

POSSIBILITIES FOR IMPROVING THE SURFACE QUALITY OF STRUCTURAL STEELS, INVAR AND TITANIUM ALLOYS THROUGH REACTIVE ELECTROSPARK TREATMENT WITH ELECTRODES OF AN ALUMINUM-SILICON ALLOYS

G. Kostadinov¹, T. Penyashki¹, V. Kamburov², M. Kandeveva², Mikhail I. Petrzhik³, A. E. Kudryashov³

¹ Institute of Soil Science, Agrotechnologies and Plant Protection (ISSAPP) - Sofia, Bulgaria

² Technical University – Sofia, Bulgaria

³ National University of Science and Technology MISIS – Moscow, Russia

Abstract

In the rough and semi-pure processing of metal alloys, as well as in additive technologies, surface defects are often observed - the appearance of scratches, creases, recesses, incisions, pores, ridges, protruding particles, and other unevenness, undesirable spiral or concentric relief, traces of cutting edges of the tool and high roughness on the treated surface.

In this work, the possibilities for improving the surface characteristics of materials such as steels, invar and titanium alloys with typical surface defects through reaction electrospark deposition (ESD) are investigated. A study of the conditions for the local formation of reactive phases and reduction of surface roughness during the electrospark modification was performed. Layering electrodes of low-melting Al-Si alloys were used. Their choice was made on the assumption that a liquid melt is formed in the process of ESD. It is expected that it will fill the recesses, pores and scratches and interact with the chemical elements contained in the substrate.

The results of the studies of the change in the roughness, thickness and microstructural characteristics of the modified surfaces depending on the energy of the pulses and their influence on the wear resistance at friction are presented. A double effect has been achieved - a significant improvement in both the surface characteristics and the wear resistance of the modified surfaces. It was found that the improvement of surface quality and wear resistance was achieved as a result of partially burning the protrusions, filling the depressions with low melting electrode alloy and local synthesis of wear-resistant surface structures and phases obtained by the chemical interaction of the melt with the substrate. The energy parameters of the electric discharges, the process parameters, and the ESD conditions, suitable for the realization of the above phenomena, have been determined and optimized.

Keywords: *additive technologies, selective laser melting, surface characteristics, roughness, microstructure, wear resistance, electrospark deposition*

1. INTRODUCTION

In recent decades, Additive Technologies (ATM) have increasingly entered the production of machine parts, tools, functional prototypes and everyday goods (Kondratiev 2018, Laplume, Petersen & Pearce 2016). Selective Laser Sintering / Melting (SLS/SLM), also known as DMLS for metal (Popkova 2015, Dikova, Panova & Simov 2011, Sufiyarov et al. 2018, Aloshin et al. 2016, Krolczyk, Raos & Legutko 2014) is one of the most common additive technologies for 3D printing. For the first time since 2010, the role of these technologies in industrial metalworking is gaining momentum (Kondratiev 2018, Laplume, Petersen & Pearce 2016).

One of the main disadvantages of SLM printing of metals, which prevents its mass application, is the high surface roughness and the presence of surface irregularities, pores, protrusions, cracks and more. According to the data of most literature sources (Sufiyarov et al. 2018, Aloshin et al. 2016, Krolczyk, Raos & Legutko 2014), the surface of the samples obtained by ATM is rougher and there are clearly visible traces of the layer-by-layer elaboration of the models. The roughest is the surface of the products produced by SLM technologies (Aloshin et al. 2016, Krolczyk, Raos & Legutko 2014, Alsoufi, Elsayed 2018, Musiał, Haracewiat & Kozłowicz 2015), whose values are most often between 15 and 40 µm.

It is known from tribology that such important functional characteristics as fatigue, wear resistance, friction and corrosion resistance of the bodies, strength, density, etc. largely depend on the quality of the surfaces. Rough surfaces usually have higher coefficients of friction than smooth ones and wear out faster. In order to achieve the required surface quality and to improve the properties of the manufactured parts, additional processing is required (pore filling and sandblasting of the surface, heat treatment, grinding, polishing, etc.). Successful printing of a part by 3D printing is usually the first, but not the last operation. Through various methods of additional processing, the average roughness can be reduced to only 3-4 μm (Alsoufi, Elsayed 2018, Musiał, Haracewiat & Kozłowicz 2015, Yasa, Kruth 2011). Often, post-processing can take up to 3 times longer than printing, and after these operations, dimensional accuracy decreases. (Aloshin et al. 2016, Alsoufi, Elsayed 2018, Musiał, Haracewiat & Kozłowicz 2015, Yasa, Kruth 2011, Tsvetkova et al. 2020).

Various methods for achieving better surface quality have been studied by many researchers. At present, there is no effective way to reduce surface roughness and porosity. Various methods are available, such as mechanical surface treatments, special pressing, re-melting and others (Sufiyarov et al. 2018, Aloshin et al. 2016, Yasa, Kruth 2011, Tsvetkova et al. 2020), but they provide for the addition of additives for further processing and the use of special technological regimes for different materials and shapes of products.

For this reason, in the present work, the possibilities for reducing the roughness and surface defects and improving the surface structure and properties by reaction electrospark layering of surfaces obtained after rough and semi-pure machining are also investigated. Electrospark deposition (ESD) is a cheap, effective and universal tool for local application of single and multilayer coatings with different purposes on metal surfaces. This method offers fast, easy and high-quality coating onto parts of various sizes and shapes. Its main advantages are high adhesion of the formed coatings, the possibility for local surface treatment, easy and affordable technology and equipment, low energy consumption and high environmental friendliness of the process (Gitlevich et al. 1985, Kostadinov et al. 2020), possibility to attach the layering module to the 3D printer and to use the file to build the part for application of coating.

A remarkable advantage of ESD is the melting and deposition of high melting point electrode materials on metal substrates and the achievement of metallurgical bonding with low thermal power. In the rough and semi-pure processing of difficult-to-machine metal alloys, as well as in additive technologies, surface defects are often observed - appearance of scratches, creases, recesses, incisions, pores, ridges, protruding particles, and other unevenness, undesirable spiral or concentric relief, traces of cutting edges of the tool and high roughness on the treated surface. After cutting, milling and other rough machining operations, finishing is usually carried out. Without finishing, many surfaces would have a much shorter life as a result of higher wear.

However, pure and finishing machining requires specific processes to achieve the required surface roughness and quality, including appropriate specific fixtures, tools, materials and equipment, which leads to increased costs and processing time. In certain cases, these treatments, the time and cost of their implementation, can be reduced through ESD coatings. ESD also implies increased wear resistance and longer service life of layered surfaces than in pure and finishing machining.

In this regard, the aim of the present work is to investigate the possibilities for improving the quality of surfaces obtained by ATM-SLM, as well as after rough and semi-pure machining by reaction electrospark surface modification.

2. MATERIALS AND METHODS

2.1. Materials, equipment for ESD and deposition conditions

In the present work, a new type of coatings obtained by local non-contact electrospark deposition (LESD) using a rotating electrode (Antonov, Panayotov & Lyutakov, 1980) and by ESD with a vibrating electrode is investigated. The research was conducted in two directions:

- Studies for filling the cavities with low-melting melt, which is formed during rapid local melting of the electrode in the conditions of pulse dilution with further occurrence of local synthesis of wear-resistant phases as a result of chemical interaction of the melt with the substrate and the elements of the air;
- Investigation of the occurring changes in the surface structure of the coating for the establishment of existing defects in its structure, as well as for establishing empirical dependences of the qualitative characteristics, composition, structure and properties of the coatings on the process parameters, which allow determining appropriate process parameters for ESD.

2.2. Substrate

Model plates of the popular metal alloys for ADT are used:

- Martensitic steel 1.2709 - obtained by SLM directly and after laser smoothing;
- Titanium alloys Ti-6Al-4V and GR2 (AISI UNS R R56200 and R50400) with different initial roughness - obtained by SLM and electroerosion cutting (EDM);
- Iron-Nickel alloy Invar (FeNi36) / 1.3912 - obtained by cutting a lathe with rough and semi-pure modes;
- Nickel superalloy Inconel 718, which is widely used in SLM - obtained by cutting a lathe with different roughness;

The chemical composition of the alloys is given in Table 1.

Table 1. Chemical composition of substrate plates

Element, %	C	Cr	Si	Mn	Ni	Mo	Ta	Ti	Co	V	Fe	Al
1.2709 steel	0.03<	-	1≤	1≤	17-19	4-6	-	0.9-1.1	9-11	0.15≤	Bal.	0.03≤
Ti6Al4V (GR5)	0.08≤	0.25≤	-	-	-	-	-	Bal.	0.25≤	3.5-4.5	0.04≤	5.5-6.75
Inconel 718	0.8≤	17-21	0.35≤	0.35≤	50.0-55.0	2.8-3.3	4.75-5.5	0.65-1.15	1≤	-	Bal.	0.2-0.8
Invar 36	0.1≤	0.15≤	0.2≤	0.2-0.4	35-37	-	-	-	0.5≤	-	Bal.	-

2.3. Equipment for ESD and deposition conditions

“Carbide Hardedge” (England, USA) apparatus- Fig.1 is used with the following parameters: Short circuit current - 0.2-2.5 A, Voltage- $U=80$ V, oscillation frequency of the vibrator - 100 Hz. The individual layering modes are numbered from 1 to 6 in the order of increase of pulse energy given in Table 2.

Table 2. Regimes for ESD whit vibrating electrode

№ of regimes	1	2	3	4	5	6
Capacity, μF	1.5	3.5	5	7	10	20
Pulse energy $E \cdot 10^{-2}$, J	0.5	1	1.6	2	3	7



Fig. 1. Device for manual electrical discharge deposition with vibrating electrode.



Fig. 2. Machine Elfa 541 for contactless LESD.

For local contactless ESD (LESD), by machine type "Elfa 541"- Fig.2, (Antonov, Panayotov, & Lyutakov 1980) was used with the following parameters of the regime: current - $I=1.6\div 32$ A; voltage - $U=90$ V; capacitance - $C=0.22\div 5$ μ F; pulse duration $T_i=3\div 20$ μ s; pulse fill factor $\tau=0.1$; 0.2 - (pulse frequency - $5\div 66$ kHz); single pulse energy $E_i=U.I.T_i=0.005\div 0.05$ J; coating speed $0.5\div 1.0$ mm/s. Number of passages of the electrode – $n=1\div 4$. The rotation speed of the electrode was 1500 rpm.

2.4. Electrode Materials

The use of electrodes of low-melting alloys of aluminum (Al) with silicon, Al-Si as a result of plasma discharges will can it easier to melt and penetrate the melt and fill the scratches obtained after machining (Kostadinov et al. 2020, Levashov et al. 2011, Mukanov et al. 2019, Milligan, Heard & Brochu 2010, Cadney et al. 2009). Composite materials of Al-Si12 alloy were selected for electrodes to provide both a higher amount of transfer from the liquid phase and obtaining new additional wear resistance in the process of forming the coatings.

The electrodes are obtained by a special technology developed at NITU Missy - Moscow with chemical composition in wt. % - Si-10-13, Fe $\leq$ 0.7, Cu $\leq$ 0.6, Mg $\leq$ 0.1, Ti $\leq$ 0.1 Zn $\leq$ 0.3, Zr $\leq$ 0.1, Al-balance (Levashov et al. 2011, Mukanov et al. 2019).

2.5. Research Apparatus and Measurement Methodology

The surface roughness Ra, μ m and thickness δ , μ m of the resulting coatings are measured by using profilometers - "Surtronic 3+", TESA Rugosurf 10-10G, and AR-132B at EN ISO 13565-2:1996 and DIN 4776 standards.

The microstructure and microhardness (HV) of the coatings have been studied by optical microscopy on cross-sectional under indentation loads of 20 g by metallographic microscopes Neophot 22. Microhardness of the layer, of the sub-layer (in depth to the base), and of the base itself has been measured.

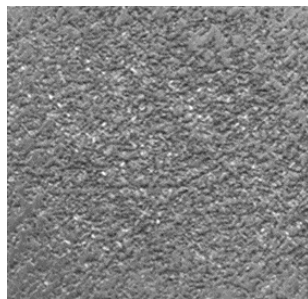
The phase identification, the distribution of elements in the surface layer and microstructural analysis of the coatings along with the interface region were performed by an X-ray diffractometer Bruker D8 Advance in "Cu K α " radiation, by optical microscope, and an electron microscope "Bruker"; by scanning electron microscopy (SEM) "EVO MA10 Carl Zeiss".

The comparative friction tests were performed with tribotester type "Thumb-on-disk" under dry surface friction with hard-fixed abrasive particles in plane contact at the following conditions: load 4 N; nominal contact area 2.25×10^{-6} m²; nominal contact pressure 1.74 N/cm²; disk rotation speed 60 rpm; sliding speed 0.239 m/s; type of abrasive surface - Corundum № 1200, ambient temperature 22 °C. The test method consists of measuring the mass wear " m " of the samples for a specific friction path " L " (friction cycles) under load " P " and glide speed " V ". The mass wear is obtained as the difference between the initial mass of the sample m_0 and its mass m_i after a certain number of friction cycles: $m= m_0-m_i$, mg. The mass of the samples before and after a given friction path is measured with an electronic balance WPS 180/C/2 to the nearest 0,1 mg.

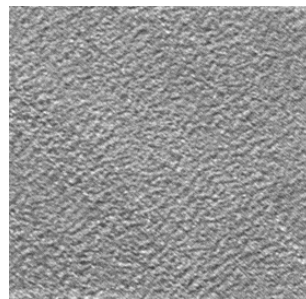
3. RESULTS AND DISCUSSION

3.1. Coating characterization - Roughness R_a and thickness δ , structure and micro-hardness of coatings

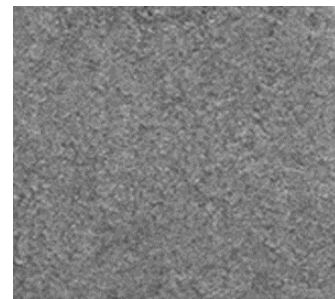
Dense and relatively uniform coatings, similar in structure and composition with acceptable repeatability of quality characteristics, were obtained on the substrates with different roughness. The general view of the topography of the surface of the coatings applied on the studied substrates is shown in Figs. 3÷6 at different microscopic magnifications. The images of the microstructures obtained on the surface of the composite coatings indicate the formation of a coating from a melt. The comparison of the topography of the surface can give some preliminary information about the surface structure and the tribological properties of the coatings.



a) Steel 1.2907- after ATM
 $R_a=13.5$, $R_z=38.2$, $R_q=16.2$,
 $R_t=38.6$

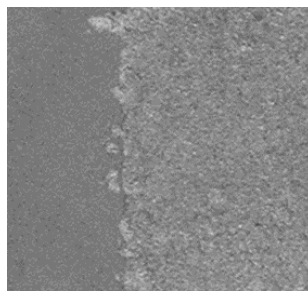


b) Steel 1.2907- after ATM end
after subsequent laser
smoothing – $R_a=7.76$, $R_z=21.9$,
 $R_q=7.84$, $R_t=23.2$

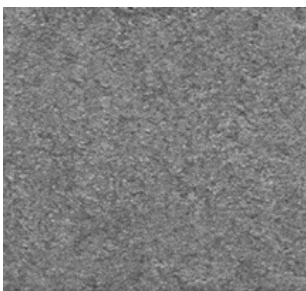


c) Steel 1.2907- after ATM and
after ESD at $E=0.04$ J –
 $R_a=4.22$, $R_z=11.8$, $R_q=4.8$,
 $R_t=8.2$

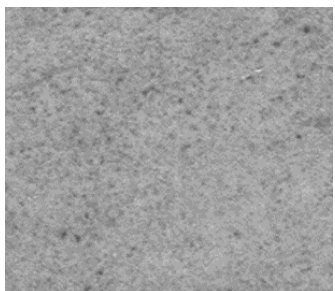
Fig. 3. General appearance of plates with different roughness of 1.2709 steel before (a,b) and after ESD-(c)



a) Ti-GR2 – after EDM (left)
and after ESD at $E=0.04$ J
(right)
Initial $R_a=2.9$, $R_z=8.5$,
 $R_q=3.48$, $R_t=10.5$ coating
 $R_a=4.2$, $R_z=12,11$, $R_q=4.25$,
 $R_t=12.9$

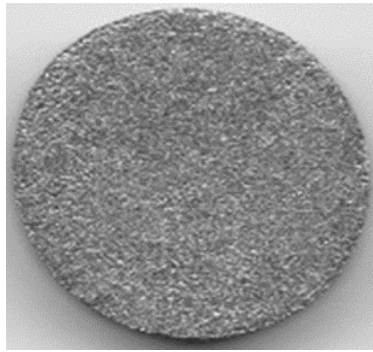


b) Ti-GR2 – after EDM and
after ESD at $E=0.07$ J –
 $R_a=5.2$, $R_z=14.7$, $R_q=5.1$,
 $R_t=15.8$



c) Ti-GR2 – after EDM and
after ESD at $E=0.02$ J- $R_a=2.7$,
 $R_z=7.5$, $R_q=2.82$, $R_t=8.12$

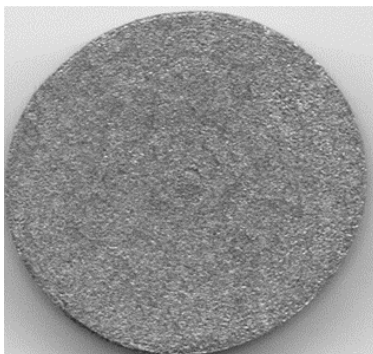
Fig. 4. General appearance of plates with different roughness after EDM and after ESD with AlSi12 electrode



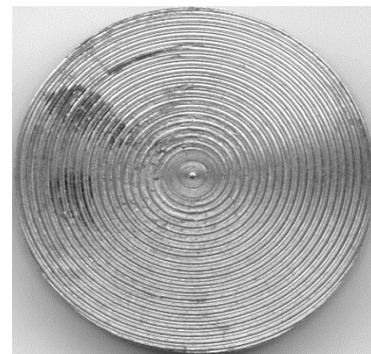
a) Al-Si12 on initial invar, after ESD at $E \approx 0.07$ J, $R_a=5.56$, $R_z=15.7$, $R_q=5.65$, $R_t=15.92$



b) initial invar after rough turning, initial: $R_a=10.7$, $R_z=30.2$, $R_q=12.24$, $R_t=29.64$



c) Al-Si12 on initial invar, after ESD at $E \approx 0.04$ J, $R_a=3.84$, $R_z=10.9$, $R_q=3.96$, $R_t=11.12$

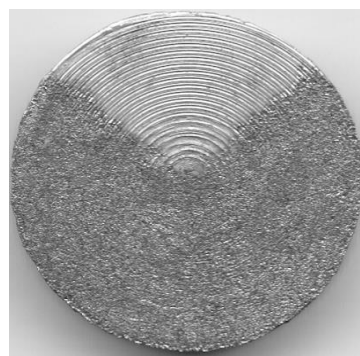


d) initial invar after semi-pure turning, initial $R_a=7.8$, $R_z=22.5$, $R_q=8.2$, $R_t=22.7$

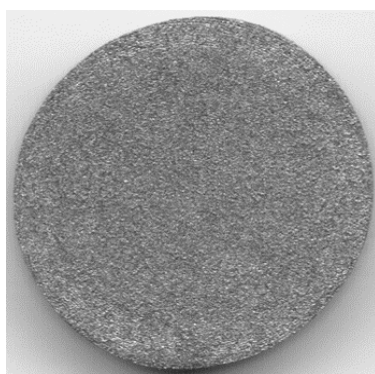
Fig. 5. General appearance of AlSi12 coatings on invar with different initial roughness



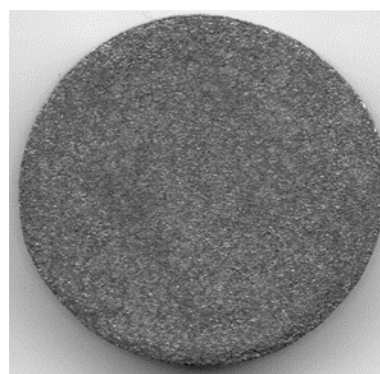
a) Inconel surface after pure turning,
and after ESD at $E=0.04$ J:
initial $R_a=3.2$, coating $R_a=3.55$



b) Inconel surface after semi-pure turning,
and after ESD at $E=0.07$ J:
initial $R_a=9.55$, coating $R_a=5.24$



c) Inconel surface after pure turning,
and after ESD at $E=0.02$ J:
initial $R_a=3.2$, coating $R_a=2.95$



d) Invar surface after pure turning,
and after ESD at $E=0.02$ J:
initial $R_a=3.2$, coating $R_a=3.56$

Fig. 6. General appearance of AlSi12 coatings on inconel with different initial roughness

The coatings form on the surface of the substrates a heterogeneous structure with a special relief. In this structure, it is distinguished between electroerosion craters and the smooth sections between them. Table 3 shows the roughness parameters of some of the coatings obtained by ESD with a vibrating electrode and by non-contact layering - LESD on substrates with different initial roughness, depending on the pulse energy. The maximum thickness at which a relatively uniform coating with surface roughness up to $R_a=3-5 \mu\text{m}$ is obtained is in the range $15 \div 16 \mu\text{m}$ at mode 6 with pulse energy $E=0.07$ J - Table 2.

Table 3. Roughness and thickness δ of coatings on different substrates with different initial roughness

№	Substrate	Process parameters	$R_a, \mu\text{m}$	$R_z, \mu\text{m}$	$R_q, \mu\text{m}$	$R_t, \mu\text{m}$	$\delta, \mu\text{m}$
1.1	Ti, GR2	Uncoated, after EDM	2.9	8.5	3.48	8.9	-
	Ti, GR2	ESD at $E=0.03$ J	4.27	12.1	4.23	12.9	10
	Ti, GR2	ESD at $E=0.07$ J	4.92	14.18	4.85	14.85	15
	Ti, GR2	LESD at $C=0.2 \mu\text{F}$, $T_i=12$, $I=16$ A	1.22	3.45	1.32	3.5	6
	Ti, GR2	LESD at $C=0.7 \mu\text{F}$, $T_i=12$, $I=16$	1.29	3.64	1.3	3.6	8
	Ti, GR2	LESD at $C=1 \mu\text{F}$, $T_i=12$, $I=16$ A	1.7	4.1	1.8		9

	Ti, GR2	LESD at C=4.4 μ F, Ti=20, I=24A	2.9	8.2	2.9	8.6	14
1.2.	Ti6Al4V	After EDM	4.32	12.6	4.47	13.5	-
	Ti6Al4V	LESD at C=0.2 μ F, Ti=12, I=16A	1.7	4.8	1.92	4.66	7
	Ti6Al4V	LESD at C=4.4 μ F, Ti=20, I=24A	2.87	8.02	2.68	8.46	11
	Ti6Al4V	ESD at E=0.02J	3.08	8.70	3.12	8.80	9
	Ti6Al4V	ESD at E=0.03J	3.56	10.1	3.6	10.16	11
	Ti6Al4V	ESD at E=0.07J	3.83	10.83	3.87	10.94	16
3.1	invar	After turning	9.75	27.6	9.875	28.825	-
	invar	ESD at E=0.03J	4.275	12.1	4.225	12.8	14
3.2	invar	After turning	12.55	35.55	12.75	37.05	-
	invar	ESD at E=0.07J	5.1	14.388	4.575	15.3	17
3.3	invar	After turning	7.8	22.5	8.2	22.7	-
	invar	ESD at E=0.04J	3.84	10.9	3.96	11.12	14
4.1	1.2709	After ATM	13.5	38.2	16.2	38.6	-
	1.2709	ESD at E=0.07J	5.9	16.6	6.2	16.8	15
	1.2709	ESD at E=0.04J	4.22	11.8	4.8	8.2	12
4.2	1.2709	After ATM	22.1	64.9	23.73	65.7	-
	1.2709	ESD at E=0.07J	8.3	29.1	8.4	31.2	16
4.3	1.2709	After ATM+ II SLM	7.76	21.9	7.84	23.2	-
	1.2709	ESD at E=0.03J	4.22	11.8	4.8	8.2	11
	1.2709	ESD at E=0.07J	5.96	16.8	6.2	17	15
4.4.	1.2709	After ATM+ II SLM	12,25	34,77	12,48	36,88	-
	1.2709	ESD at E=0.07J	6.5	18.9	6.8	19.1	16
4.5.	1.2709	After ATM	16.9	47.9	17.9	48.4	-
	1.2709	LESD, I=24A, C=4.4 μ F, Ti=20 μ s	7.5	21.1	7.44	22.9	14
	1.2799	LESD, I=24A, C=4.4 μ F, Ti=12 μ s	4.58	12.92	5.2	13.6	11

Figure 7 shows typical SEM images of the topography of the coatings on Ti-GR2 and invar. The combination of the observations with optical and electron microscopy Fig. 3-6 and the results of the measurements of the roughness parameters allows to ascertain that the coatings form on the surface of the steel parts a heterogeneous structure with a special relief, different from the initial one. It can be seen that for surfaces obtained both by ATM (Fig. 3) and after rough and semi-pure machining (Figs. 5 and 6) with an initial roughness $Ra > 4.5 \mu m$, the reaction electrospark surface modification with AlSi12 electrode allows to smooth out the unevenness of the previous treatment, fill the pores and cavities and obtain surfaces with improved uniformity and reduced roughness. The results obtained show that ESD coatings in most cases do not need further processing. They have improved geometrical characteristics – high density and uniformity, lower roughness, and lower scratches and bumps than those of the initial surface.

The coatings applied on surfaces obtained after a finishing mechanical machining treatment with roughness $Ra < 3 \mu m$ sharply change the morphology of the surface - Fig. 3, but their roughness is commensurate, or higher than that of the uncoated samples.

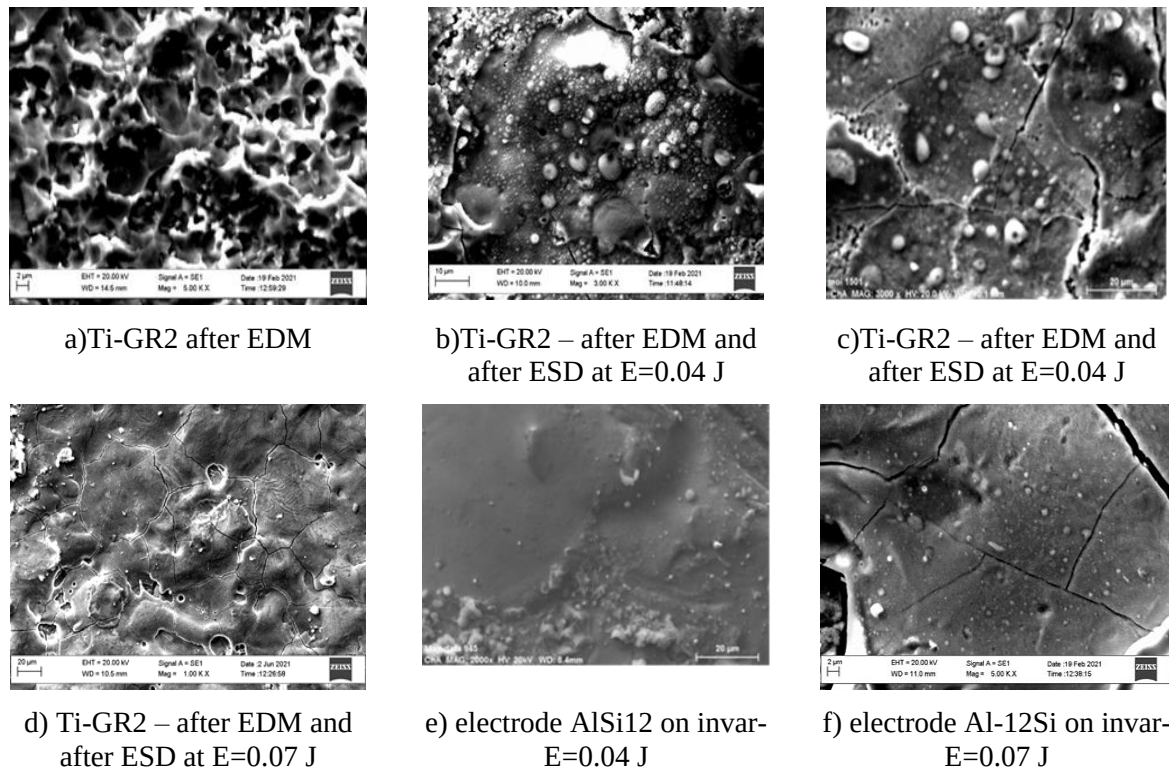


Fig. 7. General appearance of the SEM topography of coatings applied by vibration ESD on Ti6Al4V and invar alloys at different magnification

The comparison of the obtained surfaces with different impulse energy - Fig. 4, b and c, Fig. 5 a and c, Fig. 6 and 7 (the latter at a higher magnification), show that the surface of the coatings obtained at the maximum energy of the apparatus 0.07 J is rougher and with more irregularities and protrusions than those at an average energy of 0.04 J. The surfaces obtained on different substrates at the same energy are similar but show certain individual characteristics, which is determined by the physical nature of the ESD process. The coatings on martensitic steel and Ti-GR2- Figs. 1 and 6 are more homogeneous and with a more homogeneous and fine-grained structure than those obtained on invar, and the coatings obtained on titanium alloys – (Fig. 4) show lower roughness than those on iron-containing alloys. At higher energy, however, individual protrusions and irregularities appear, Fig. 4b, 5a, 6b. It is also noted that in the ESD of titanium samples with initial roughness $Ra = 4 - 5 \mu m$, the parameters Ra of the coating roughness at pulse energy up to 0.02 J are lower than those of the initial titanium surface (Table .3). As the impulse energy increases to 0.04 J, the roughness of the coatings also increases but remains lower than that of the initial roughness Ra .

Therefore, ESD at a pulse energy of up to 0.04 J can be used to smooth out irregularities and traces of previous treatment of the original titanium surface after additive technologies, or after rough and semi-pure mechanical or electroerosion treatment of titanium surfaces. At higher energy, however, separate protrusions and irregularities appear, Figs. 4b, 5a, 6b. From the data presented in Table 3 it is found that obtained in the contactless local electrospark layering - LESD coatings have significantly lower values of the roughness parameters, but the lower energy used and the technological features of this process do not allow complete filling of the cavities, pores and depressions at initial substrate roughness values $Ra > 5-6 \mu m$. The surface of the coatings applied at energy $E = 0.03-0.04 J$ is the most uniform, but even this

energy is not always sufficient for filling the pores and cavities - especially at the rougher initial surfaces of the substrate with roughness $Ra > 12-15 \mu\text{m}$. Fig. 8 shows the change of the average roughness and the coefficient of variation of the coatings on Ti6Al4V depending on the energy of the pulses in ESD with a vibrating electrode.

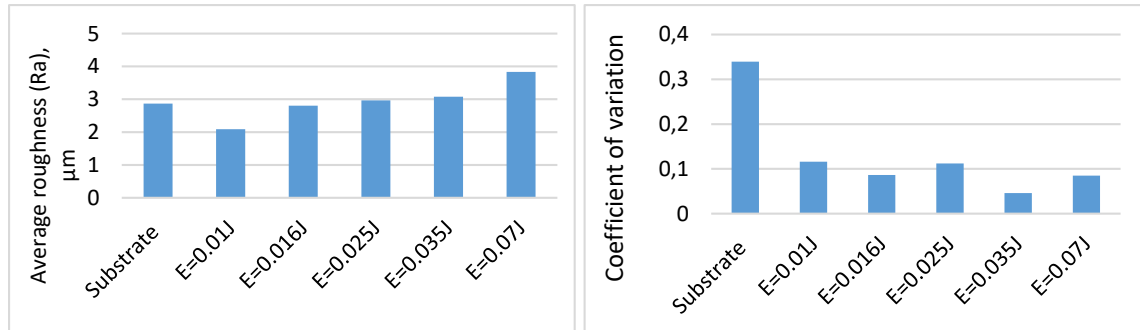


Fig. 8. Average roughness (Ra), and coefficient of variation /vs the impulse energy of AlSi12 coatings on Ti6Al4V at initial roughness $Ra = 4.32 \mu\text{m}$ obtained after EDM.

It can be seen that with the increase of the pulse energy the roughness of the coatings increases monotonically. In the coatings applied with pulse energy up to 0.03 J, the amount of microcracks and irregularities is less. At this energy, the highest homogeneity and smoothness are reported in the coatings of titanium alloys. The number of microcracks, irregularities and protrusions increase significantly with increasing pulse energy above 0.04 J. In the case of iron-nickel substrates and martensitic steel, the micropores appear at the highest pulse energy for the apparatus E0.07 J (Fig.7f).

In Fig. 9 ÷ 11 are given the profiles of the surface and of the functional parameters of roughness R_k and R_{ku} and the values of the other controlled parameters of the profile according to EN ISO 13565-2: 1996, ISO 25178 and Bulgarian State - BDS ISO 4287 standards.

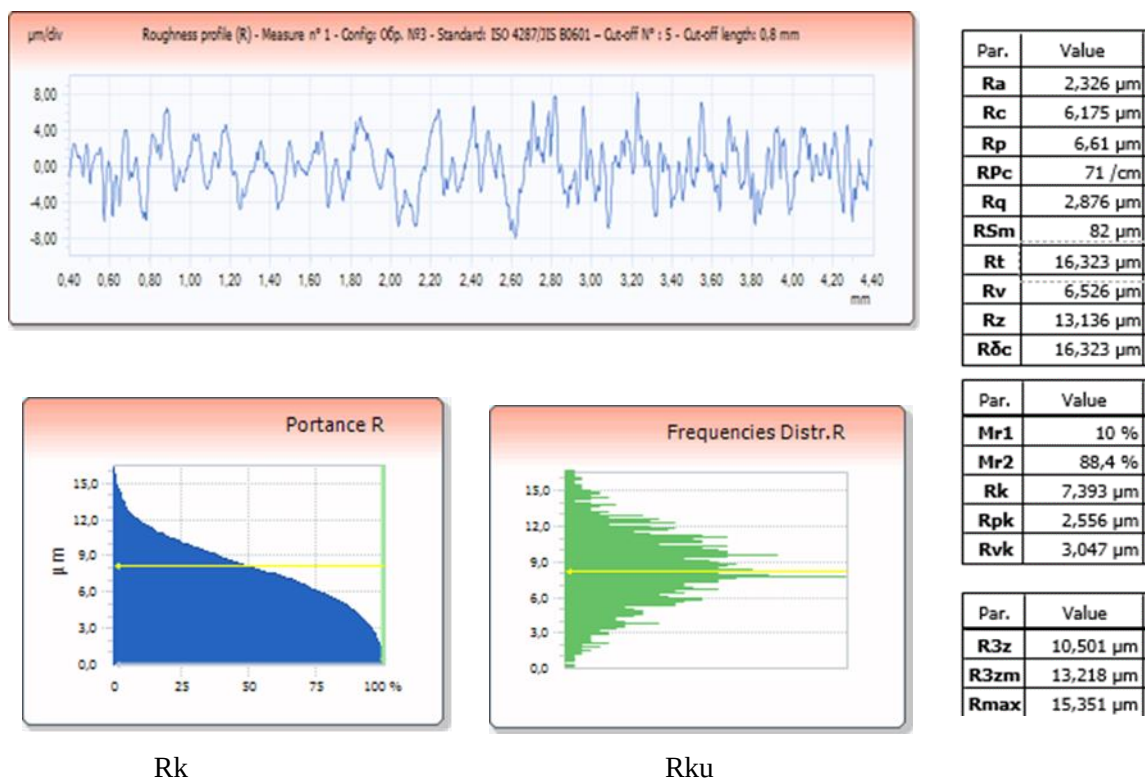


Fig. 9. Profilogram and Functional parameters Rk and Rku of AlSi12 electrode coating on Ti - Gr2 at pulse energy $E = 0.04$ J, two electrode passes - profile: left (0.4-2.2) - coating, right base (2.2-4.4).

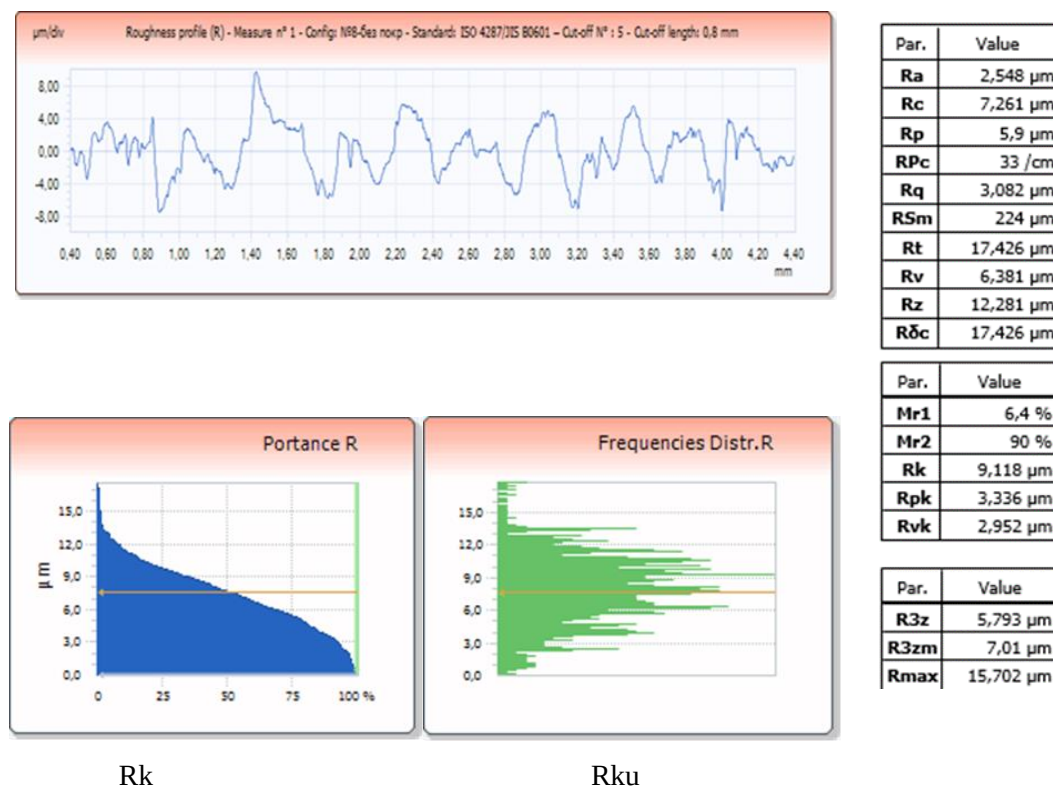


Fig. 10. Profilogram and Functional parameters Rk and Rku on substrate inconel 718- (after semi-pure turning)

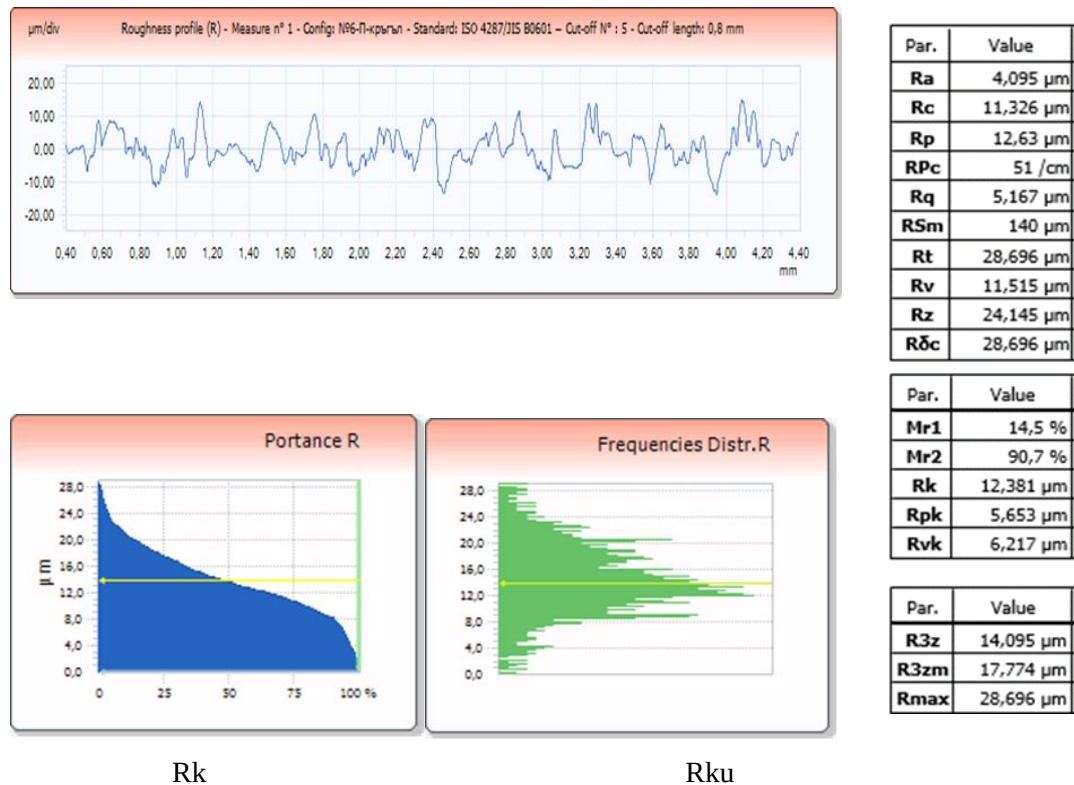


Fig. 11. Profilogram and Functional parameters Rk and Rku of coating from AlSi12 electrode at pulse energy $E=0.07$ J, two electrode passes on substrate inconel 718 (after semi-pure turning

From the figures and from obtained results it is established that the influence of ESD on the other studied roughness parameters is diverse:

- In ESD coatings, the asymmetry parameter Rsk decreases compared to that of the initial uncoated surfaces, and remains negative, which predetermines relatively good support properties of the layered surfaces;
- The parameter Rku, which determines the sharpness of the shape of the roughness amplitude distribution, also decreases, compared to the same parameter at the initial surface. For most of the coatings, the parameter Rku has values close to the optimal $Rku \approx 3$, which is favorable under conditions of friction and wear of the layered surface, because most of the protrusions and depressions are concentrated around the middle line of the profile of the irregularities;
- The parameter Rk, (according to the international standard EN ISO 13565-2: 1996), which determines the depth of the core of the roughness and the wear of the surface in the area of the coating increases compared to that of the base. Higher Rk values can adversely affect wear resistance and reduce service life and lubrication retention index. The sum of the roughness parameters $Rpk + Rk$, which characterizes the wear of the surface in the coating area, increases compared to that of the uncoated surfaces, which can characterize the coating surface as less wear resistant in terms of roughness parameters. The lowest degree of increase of this sum has been reported for the coatings onto substrates with initial roughness Ra 4-12 μm at a pulse energy of less than 0.03 J. With an increase in the energy to 0.07 J, the sum $Pk + Ppk$ increases also. At initial roughness $Ra < 3 \mu\text{m}$, the sum of these parameters after ESD is always higher than that of initial uncoated surfaces.

ESD with Al-Si electrodes manages to erase traces of the previous machining of the invar substrate. Despite the different initial roughness Ra of the substrate, the roughness of the coatings applied in the same mode is approximately the same. This shows that ESD can be successfully used to reduce the initial roughness when it is higher than a certain value - in this case at $Ra \geq 4.5 \mu\text{m}$. After coating a

surface with an initial roughness of $Ra \geq 4 \mu m$, all height and roughness step parameters decrease on average 1.15 to 1.5 times compared to the roughness parameters of the initial surface. Therefore, ESD can also be used to reduce of scattering and for leveling the relief of the original surfaces.

The microhardness of the coatings varies in a very wide range, but its average values for the studied coatings are similar. The values of the microhardness of the coatings presented in Table 4 are averaged over 10 measurements. Due to the large differences in the measured values (the lowest measured value is 7 GPa and the highest 12 GPa), they cannot be used to accurately compare the properties of the individual coatings. They can only be used as a guide for comparisons. The obtained surface hardness exceeds the normal value for the respective substrate 1.5 to 2.5 times due to the change in the phase composition and the fragmentation of the structure of the surface layer and exceeds the values of the corresponding initial hardness of the aluminum electrodes. The highest values of microhardness were measured in the coatings applied on martensitic steels (Table 4).

Table 4. Microhardness of coatings

№	substrate	HV, GPa	Coatng HV diapason, GPa	Coating HV average, GPa	Hardening coefficient
1	Ti-GR2	3,54	7.1 -10.2	8.2	2.3
2	Ti6Al4V	3.71	7.3 -11	8.5	2.3
3	1.2709 steel	4.2	7.5-12	9.6	2.3
4	Invar FeNi36	3,9	7.3-10	8.6	2.2
5	Inconel 718	3.95	7.3-10.5	9.1	2.3

The coatings have a compact and uniform microstructure - Fig. 9 - layer with a low degree of porosity, and a certain degree of mixing with the substrate is observed along the boundary. Microscopic unevenness is observed in different locations on the surface of the coatings in the cross-section. However, is seen a uniform, equal, dense layers, with about 12-15 μm thickness, snugly connected and firmly attached to the substrate with visible rare microcracks.

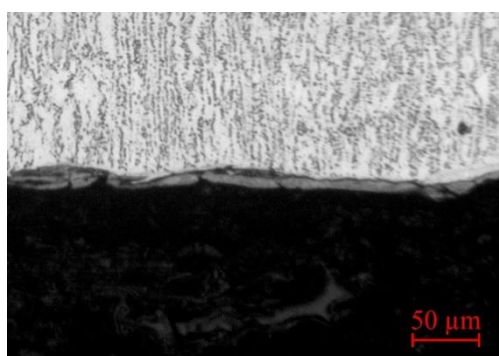


Fig. 12. Cross-section images of metallographic samples at ESD coating on invar

3.2. Phase Composition of Coatings

The results of X-ray diffraction analysis show that the resulting layer is a mixture of nonequilibrium heterogeneous fine-grained substrate structure saturated with modified ultrafine anode components, solid solutions, intermetallic compounds and metastable amorphous phases, part of which increases with increasing energy. In ESD with Al-Si12 electrodes, the main phases in the composition of the coating on iron-containing substrates are martensite α -Fe, austenite γ -Fe, Ni, Al. The characteristic diffraction peaks of the substrate are present for all coated samples. They are expanded and displaced, indicating

the presence of ultrafine to amorphous structures, solid solutions, crystalline defects, and internal stresses (Penyashki et al. 2016). In small quantities (with low peak intensity) and traces of Fe_3Al , FeAl , Fe_3Si , AlN , Si_3N_4 , Al_2O_3 , Fe_2O_3 , Fe_3O_4 are registered and monitored. The recorded traces of Al_2O_3 , as well as the traces of AlN and SiN , show that during the transfer process aluminum and silicon react with oxygen and nitrogen from the air, forming highly wear-resistant compounds. The main phases registered in the coatings on titanium alloys are, $\alpha\text{-Ti}$, Ti_5Si_3 , Al_3Ti , AlSi_3Ti_2 , $\text{TiN}_{0.26}$, Al , Al_2O_3 .

Figure 13 shows the radiographs of the coatings applied on Ti - GR2, Invar and 1.2709 steel, and Table 5 shows the phases registered in the coatings.

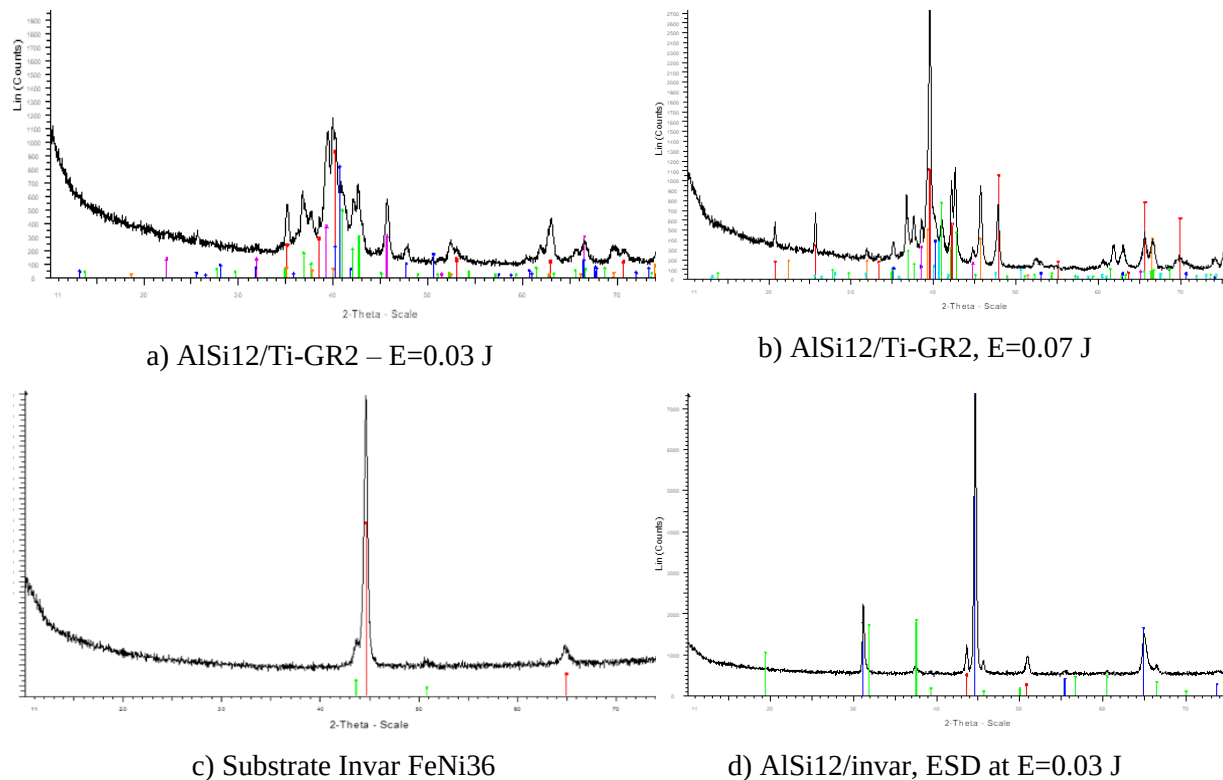


Fig. 13. Some exemplary radiographs of a substrate and coatings applied on Ti - GR2, and Invar

Table 5. Phase composition of coatings

№	Electrode	Substrate	Modes ESD, J / number electrode passes	Registered phases
1		Ti - GR2	--	$\alpha\text{-Ti}$, $\text{TiN}_{0.3}$, Ti_5Si_4 - traces
2	Al-12%Si	Ti - GR2	ESD; E=0.04J / 2	$\alpha\text{-Ti}$, TiAl_3 , TiAl , TiO_2 , Ti_5Si_3 , $\text{TiN}_{0.3}$, Al_2O_3
3	Al-12%Si	Ti - GR2	ESD; E=0.07J / 2	$\alpha\text{-Ti}$, Al , TiAl_3 , TiAl , TiO_2 , Ti_5Si_3 , Al_3Ti , AlSi_3Ti_2 , Al_2O_3 , Ti_2O , $\text{Al}_{2.5}\text{Ti}_{1.5}$, $\text{TiN}_{0.3}$
4	substrate	Invar (36%Ni+Fe)	--	Fe-Ni , (Fe,Ni) , $\text{Fe}_{0.7}\text{Ni}_{0.3}$
5	Al-12%Si	Invar (36%Ni+Fe)	ESD; E=0.04J / 4	(Fe,Ni) , NiAl_3 , Al_2O_3 , AlFe , $\text{Al}_{0.9}\text{Ni}_{1.1}$, $\text{AlFe}_{0.23}\text{Ni}_{0.77}$, $\text{Fe}_{0.7}\text{Ni}_{0.3}$
6	Al-12%Si	Invar (36%Ni+Fe)	ESD; E=0.07J / 4	(Fe,Ni) , Fe-Ni , $\text{Al}_{0.9}\text{Ni}_{1.1}$, $\text{Al}_{0.9}\text{Ni}_{1.1}$, $\text{AlFe}_{0.23}\text{Ni}_{0.77}$, Al_2O_3 , AlNi
7	Substrate	19%Ni,11%Co+Fe1.2709 steel	-	(Fe,Ni) , Fe-Ni , Co ,
8	Al-12%Si	19%Ni,11%Co+Fe	ESD; E=0.04J / 3	(Fe,Ni) , NiAl_3 , Al_2O_3 , AlFe , AlNi

9	Al-12%Si	19%Ni,11%Co+Fe	ESD; E=0.07J / 3	(Fe,Ni), NiAl ₃ , AlFe, AlFe _{0.23} Ni _{0.77} , Al ₂ O ₃
10	Al-12%Si	19%Ni,11%Co+Fe	LESD; I=24,4A, Ti=20μs, C=4.4μF / 4	NiAl ₃ , AlFe, Al ₂ O ₃ , AlNi, FeSi, Al _{2.67} O ₄ , Al ₅ Fe ₂ , Fe _{0.9} Si _{0.05}
11	Al-12%Si	19%Ni,11%Co+Fe	LESD; I=24,4A, Ti=12μs, C=4.4μF / 4	(Fe,Ni), AlFe _{0.23} Ni _{0.77} , Al ₂ O ₃ , FeSi, AlNi, AlFe, Al ₅ Fe ₂ , Fe _{0.9} Si _{0.05}

The summary of the results obtained from X-ray examinations shows that in the process of ESD with AlSi electrodes as a result of the reactions between the anode and cathode materials and nitrogen and oxygen from the environment in the coatings many new intermetallic phases are formed such as Ti₅Si₄, TiAl₃, AlSi₃Ti₂, Fe_{0.7} Ni_{0.3}, AlFe_{0.23}Ni_{0.77}, NiAl₃, AlFe, FeSi, etc., the presence of which implies higher microhardness, a stronger bond with the base and correspondingly increased wear resistance. As a result of the reactions of the melt with the elements of the ambient air, highly wear-resistant phases such as TiN and Al₂O₃ are obtained, which also imply high hardness and wear resistance. As from the extremely high heating and cooling rates, the structure of the surface layer grind to nano-dimensions, and coatings with the ultrafine structure are formed to an amorphous structure, which is also favorable for the mechanical properties and wear resistance of the modified surfaces. As is known, amorphous metal alloys based on Al have excellent mechanical properties, high strength and good ductility, which is also favorable for the mechanical properties and wear resistance of the coatings.

3.3. Coatings tribo tests results - Wear of coatings

The influence of the microgeometric parameters, the composition and the structure of the coatings on their tribological properties and wear resistance were studied by comparative tests of friction and wear. Table 6 shows the wear depending on the friction path of coated specimens and of invar substrate specimens. Table 7 shows the development of wear of samples from 1.2709 steel with coatings of AlSi12 electrode.

Table 6. Mass wear of AlSi coatings on invar alloy

Number of cycles		30	60	90	120
Friction path, m		6.9	13.8	20.7	27.6
Electrode	ESD-Pulse energy, J / number of electrode passes	Mass wear, mg			
AlSi12	E=0.04 / 6	1.0	2.0	3.1	4.1
AlSi12	E=0.07 / 6	2.2	2.5	3.1	3.9
AlSi12	E=0.04 / 3	1.6	2.1	2.8	3.7
AlSi12	E=0.07 / 3	1.0	2.2	2.6	3.0
substrate	-	1.9	2.9	4.5	5.6

Table 7. Mass wear at different friction paths of AlSi coatings on 1.2709 steel substrate

Number of cycles		30	60	90	120
Friction path, m		6.9	13.8	20.7	27.6
Electrode	ESD-Pulse energy, J / number of electrode passes	Mass wear, mg			
AlSi12	E=0.07 / 3	0.8	1.3	1.8	2.2
AlSi12	E=0.04 / 3	0.6	0.8	1.0	1.2
substrate	-	1.1	1.5	1.9	2.4

Friction tests showed that the wear of the layered surfaces was 1.1 to 2 times lower than those without coating. The results of the comparative studies of the influence of the ESD regimes on the wear of the coatings show that the mass wear of the coated samples of invar alloy is 1.4-1.9 times lower than that of the uncoated ones. The change in the wear rate, the wear intensity, the wear resistance and relative wear resistance of the coatings is similar. The lowest wear was shown by the samples layered at energy $E = 0.04$ J. with three passes of the layering electrode. Obviously, the higher values of the roughness parameters of the coatings applied at energy 0.07J adversely affect wear.

The coatings obtained on 1.2709 steel substrate reduced the wear up to 2 times than uncoated alloys.

In both substrate materials, lower wear is shown by the samples coated at $E=0.04$ J.

Figure 14 shows micrographs of worn uncoated and coated with the test electrodes surfaces of invar alloys. The micrographs show that on the uncoated surface -Fig.14a) there are traces (scratches) of abrasive and relatively deep craters from adhesive wear. The debris is in the form of chipped or ripped particles with a rounded shape.

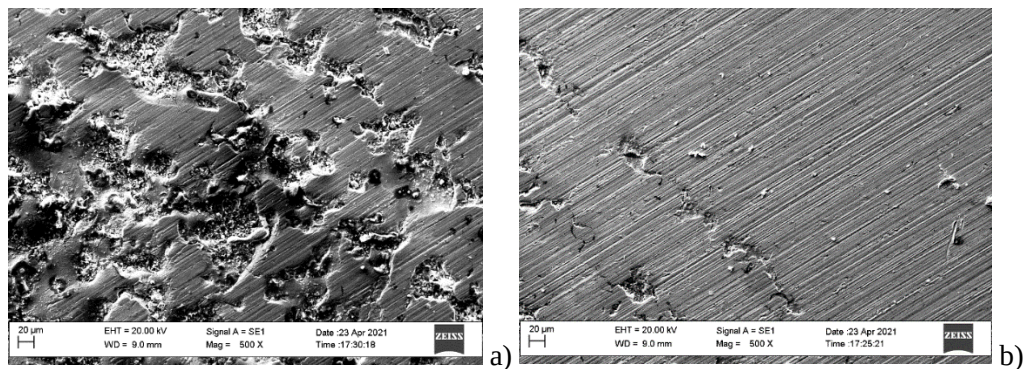


Fig. 14. Micrographs of uncoated invar (a), and of coated with pulse energy $E=0.03$ J (b) worn surfaces at a friction path 28 m

Superalloys based on nickel and titanium alloys are suitable for aggressive environments but show poor abrasion resistance, low surface hardness and high friction coefficient. ESD coatings from Al-Si electrodes on these alloys reduced the wear, slow down its development in time, and can be used both to erase the traces of pre-treatment of the original surface and to increase the durability of titanium and Fe-Ni surfaces, subjected to abrasion. Due to the lower roughness, better uniformity, finer structure, lower thermal impact on the substrate the ESD with lower pulse energy shows higher wear resistance and is suitable for the layering of parts with high demands on surface accuracy and quality.

Based on the results obtained in this work, it can be concluded that the reaction electrospark surface modification with AlSi electrodes is one of the promising, light, economical and affordable ways to simultaneously fill surface pores and cracks, to reduce roughness, increase the surface hardness, and wear resistance of products obtained by SLM.

4. CONCLUSIONS

The use of ESD with low pulse energy (up to 0.07 J) with AlSi12 electrode on the surface of titanium and iron-nickel alloys with different initial roughness, dense and uniform coatings with roughness and thickness is obtained, which can be changed by processing modes in the limits roughness 1.6-6 μm and thickness 6-16 μm .

The obtained results from the topography, morphology, composition, structure and friction wear of the coatings show an opportunity for the determination of modes for the ESD to obtain coatings with preset roughness, thickness, hardness and wear resistance on titanium and nickel surfaces.

It has been found that the quality of surfaces obtained after ATM-SLM process and after rough and semi-pure machining can be improved in the process of reactive electrospark surface treatment with electrodes of low-melting alloys Al-Si12 as a result of plasma combustion of protrusions and the filling of the depressions and scratches with the low-melting eutectic melt, which is formed in the process of spark discharges. The use of electrodes of low-melting alloys Al-Si12 as a result of plasma discharges makes it easier to melt and penetrate the melt in the substrate. The parameters of the process and the conditions for ESD, suitable for the realization of the above phenomena, are determined.

Friction tests show that as a result of the lower roughness, the better homogeneity, finer structure, lower thermal impact on the substrate and of the new wear-resistant phases formed in the ESD process, the wear of layered surfaces is up to 2 times lower than that of the uncoated. ESD with lower impulse energy (up to 0.04J) shows higher wear resistance and is suitable for layering parts with simultaneously high requirements both for surface accuracy and quality and for wear resistance. The conditions for ESD, under which the simultaneous erasure of the traces from the previous treatment and improvement of the surface characteristics are obtained and which presuppose the maximum possible increase of the wear resistance of the coated surfaces are determined. Among the main benefits of the implementation of reaction ESD with AlSi12 electrodes is the significant improvement of the surface quality and their durability obtained at the same time.

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