EFFECT OF ADDITION OF NANOPARTICLES TiO_2 INTO PTFE COATING

Irena Lysonkova, Jan Novotny, Jaromir Cais, Stefan Michna
J. E. Purkyne University in Usti nad Labem, Czech Republic
lysonkova@fvtm.ujep.cz, novotny@fvtm.ujep.cz, cais@fvtm.ujep.cz, michna@fvtm.ujep.cz

Abstract. This article deals with a possibility of another extension longer lasting addition of coatings of titanium dioxide. The research based on the use of nanoparticles has development at present. Potential of added nanoparticles has large use. Particles of nanosize, i.e. below 100 nm exhibit other properties than larger particles and because of that this area is being explored in all possible sectors. This article deals specifically with the possibility of extending the lifetime of working surface molds made of aluminum alloy injection rubber mixtures used in automotive. Lifetime is longer lasting with PFTE coatings. Subsequently, it shows the effect of adding these particles at different mechanical properties in coatings on various surfaces (separated on a band saw, milling work, ground roughness 800 and 80 polished). In these cases roughness and hardness are evaluated. In the last part particles, their even distribution and size are identified. This is evaluated by electron microscopy.

Keywords: aluminum alloy, nanocoating, titanium dioxide.

Introduction

Nanoparticles are now areas of interest of diverse sectors, mainly because of their special properties. Nanoparticles are in size 100 nm. Particles in this size exploit physical and chemical properties at the level of atoms and molecules. The size “nano“ is not easy to achieve (it shows up the evaluations of the size with electron microscopy).

In this article there is a research in added nanoparticles, namely titanium dioxide, into polytetrafluoroethylene nanocoatings, which are applied on the working area used for injection molding of rubber compounds. The purpose of this addition is to increase the strength and durability of the said nanocoating.

Titanium dioxide and its nanoparticles

Titanium dioxide is at present very often used material by all sectors. In the size as micro it is often used mainly for coloring in food industry (E171), paints, and variety materials as polymers, ceramics and paper. These particles are of size 200-300 nm. The possibility of using this material is much wider and constantly explored, mainly in sizes below 100 nm and smaller – the size “nano“. Therefore, the use in the form of nanopowders in all possible sectors is examined, in this article specifically the automotive industry. Figure 1 shows the particle of titanium dioxide with a typical shape [8].

Nanoparticles give possibly new interesting usage. However, it is important to follow safety practices when working with nanoparticles of titanium dioxide because it can cause serious health problems [1].

Titanium dioxide exists in nature as rutile, brookite and antas. Furthermore, it is possible to prepare a monoclinic TiO_2 (B). In this experiment particles of ground tablets are used prepared in a ball mill with using ceramic grinding stones [4].

Polytertafluoroethylene coatings

This type of coating belongs to nanocoating, also we tried to implement to it nanoparticles. This coating is often referred as Teflon.

The coating technology is used for coating aluminium forms from material Al – Si and Al – Mg. In this case alloy with aluminum and silicone was used. These materials are for the production of tires in the auto move industry.

An example of the experimental data plot can be found in Fig. 2. An example of a graph of the function is given in Fig. 3. Fig. 4 shows an example of a column chart. Do not use 3D bar type charts.

The procedure of the coating is as follows: pickling – rinse in demineralised water – coating I phase -rinse in demineralised water – drying – coating II phase – drying. Coating alone extends the possibility to use it without purification of 200-400 %, which reduces wear of the mold cleaning [7].

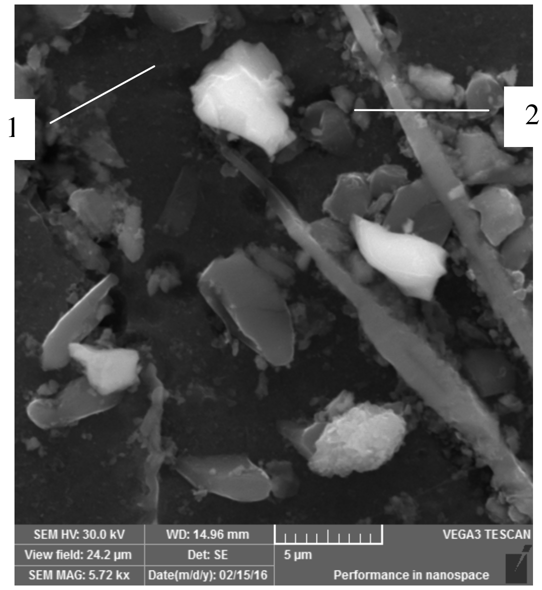

Fig. 1. **Titanium dioxide**

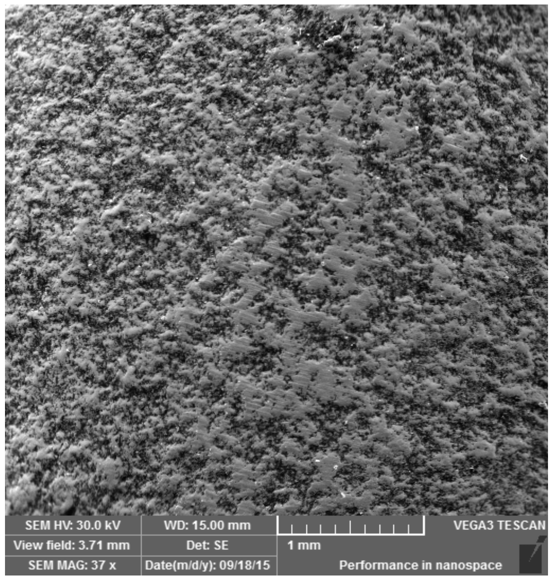

Fig. 2. **PTFE nanocoating without titanium dioxide**

The main components of the coating are C, O, F. Figure 2 shows nanocoating without addition of titanium dioxide. If we add particles it is in II phase. Further increased durability is achieved. Powder was simply thrown into the coating and stirred manually. Any increase in life forms reduces manufacturing costs.

Coated material

The used material is aluminium alloy of type Al – Si – Cu, specifically AlSi10CuNiMn. This alloy is used for low-pressure casting. Its advantage is the possibly to use it at higher temperatures, which exhibits stable mechanical properties. Because this research relates specifically to the processing of polymer material, it may indicate a temperature from 150 to 170 °C. The melt for the formation of each segment is at the casting temperature mained in the casting furnace, and therefore requires even temporal stability of the melt [3]. Recommended composition of the alloy is shown in Table 1 and it is real in Table 2.

Table 1

Suggested chemical composition of AlSi10CuNiMn

Element	**Si**	**Cu**	**Ni**	**Mn**	**Mg**	**Sr**
Content, %	8.5-10.0	0.6-1.2	0.6-1.0	0.3-0.7	0.3-0.5	0.030-0.005

Plan and preparation of the experiment

During the experiment it was necessary first to prepare the samples. They were prepared from aluminium alloy, which is stated in section 4. The specific composition of this alloy is in Table 2, which was measured by the optical emission spectrometer Q4 TASMAN. Selection of the alloy was conducted due to particular application in industrial practice.

Table 2

Averages of the measured values of roughness

Element	**Si**	**Cu**	**Ni**	**Mn**	**Mg**	**Sr**
Content, %	10.7	1.02	0.98	0.31	0.56	0.032

Individual samples were cut subsequently modified for various properties of coating. The surface was cut with a band saw, milled grinding SiC with grain 80 and 800 and polished. The main emphasis should be put to the milled and grinding surfaces, because it is closest to the surface roughness of the low-pressure casting. Other surface treatments are only experimental. For example, polished cannot be achieved in real production.

The so prepared samples were coated with PFTE coating, to which nanopowder titanium dioxide at concentration 0 %, 2 %, 5 % and 9 % was added. Subsequently were the samples dried and light wiped with a cotton pad on the ground layer of nanoparticles that not completely cling and the main concentration 9 %. Because the solution is stirred only slightly manually substantial adhesion of the coating on the bottom of the container for coating occurred.

Roughness values

Subsequently roughness was measured on the machine Hommel Tester T8000 according to the standard ČSN EN ISO 4288. Parameter measurements were R_a, R_z and R_t. First in the upright and the second time in the longitudinal direction. Each measurement was repeated ten times. Table 3 shows the average measured values.

Hardness values

Further Brinell hardness was measured at a load of 62.5 KgF HB10 by the machine ERNST Härteprüfer Sa according to the standard ČSN ISO 6506-1. For each sample the measurement was repeated ten times again and Table 4 shows their arithmetic means.

Table 3

Averages of the measured values of roughness

Finish of surface	Coating 0 % TiO_2						Coating 2 % TiO_2					
	upright			along			upright			along		
	R_a, µm	R_z, µm	R_t, µm	R_a, µm	R_z, µm	R_t, µm	R_a, µm	R_z, µm	R_t, µm	R_a, µm	R_z, µm	R_t, µm
Cutting bandsaw	13.22	71 10	117.43	2.16	12.05	19.91	12.42	62.39	141.75	2.53	13.41	2291
Milled	2.99	12.65	14.81	0.70	5.23	8.44	2.77	12.30	15.26	0.37	5.01	7.39
Grinding with grid SiC 80	1 17	8 32	11.17	1 15	7.51	10.65	0.99	5.46	7.12	1 21	8.45	10.67
Grinding with grid SiC 800	0.29	1.54	2.02	0.27	1.50	1.93	0.52	2.37	3.71	0.37	1.95	2.94
Polished	0.54	2.63	4.74	0.52	2.29	3.57	0.47	2.17	3.28	0.48	2.09	2.91
Finish of surface	Coating 5 % TiO_2						Coating 9 % TiO_2					
	upright			along			upright			along		
	R_a, µm	R_z, µm	R_t, µm	R_a, µm	R_z, µm	R_t, µm	R_a, µm	R_z, µm	R_t, µm	R_a, µm	R_z, µm	R_t, µm
Cutting bandsaw	10.82	57.61	125.24	2.96	16.56	26.83	11 03	57.25	106.53	2.04	11.57	16.53
Milled	1 90	9.52	1269	1.71	9 .55	12 33	1.71	9.37	12 33	2.18	10.48	13.79
Grinding with grid SiC 80	1 03	7.26	10.11	1 15	7. 03	9.38	1 32	8.01	12 01	0.92	5. 78	7.80
Grinding with grid SiC 800	0.21	1.15	1.82	0.11	0. 61	0.91	0.64	3. 13	4.44	0.37	1.92	2.67
Polished	0.42	2.08	3.06	0.45	2.71	3.94	0.45	2.31	3.29	0.45	2.06	2.95

Table 4

Arithmetic means of hardness values

Finish of surface	Content of nanoparticles			
	0 %	2 %	5 %	9 %
Cutting bandsaw	83	91	79	79
Milled	82	84	83	83
Grinding with grid SiC 80	82	85	82	82
Grinding with grid SiC 800	92	87	90	83
Polished	82	83	81	83

Identification and analysis

For analysis we used a scanning electron microscope TESCAN Vega 3equipped EDS analyzer Bruker X-Flash. With this machine we make analysis of the particles of titanium dioxide. This analysis is shown in Figure 3. We identified the market particles on the surface of the coating. Other particles are in the coating. The chemical composition is given in Figure 4 and Table 5.

This sample is polished with 9 % titanium dioxide. We can identify titanium and oxygen from titanium dioxide, aluminium and silicon from the basic material and fluorine, carbone and oxygen from PTFE coating.

We can see the size of particles, the evaluated particles are not "nano", they are approximately 8 µm. We can observe in this figure too small particles, which can have the size under 1 µm.

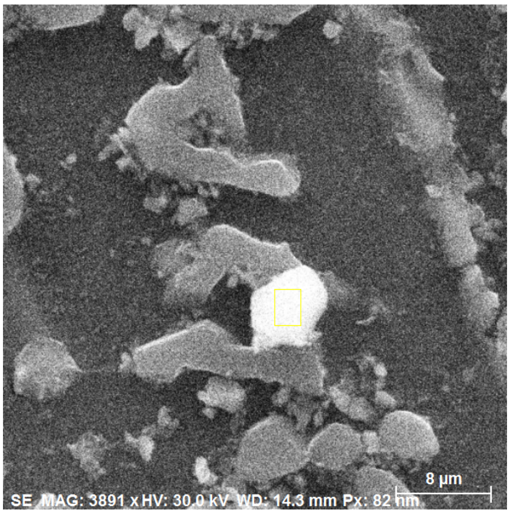

Fig. 3. **Nanoparticles of titanium dioxide observed with electron microscopy**

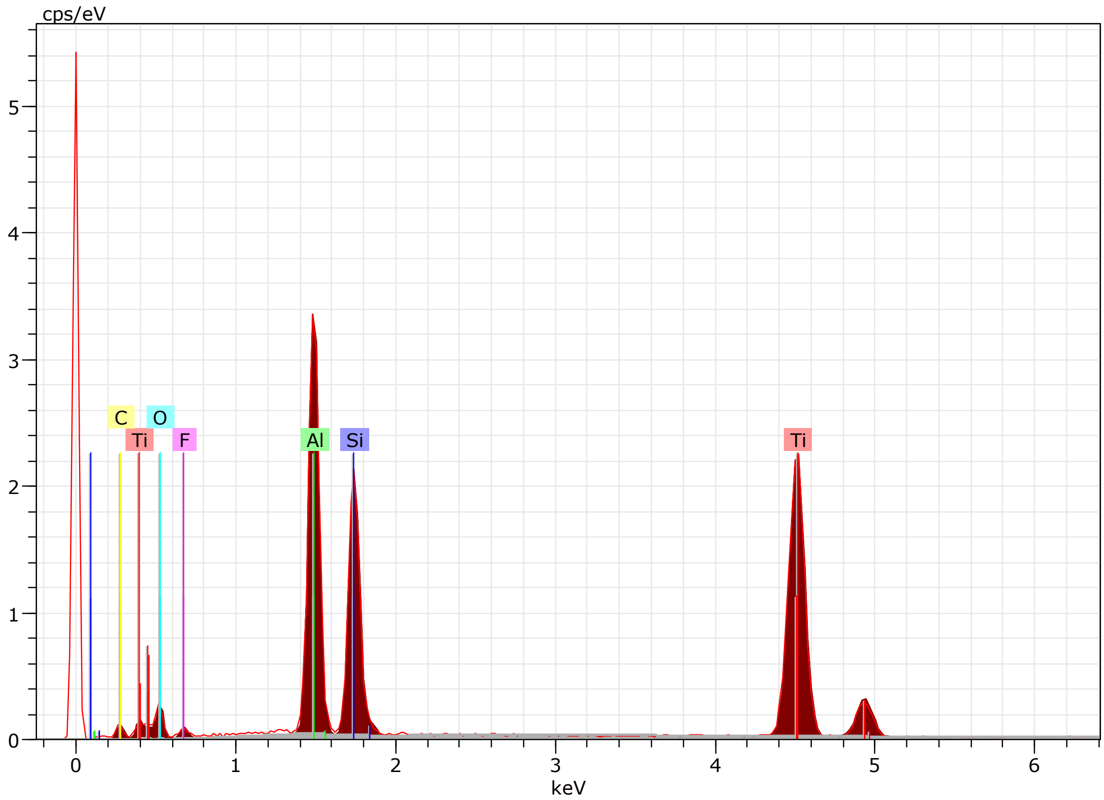

Fig. 4. **Content of individual elements**

Table 5

Chemical composition of the investigated locations

Element	Mass concentration, %	Atomic concentration, %
Titanium	25.91	12.30
Aluminium	21.43	18.06
Silicon	16.45	13.32
Oxygen	18.58	26.40
Fluorine	4.94	5.91
Carbone	12.96	24.01

Conclusions

The aim of the work was to find the possibility of extending the lifetime of working surface molds. The next goal was to find the optimum conditions and analysis of the grain resulting surfaces.

1. At the roughness measurement visible results are not reported. Measurements may be distorted by different pre-treatment of the surface.
2. Hardness measurements showed unsuitable method. From similar results, we can conclude that it was measurement of the basic material. Hardness test trough the instrument ERNST Härteprüfer is not a suitable method. A better method for measurement like this can be measuring of nanohardness, for example by DSI.
3. Electron microscopy identified the particles of titanium dioxide, the basic material and coating. In the figure we can see the biggest particles and nanoparticles. It can also be that one is a particle at the surface and the other is directly in the coating, what is desirable. Particles in solid are mixed by hand, causing sticking on the bottom. The next adequate outcome is a suitable mixing device.

Acknowledgement

This article was created due to the project No. 48202 15 0010 01 “SGS“ under the specific university research on FVTM JEPU.

References

1. Vít M., Kotlík B. Zdravotní aspekty užití nanočásticvčetně nástřiků s TiO2, SZU Praha. (In Czhech).
2. Cais J., Žihalová M., Bolibruchová D. Microstructural Analysis of Nickel Influence in AlSi10MgMn Alloy with Increase Iron Level, Manufacturing technology, 2015, Vol. 15, No. 4, p. 743-748, ISSN 1213-2489.
3. Michna Š., Cais J. CZ/28.07.15/CZA 20150521 Aluminium alloy in particular for the production of mould segment castings for forming types and the method of heat treatment of mould segment castings.
4. Svobodová J., Kušmierčak S. New trends in surface pre-treatment. Toyotarity. Value Engineering of Production Processes. Borkowski, S., Stasiak-Betlejewska, R. Fakulty of Logistics, University of Maribor: AWR Edytor, 2012. ISBN 978-961-6562-60-7.
5. Novotny J., Jaskevic M. Morphology and producing of TiO2 particles, Engineering for Rural Development, 2016 Vol. 15, Latvia, pp. 1218-1223, 2016, ISSN 1691-5976.
6. Procházka J. Studium vlastností nanočástic oxidu titaničitého. Praha, 2009. Disertační práce. Karlova universita v Praze. Prof. RNDr. Ladislav Kavan, DSc (In Czhech).
7. Užitný vzor: 13420/13:43885462 Zavedení technologie chemického povlakování Al forem za vzniku ochranných nanovrstev (In Czhech).
8. Proces výroby nano TiO2 a jeho uplatnění. Dostupné z: www.amjtj.com/projekty/proces-vyroby-nanotio2-a-jeho-uplatneni/ (In Czhech).