samples (tribometric test), furthermore was performed evaluation of selected roughness parameters (how the surface roughness will affect the surface roughness) and also SEM and EDS analysis (surface and morphology analysis after thermal loading).

2 Coating technology

The coatings were applied to experimental samples prepared from the mould segments used for the production of tires. We have achieved to the authentic base material that is actually used in production. The basic material is Al-Si alloy (silumin). Experimental samples have a size of approximately 35 x 45 x 10 mm. The surface of the samples was without further mechanical treatment, for reason of the surface quality preservation, which is similar to milling after the production.

The chemical composition of the base material was verified using an optical emission spectrometer TASMAN Q4. This is an AlSi10CuNiMn alloy. The chemical composition is shown in tab. 1. It is a nonstandard Al alloy. [2]

Tab. 1 Chemical composition of basic material

Element	Al	Si	Fe	Cu	Mg	Mn	Cr	Other
Content [%]	89.41	8.51	0.63	0.005	0.508	0.05	0.03	0.86

The coating technology was performed in a chemical coating bath. These are chemical solutions with precisely defined composition and pH. The coating technology process is patented. The whole process can be divided into two phases. The first phase of the process is degreasing and zircon nanopassivation and the second phase is the application of the teflon coating. The whole process is illustrated graphically in fig. 1.

The two variants of coatings are applied in this experiment. The PTFE coating itself is applied to the surface of the base Al material in the first variant. The titanium dioxide was applied to the PTFE matrix in the case of the second variant. The coating technology requires thorough mixing of the coating bath to avoid settling of TiO₂ particles at the bottom of the coating bath. Thanks to the perfect blending of the bath we achieve the uniform coating exclusion and TiO₂ particles distribution. The particles concentration used in the solution was 0.01%. This concentration is based on experience from previous experiments. [3, 4]

3 Evaluation of the experiment

140 samples were coated in the experiment in total. Labelling of the each sample is shown in the tab. 2. The experiment included not only the influence heat resistance of the coating, but also the influence of time. The first half of the samples were loaded for 30 minutes and the second half for 60 minutes.

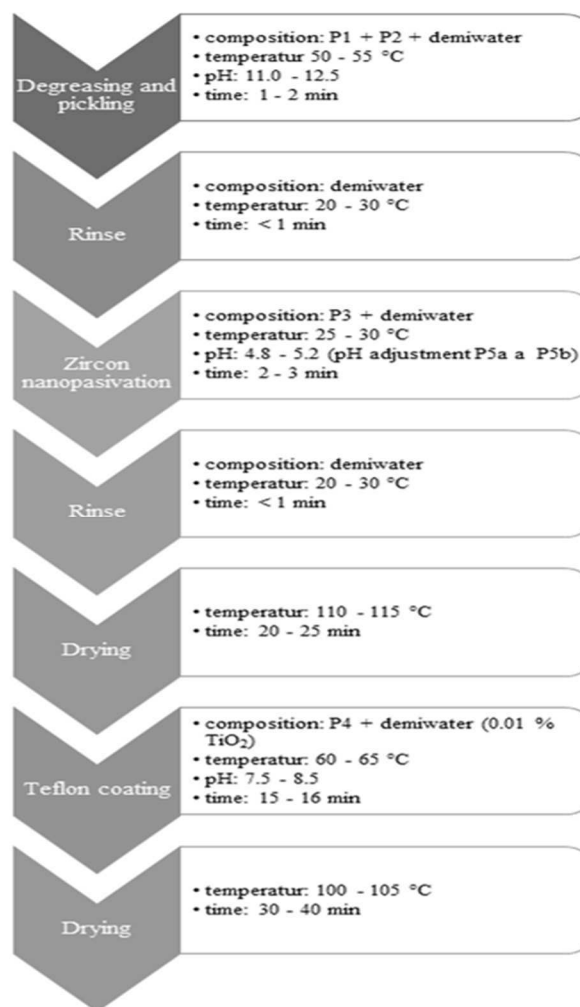


Fig. 1 Scheme of the coating process

Tab. 2 Labelling of experimental samples

Coating	Time of load	Thermal load						
		150 °C	200 °C	250 °C	300 °C	350 °C	400 °C	500 °C
PTFE + TiO ₂	30 min	1A (1, 2, 5, 6, 7)	2A (1, 2, 5, 6, 7)	3A (1, 2, 5, 6, 7)	4A (1, 2, 5, 6, 7)	5A (1, 2, 5, 6, 7)	6A (1, 2, 5, 6, 7)	7A (1, 2, 5, 6, 7)
PTFE + TiO ₂	60 min	1A (3, 4, 8, 9, 10)	2A (3, 4, 8, 9, 10)	3A (3, 4, 8, 9, 10)	4A (3, 4, 8, 9, 10)	5A (3, 4, 8, 9, 10)	6A (3, 4, 8, 9, 10)	7A (3, 4, 8, 9, 10)
PTFE	30 min	1B (1, 2, 5, 6, 7)	2B (1, 2, 5, 6, 7)	3B (1, 2, 5, 6, 7)	4B (1, 2, 5, 6, 7)	5B (1, 2, 5, 6, 7)	6B (1, 2, 5, 6, 7)	7B (1, 2, 5, 6, 7)
PTFE	60 min	1B (3, 4, 8, 9, 10)	2B (3, 4, 8, 9, 10)	3B (3, 4, 8, 9, 10)	4B (3, 4, 8, 9, 10)	5B (3, 4, 8, 9, 10)	6B (3, 4, 8, 9, 10)	7B (3, 4, 8, 9, 10)

3.1 Tribometric test - Coefficient of friction

The coefficient of friction was evaluated on a Bruker UMT TriboLab tribometer. The indenter was the 12.7 mm diameter steel ball. This ball was moved 10 mm with load 4 N (load condition 1) or 2 N (load condition 2) on the sample surface. The test was evaluated according to the standard ASTM G 132 – 96.

The values of the base material and the heat-untreated samples are shown in the tab. 3. The values of the teflon coating are shown and in tab. 4 and the values for the

composite coating composed of teflon and titanium dioxide particles are listed in the tab. 5. The values indicated in the tables are the arithmetic average of the five measured values.

Tab. 3 Friction coefficient measured on base material and heat-free samples

Sample	Coating	Time load [min]	Temperatur [°C]	Average value of COF	Measurement conditions
Basic material	0	0	0	0.148	1
6A10	PTFE + TiO ₂	0	0	0.101	1
6B10	PTFE	0	0	0.100	1

Tab. 4 Friction coefficient measured on samples with te-flon coating

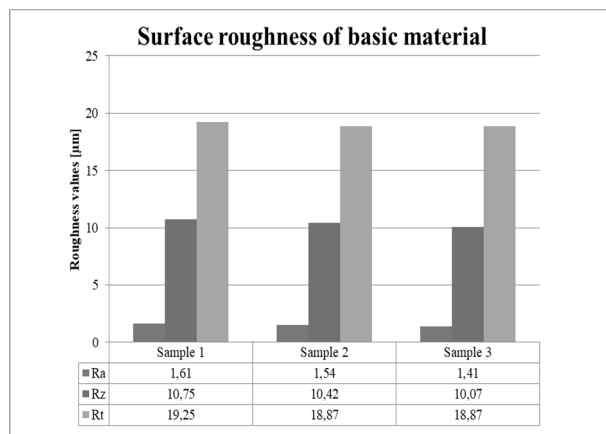
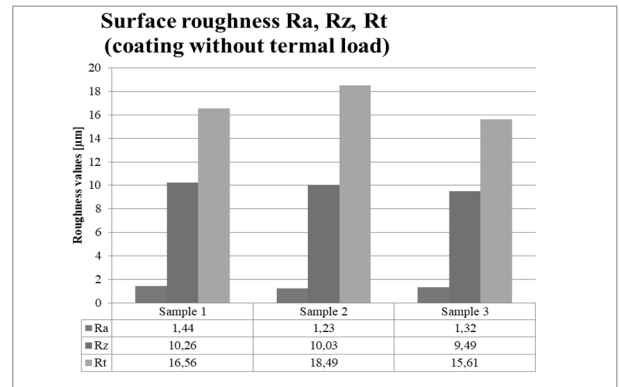
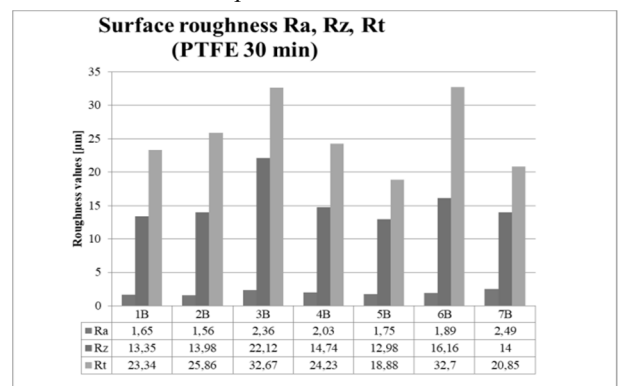
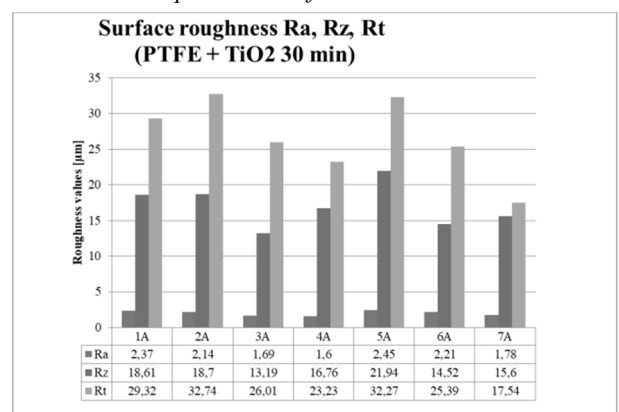
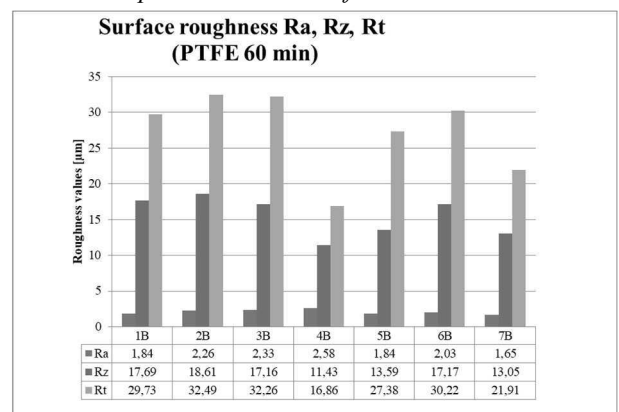
Sample	Coating	Time load [min]	Temperatur [° C]	Average value of COF	Measurement conditions
1B5	PTFE	30	150	0.163	1
1B9	PTFE	60	150	0.102	1
2B7	PTFE	30	200	0.103	1
2B10	PTFE	60	200	0.119	1
3B7	PTFE	30	250	0.182	1
1B10	PTFE	60	250	0.184	1
4B6	PTFE	30	300	0.165	2
4B8	PTFE	60	300	0.435	2
5B6	PTFE	30	350	0.861	2
5B9	PTFE	60	350	0.838	2
6B6	PTFE	30	400	0.846	2
6B8	PTFE	60	400	1.335	2
7B6	PTFE	30	500	1.197	2
7B9	PTFE	60	500	1.118	2

Tab. 5 Friction coefficient measured on composite coating PTFE + TiO₂

Sample	Coating	Time load [min]	Temperatur [° C]	Average value of COF	Measurement conditions
1A5	PTFE + TiO ₂	30	150	0.165	1
1A10	PTFE + TiO ₂	60	150	0.115	1
2A7	PTFE + TiO ₂	30	200	0.113	1
2A8	PTFE + TiO ₂	60	200	0.201	1
3A6	PTFE + TiO ₂	30	250	0.106	1
3A8	PTFE + TiO ₂	60	250	0.179	1
4A8	PTFE + TiO ₂	30	300	1.297	2
4A6	PTFE + TiO ₂	60	300	1.134	2
5A7	PTFE + TiO ₂	30	350	1.182	2
5A8	PTFE + TiO ₂	60	350	0.879	2
6A5	PTFE + TiO ₂	30	400	1.110	2
6A9	PTFE + TiO ₂	60	400	1.134	2
7A8	PTFE + TiO ₂	30	500	1.182	2
7A6	PTFE + TiO ₂	60	500	0.879	2

3.2 Surface roughness measurement

Within the measurement of surface roughness of the experimental samples, the following parameters were chosen: the average arithmetic deviation of the surface Ra [μm], the greatest height of the surface profile Rz [μm] and the total height of the profile Rt [μm]. The roughness measurement was carried out on the Hommel Tester T8000 according to ČSN EN ISO 4287. The first measurement was carried out on the uncoated base material (figure 2) and subsequently with a coating without thermal load (figure 3). The other graphs (figures 4 - 7) show the results of the measured values for both types of coating, always with the given thermal load - time and temperature.

**Fig. 2** Resulting values of the roughness parameter of the base material**Fig. 3** Resulting values of the roughness parameter of the samples without heat load**Fig. 4** Resulting values of the roughness parameter of the samples PTFE after heat load 30 min**Fig. 5** Resulting values of the roughness parameter of the samples PTFE s TiO₂ after heat load 30 min**Fig. 6** Resulting values of the roughness parameter of the samples PTFE after heat load 60 min

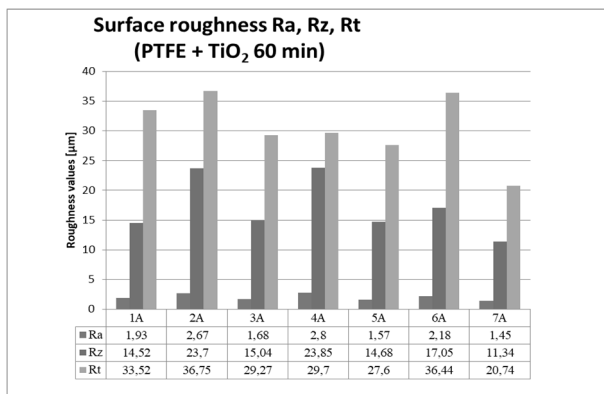


Fig. 7 Resulting values of the roughness parameter of the samples PTFE + TiO₂ after heat load 60 min

3.3 Electron microscopy – SEM and EDS analysis

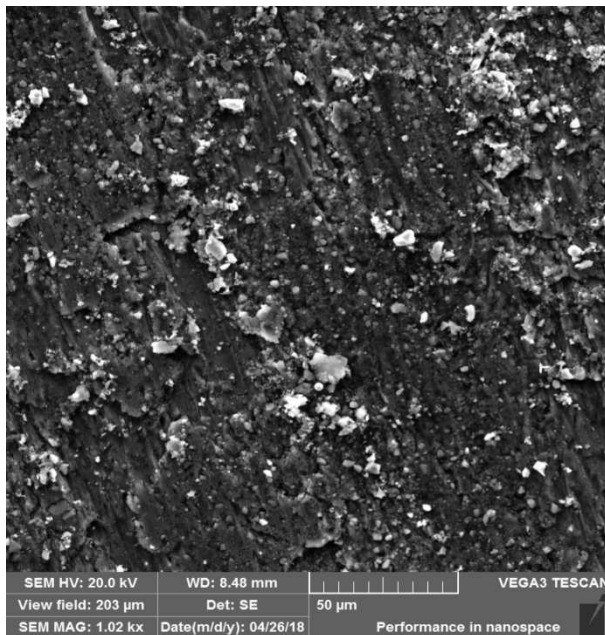


Fig. 8 SEM analysis of sample 1A6 – PTFE + TiO₂, heat load 30 min

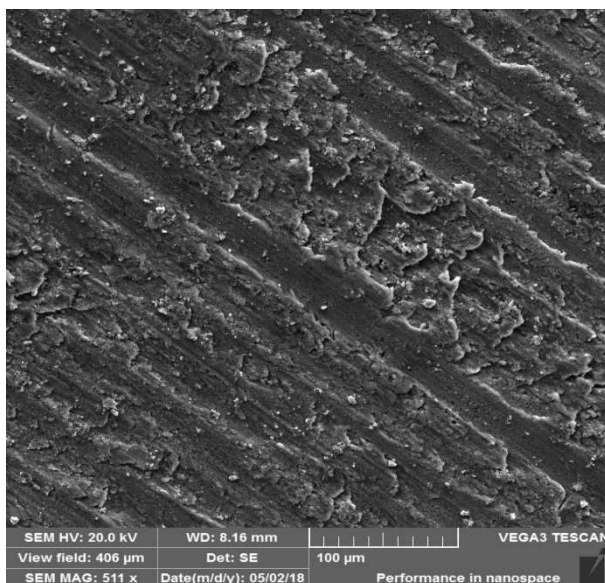


Fig. 9 SEM analysis of sample 1A8 – PTFE + TiO₂, heat load 60 min

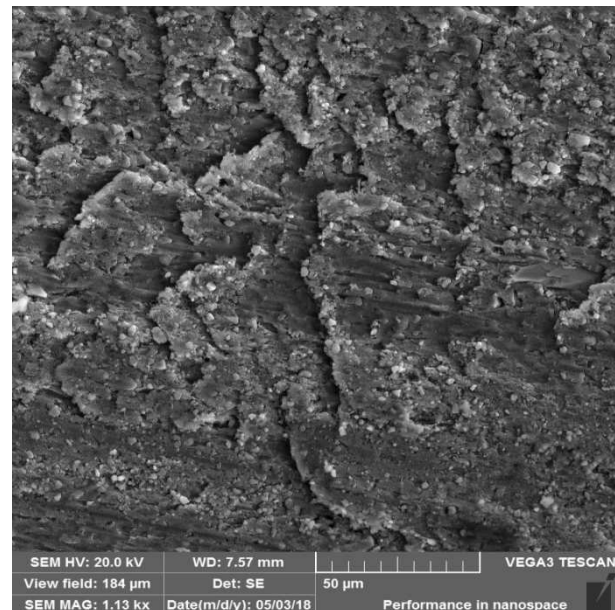


Fig. 10 SEM analysis of sample 1B7 – PTFE, heat load 30 min

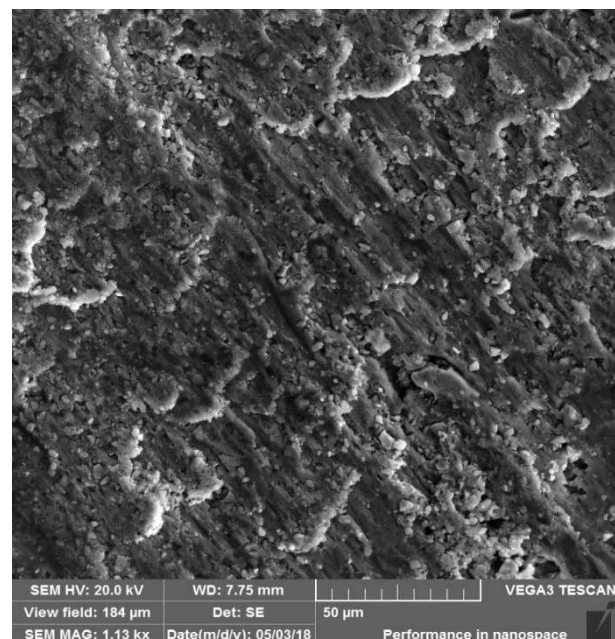


Fig. 11 SEM analysis of sample 1B8 – PTFE, heat load 60 min

Electron microscopy evaluation was performed using TESCAN VEGA 3 scanning electron microscope with a Bruker X-Flash EDS analyser. The figures 8 - 11 show selected SEM analysis figures. SEM analysis was mainly used to compare the overall state and morphology of the surface of the experimental samples. In the case of samples marked A, it was mainly documented whether the particles has been attached to the PTFE matrix. Furthermore, the uniformity of particle distribution and the total homogeneity of the coating excluded on the base material before and after the thermal loading was observed. Using the EDS analysis the contents of the chemical elements (F, C, Ti, O, Zr or impurities, etc.) were identified in the coating. Results from SEM and EDS analysis are documented in the following fig. 12, 13 and tab. 6.

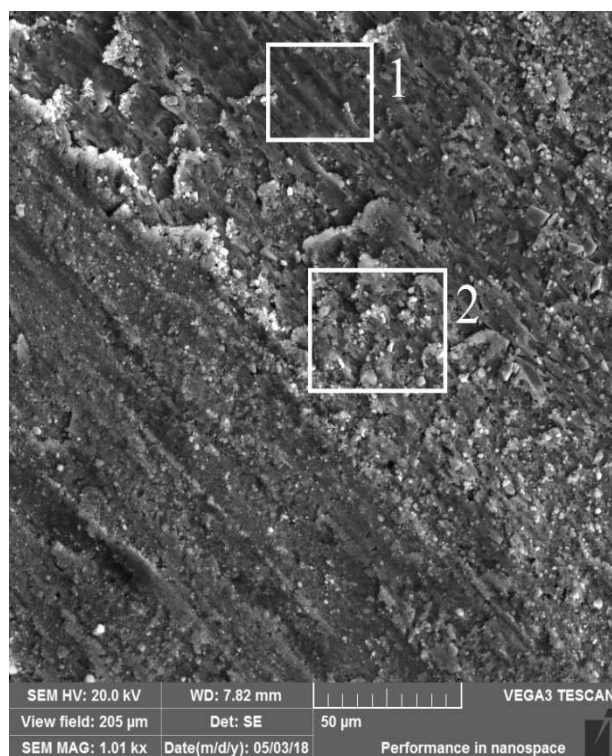


Fig. 12 EDS area analysis of composite coating PTFE + TiO₂ with heat load 300°C and 60 min

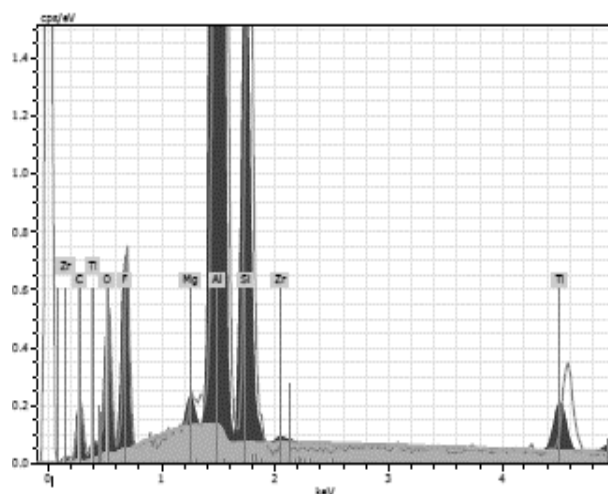


Fig. 13 Spectrum of elements of area 1 from fig. 12

Tab. 6 Chemical composition composite coating PTFE + TiO₂ with heat load 300°C and 60 min

Element	Norm C [wt. %]	Atom. C [%]
Al	52.76	41.47
C	14.10	24.90
Si	9.69	7.31
O	10.60	14.5
F	9.58	10.70
Ti	2.49	01.10
Zr	0.35	0.008
Mg	0.44	0.38

4 Conclusion

The experiment, described in this article, was based on the exclusion of two coatings types on the basic Al material surface. The first variant of the coating is teflon and the second variant is a composite coating with applied titanium dioxide particles in the teflon matrix. Both coating variants were loaded at 30 minutes and 60 minutes at 150°C, 200°C, 250°C, 300°C, 350°C, 400°C a 500°C. This experiment was focused on possible degradation of the applied coating after loading at elevated temperatures at a certain load time. The evaluation of the coefficient of friction on the tribometer, surface roughness measurement and SEM analysis was used to evaluate the condition and the resistance of the coating. On the basis of the measurements and carried out analyses, we can state that the degradation of the coating due to temperature and time almost does not occur. The coefficient of friction and roughness parameters Ra, Rz and Rt show only minimal changes, so the parameters can be marked as unchanged. The SEM analysis confirmed the results from the friction coefficient analysis and surface roughness measurement. The significant differences was not identified on the surface of experimental samples, which would fundamentally point to the disintegration of the coating due to temperature or time. EDS analysis identified the expected chemical elements on the surface of the experimental samples. Aluminium, silicon and magnesium are chemical elements of the base material, carbon, oxygen, fluorine and zircon are part of the coating, and titanium is the applied particle itself. We can conclude the following: the addition of titanium particles into the teflon matrix is at this stage of experimental works ineffective. In the next phase of the experiment, it is necessary to focus on the matrix-particle bonding and possible application of another type of matrix and of other type and size of the particles themselves.

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