COMPOSITE SURFACE TREATMENT USING INORGANIC BASE STRUCTURING

KOMPOZITNÍ POVRCHOVÁ ÚPRAVA VYUŽÍVAJÍCÍ STRUKTURACI ANORGANICKÉHO ZÁKLADU

Mészáros M., Foitlová A., Steiner V., Krčmářová Ž.

SVÚM a.s., Čelákovice, Czech Republic, meszaros@svum.cz

Summary

The scope of the work was to produce and test inorganic-based surface-modified coatings with polymer impregnation, which aim to combine good heat and corrosion resistance with improved sliding and mechanical properties. Laser ablation of ceramic thermal spray coatings and impregnation by polyimide and polytetrfluorethylene based materials were carried out and tested.

Key words

Composite materials, coatings, thermal spraying, fluoropolymers, sealing, impregnation, sliding properties, corrosion resistance.

1 Introduction

One of the important parameters of metal products is their service life. One approach to extending the service life of parts and structural components, combined with improving their functional properties, is the use of special surface treatments. One promising area of such surface treatments is inorganic-based composite coatings. Main aim of the work is preparation and testing of surface-modified inorganic coatings with polymer impregnation, which promise a combination of good thermal and corrosion resistance with improved tribological and mechanical properties. Ceramic thermal spray coatings (flame/plasma) were used as the inorganic component of the composite coating. These coatings were surface-modified – structured (mechanically, by etching, by laser) and the created structure was coated by a polymer suspension. The aim was to create a surface with domains of hard and mechanically resistant ceramic domains and slippery, sealing polymer domains. One of the main challenges was therefore to create a surface with an optimal ratio of these domains in relation to the application requirements. Based on the combination of materials and the nature of the surface treatment, it is expected that a surface treatment tailored to the specific purpose can be prepared.

2 Materials and methods

2.1 Inorganic part of coating

Samples of inorganic coatings produced using atmospheric plasma and flame thermal spraying technologies were obtained for structuring tests. This group of surface treatments can be applied on an industrial scale even to more complex parts and generally provides good wear resistance, hardness, and corrosion resistance [1]. The samples are based on five types of inorganic powders applied to two types of base material (S235 steel and EN AW 2024 aluminum alloy). For good adhesion, there is always a bonding interlayer, known as a bondcoat, between the base material and the functional layer (Tab. 1, Tab. 2, Fig. 1).

Table 1: Overview of inorganic coating materials created using atmospheric plasma spraying technology

TECHNOLOGY	**ATMOSPHERIC PLASMA SPRAYING**			
BONDCOAT MATERIAL	NiCr (IKH 810), thickness approx. 50 µm			
SAMPLE SET	A1	A2	A3	A4
TOPCOAT MATERIAL	Cr_2O_3	Al_2O_3-3TiO_2	Al_2O_3-40TiO_2	Al_2O_3-13TiO_2
POWDER	Amperit 704.001	Amperit 742.001	Amdry 6257	Amdry 6224
COATING THICKNESS [µM]	220-250	230-340	240-330	280-340

Table 2: Overview of inorganic coating materials created using flame spraying technology

TECHNOLOGY	**FLAME SPRAYING**	
BONDCOAT MATERIAL	NiAl (450 NS)	
SAMPLE SET	A5	A6
TOPCOAT MATERIAL	Al_2O_3-3TiO_2	Al_2O_3-13TiO_2
POWDER	Amperit 742.001	IKH 2102
COATING THICKNESS [μM]	280-340	240-300

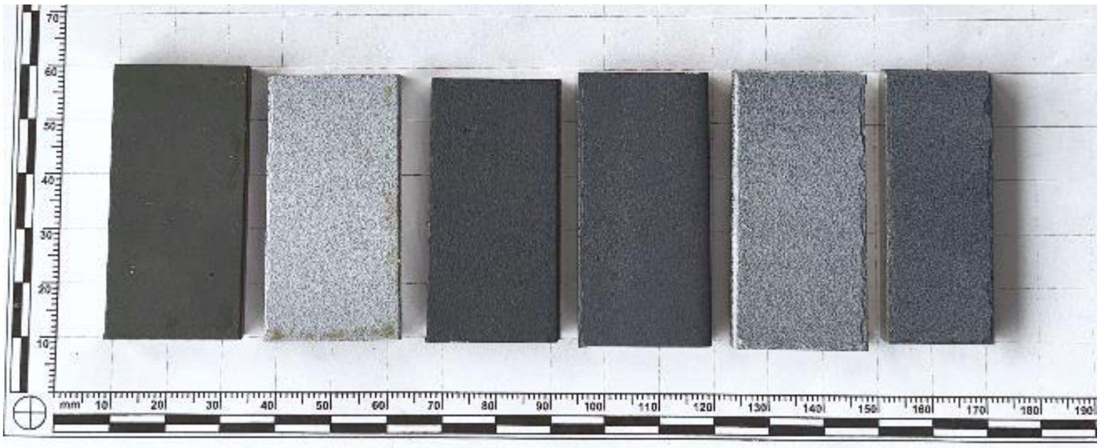

Figure 1. Samples cut from coupons with thermal spray coatings (from left to right: A1 – A6)

2.2 Structuring of inorganic coatings

Three general approaches were considered for the preparation of structured inorganic coatings:

- structuring of the created coating (chemically, physically, mechanically) [2,3,4,5,6,7,8,9],
- creation of a ceramic/metal composite spray coating with subsequent chemical dissolution of metal parts [10],
- structuring of the base material prior to the actual spraying of inorganic coatings.

Within the framework of R&D the use of laser was selected (Fig. 2). This method, in addition to structuring, can also lead to morphological changes in the inorganic layer, i.e., to additional modification of properties. This one-step technology enables highly accurate non-contact processing of almost any shaped parts. It can be applied precisely to submicrometric areas, but laser beam scanning also makes it suitable for large surface areas of several square meters [6].

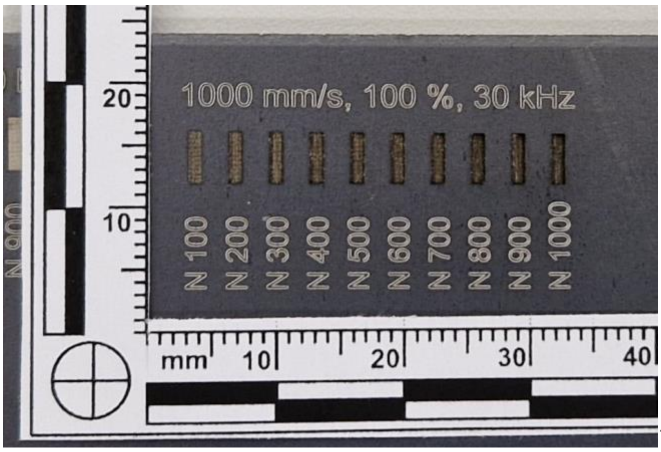

Figure 2: Steel substrate made of S235 material structured by 20 W laser with different numbers of application cycles

2.3 Composite surface treatments for sliding applications

Due to their extremely high hardness, excellent corrosion resistance, strength at high temperatures, abrasion resistance, and specific weight, ceramic materials can be an excellent material for tribological applications. Although ceramics can be used without lubrication much more successfully than metals, reliable performance of tribological elements can usually only be achieved in the presence of lubricants. For minimum friction and wear in lubricated sliding applications, it is necessary to completely separate the contact surfaces with a lubricating film [7]. The lubricating film can be formed by a liquid or solid (dry) lubricant, and its stability is highly dependent on the surface topography. In general, liquid lubricants exhibit better lubricating properties than solid ones – the coefficient of friction for liquids can be up to 1-2 orders of magnitude lower than for solid lubricants. However, replenishing and maintaining the lubricating film on the surface of the material during the friction process remains a significant problem limiting the use of liquid lubricants. This limitation can be partially overcome by integrating closed lubricating pockets into the volume of the tribologically stressed material – as it wears, the originally closed pockets reach the surface, open, and replenish the liquid to form a lubricating film. Solid lubricants (Fig. 3) have become a popular alternative to liquid and plastic lubricants due to their ability to reduce maintenance costs and environmental impact without the need for replenishment [11,12,13,14].

The aim of the work is to create a composite surface treatment consisting of surface lubrication pockets with solid lubricant and, where appropriate, a surface film of solid lubricant anchored to a hard and mechanically resistant structured inorganic coating. Pneumatic spraying of suspensions, emulsions, or solutions was chosen as the primary method of applying the sliding and sealing components.

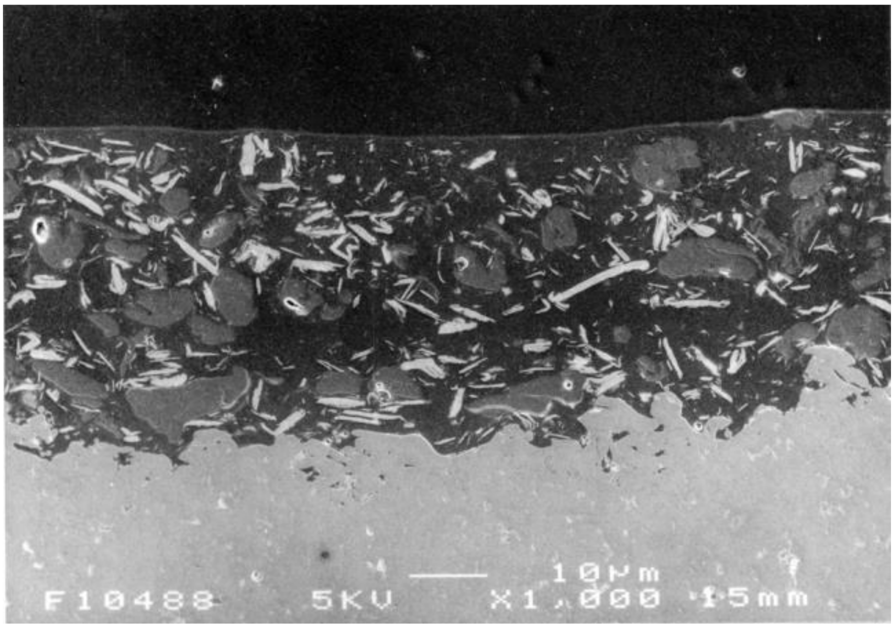

Figure 3: Microscopic image of a lubricating coating based on filled PTFE applied to a thermal spray of titanium dioxide [12]

2.4 Materials for solid lubrication

Polytetrafluoroethylene (PTFE), also known under the trade name Teflon®, is the most commonly used solid lubricant due to its chemical resistance, high melting point, and biocompatibility. Its low dry friction coefficient ($\mu < 0.2$) against various materials is attributed to the low shear strength of long chains (CF2-CF2) and the transfer of polymer material to the opposing surface, creating a thin transfer layer that results in the sliding of two soft polymer surfaces. Despite its excellent sliding properties, PTFE exhibits low wear resistance and significant viscoelastic deformation under load, which greatly limits its use in high-load applications. The use of suitable fillers can mitigate these shortcomings, reducing the wear rate of PTFE and increasing its load-bearing capacity [11]. Other polymer materials commonly used for tribological applications include epoxy resins (EP), phenolic resins (PF), aromatic thermosetting copolyesters (ATSP), polyimides (PI), polyamide-imides (PAI), polyurethanes (PU), polyamides (PA), polyetheretherketone (PEEK), ultra-high molecular weight polyethylene (UHMWPE), polyphenylene sulfide (PPS), polyoxymethylene (POM), and fluorinated ethylene propylene copolymer (FEP) [14].

Commercial products based on special polymer dispersions were selected as impregnation materials, which can be applied by manual pneumatic spraying followed by drying and curing (Tab. 3).

Table 3: Overview of selected lubricating materials

TRADE NAME	POLYMER	TYPE	DRYING AND CURING TEMPERATURE
GREBLON INS PAI 100	PAI + PFTE	suspension	10 min / 200 °C
XYLAN 8870	PTFE	suspension	10 min / 150 °C 15 min / 375 °C
DUPONT 959G-205	FEP	suspension	15 min / 150 °C 15 min / 345 °C

3 Experimental

3.1 Structuring of inorganic surfaces

The following directions were selected for the creation of structured inorganic coatings within the framework of R&D activities:

- structuring of the inorganic ceramic coating – physically (laser ablation),
- structuring of the base material prior to the actual spraying of inorganic coatings.

Laser ablation was chosen as the structuring method due to its speed of preparation, safety, adjustability (of parameters and shapes of the structures created), and scalability. The device used for the experiments is a 20 W Gweike G2 fiber laser (Fig. 4).

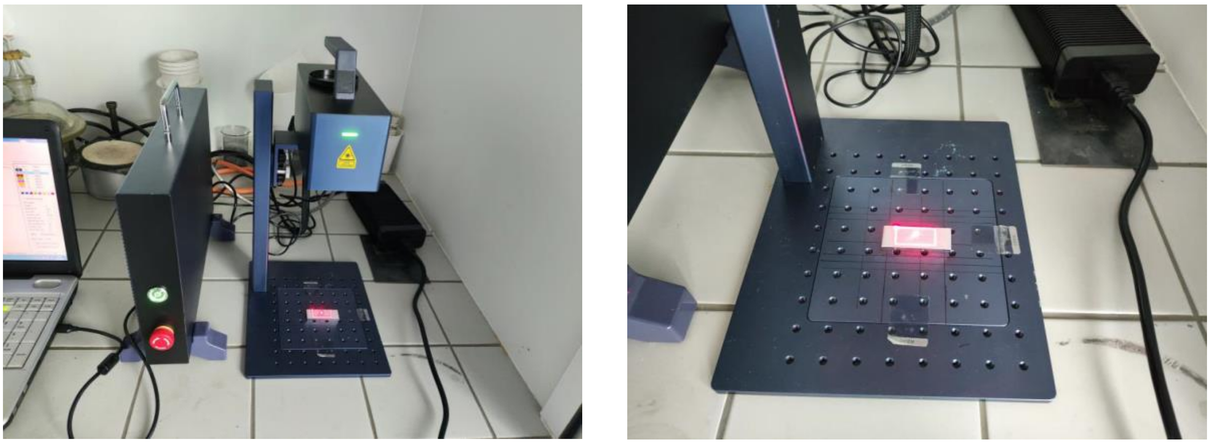

Figure 4: Gweike G2 20 W assembly (left) and detail of the work surface when setting the structure (right)

The varied laser settings parameters were: power percentage, pulse frequency, speed, and number of application cycles. It can be said that in order to achieve the greatest possible ablation depth, it is necessary to use high power, low frequency, and slower speed. Based on the trial tests, parameters were selected for structuring metal and inorganic materials (Tab. 4) which, with a suitable number of application cycles, lead to the rapid formation of deep structures without significant damage to the surrounding material (Fig. 5).

Table 4: Selected settings for the Gweike G2 laser for deep structuring of substrates

POWER	**20 W (100 %)**
SPEED	1000 mm/s
PULSE FREQUENCY	30 kHz
NUMBER OF APPLICATION CYCLES	10–1000

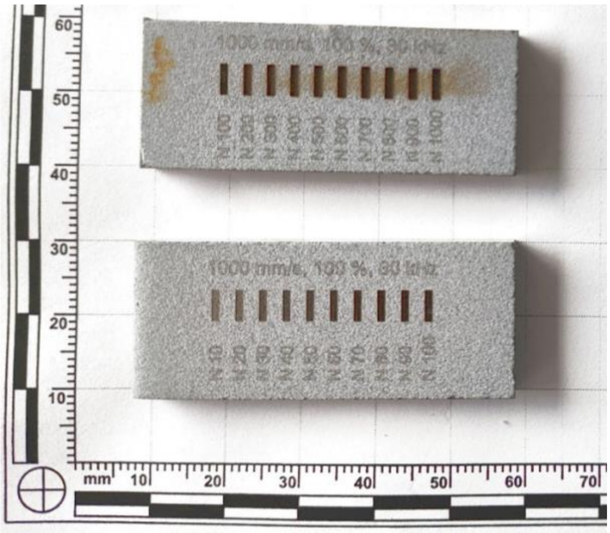

Figure 5: Samples from set A2 after testing the effect of the number of application cycles on ablation depth

3.2 Evaluation of structured surfaces

The depth profiles of structured surfaces were measured in two ways:

- *non-contact* – using an Olympus DSX1000 digital microscope and Olympus Stream software,
- *contact* – using a Surftest SJ 500P contact roughness tester with a 2 µm tip.

The result of the measurement is the average groove depth at a given laser setting (1000 mm/s, 100% power – 20 W, 30 kHz) and number of application cycles (Tab. 5, Tab. 6, Fig. 6 – Fig. 9).

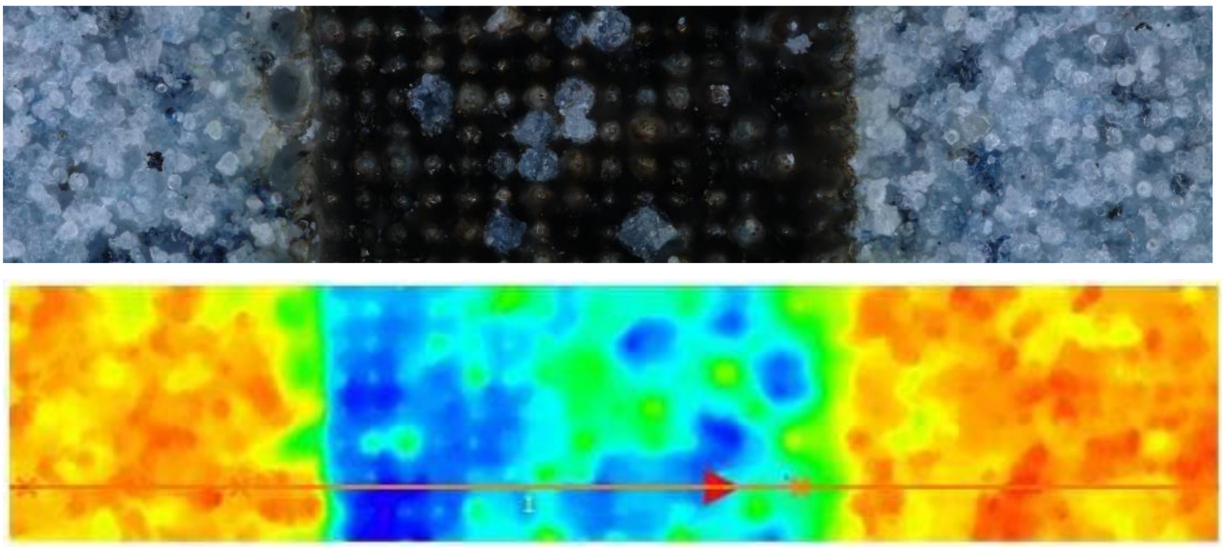

Figure 6: Characterization of the structured groove of sample set A2 using an Olympus DSX1000 digital microscope

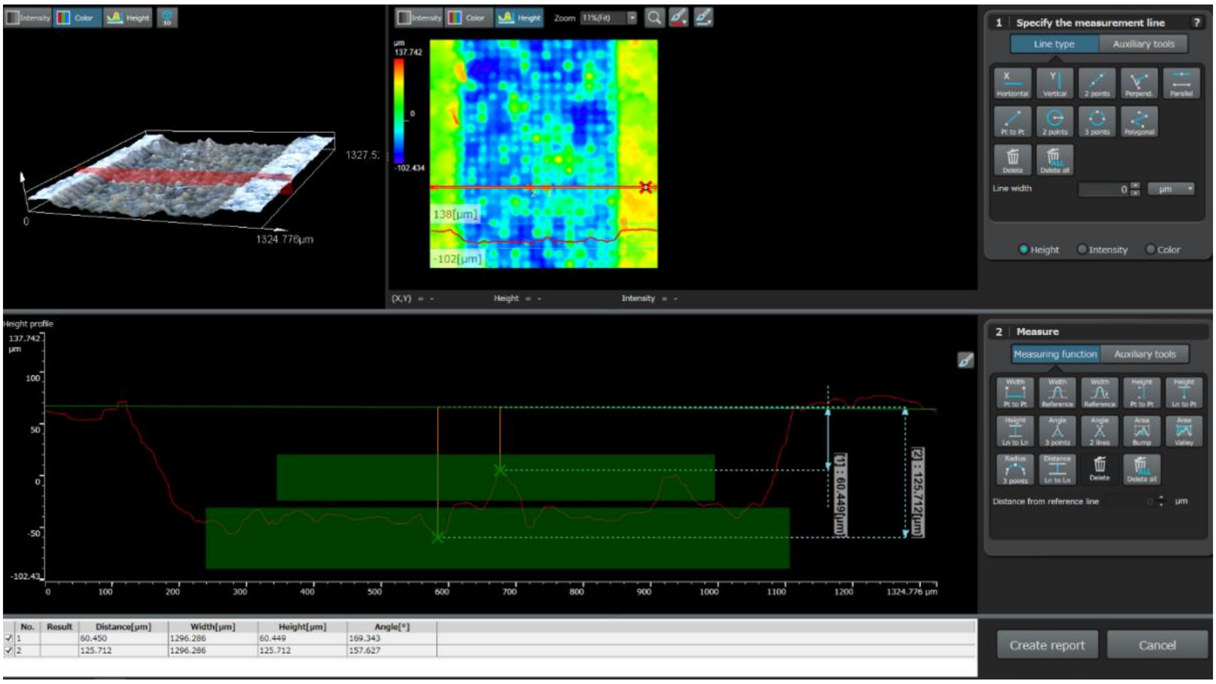

Figure 7: Example of evaluating the minimum and maximum depth of structuring on a sample from set A2 after 50 application cycles using Olympus Stream software

Table 5: Results of evaluating the depth of structuring of sample set A2 using a digital microscope

NUMBER OF APPLICATION CYCLES	**10**	**20**	**30**	**40**	**50**	**60**	**70**	**80**	**90**	**100**
AVERAGE DEPTH [µM]	40,96	64,77	76,96	86,90	104,50	97,87	108,80	139,94	128,21	139,94

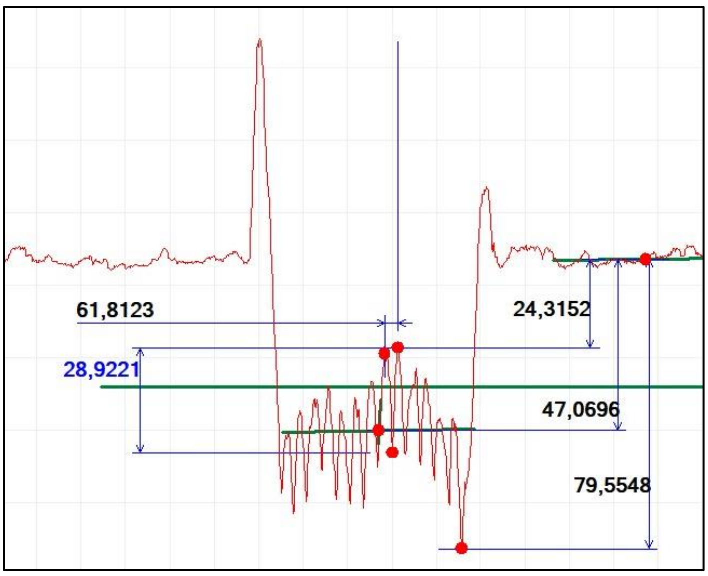

Figure 8: Example of evaluating the depth of structuring on a steel substrate using Surftest SJ 500P roughness tester software

Table 6: Results of evaluating the depth of structuring of the S235 steel substrate depending on the number of application cycles using Surftest SJ 500P roughness tester software

DEPTH [µM]	NUMBER OF APPLICATION CYCLES									
	100	200	300	400	500	600	700	800	900	1000
AVERAGE	47,07	93,04	142,10	174,53	201,70	243,95	265,35	310,58	354,03	401,41
MINIMAL	24,32	67,69	115,74	153,21	178,19	211,24	231,58	272,87	319,78	366,63
MAXIMAL	79,55	128,04	177,09	201,18	231,44	281,05	299,12	347,57	393,19	443,05

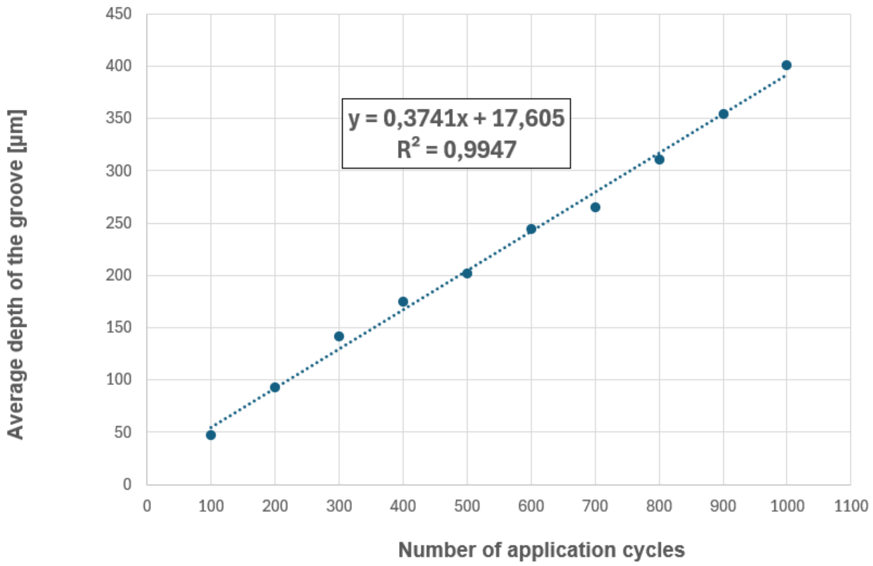

Figure 9: Dependence of the depth of structuring of the steel substrate on the number of application cycles

The measurements showed a linear dependence of the depth of structuring on the number of application cycles for the S235 steel substrate. For samples with thermal spray coatings, greater heterogeneity was observed at higher numbers of application cycles. As for example, in sample set A2, 50 application cycles were selected based on measurements – these led to a groove depth of approximately 100 µm and a homogeneous surface of the structured area.

3.3 Tribological testing

Tribological tests are used to determine tribological properties – in particular, the coefficient of friction and abrasion resistance. The materials were tested in a rotational arrangement – using the Ball-on-Disc method on a T100 tribometer from Nanovea. The principle is to press a firmly fixed ball with a defined load into a test sample (flat disc, prism, cube), which rotates at a predetermined speed and performs a predetermined number of rotation cycles or travels a certain distance (Fig. 10). Tribological testing has been used to evaluate thermal sprays, lubricating materials applied to a steel substrate, and composite surface treatments without substrate structuring.

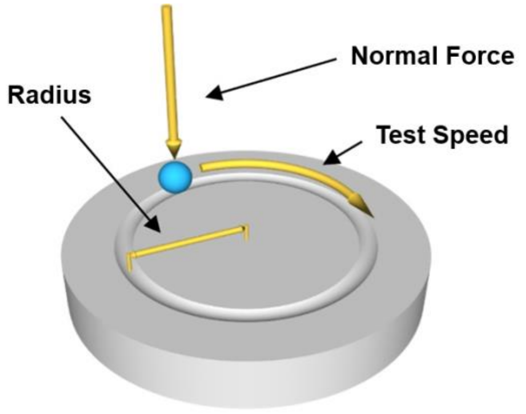

Figure 10: Principle of Ball-in-Disc method

The coefficient of friction (CoF) between the ball and the disc was used as one of the evaluation parameters for selecting the lubricating material for impregnation of the inorganic substrate (Tab. 7). This parameter is evaluated from the measurement of the forces acting on the disc. The measurement conditions were: force 10 N; speed 0.1 m/s; test duration 1 hour.

Table 7: Coefficient of friction of lubricating materials applied to a steel substrate

MATERIAL	THICKNESS [μM]	COF AVERAGE	COF MINIMAL	COF MAXIMAL
GREBLON INS PAI 100	37,0 ± 2,3 μm	0,158	0,085	0,198
XYLAN 8870	39,8 ± 3,4 μm	0,297	0,174	0,365
DUPONT 959G-205	27,1 ± 2,1 μm	0,165	0,110	0,219

As a result of tribological measurement, a groove is created in the sample, the dimensions of which (width, depth, cross-section) indicate resistance to abrasion. The dimensions of the resulting grooves were evaluated using a Surftest SJ 500P roughness tester (Fig. 11). Each groove was measured at three points and the average was calculated from the values obtained (Tab. 8). The resulting grooves are shown in the figures below (Fig. 12) and were photographed using an Olympus DSX1000 digital microscope. The sample with Xylan 8870 surface treatment showed wear through to the metal substrate.

Figure 11: Example of groove measurement after tribological testing for Xylan 8870 material on a steel substrate

Table 1: Dimensions of grooves (b – width, h – height, A – cross-sectional area) on samples after tribological testing

MATERIAL	b [µM]	h [µM]	A [µM²]
GREBLON INS PAI 100	574,3 ± 13,3	13,8 ± 0,6	5286,2 ± 309,9
XYLAN 8870	1103,9 ± 19,3	37,4 ± 1,4	27494,3 ± 1486,2
DUPONT 959G-205	739,4 ± 74,8	22,1 ± 1,8	10896,4 ± 1790,2

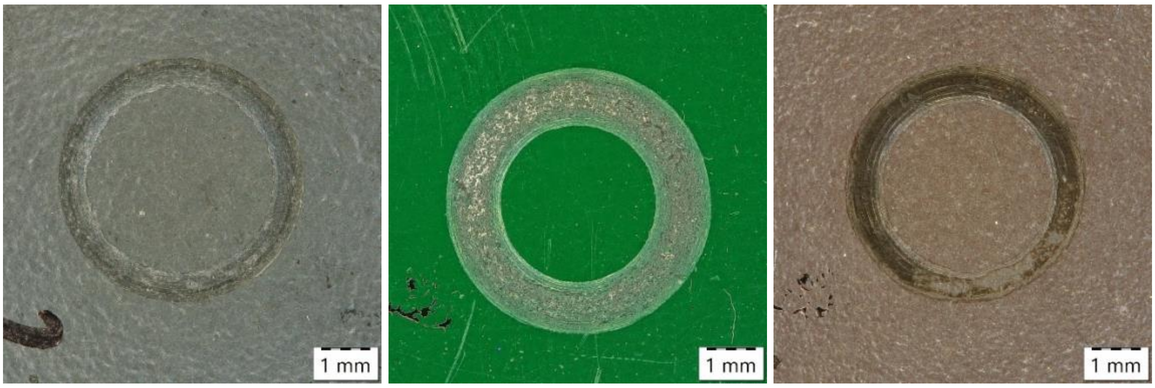

Figure 12: Grooves created during tribological measurement (from left: Greblon INS PAI 100, Xylan 8870, DuPont 959G-205)

4 Results, discussion

Sliding materials based on special polymers and ceramic thermal sprays were selected for the creation of composite surface treatments. A method of laser surface structuring was tested for anchoring the dry lubricating film/lubricating pockets, and a methodology for its evaluation was found. The parameters of the Gweike G2 fiber laser were selected to achieve deep homogeneous ablation. Based on the results of tribological measurements, the most promising combination of inorganic thermal spray (A2) and impregnation material (Greblon INS PAI 100) was selected. Following the activities carried out, the production of a composite surface treatment using the structuring of an inorganic base and its testing is underway.

5 References

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