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Effect of Speed and Impact Angle on Solid Particle Erosion of Vanadium Carbide Coatings Produced by Thermo-Reactive Diffusion Technique

This paper presents research of the solid particle erosion of conventionally and duplex treated vanadium carbide (VC) coatings produced on the C45E steel substrate by the thermo-reactive diffusion (TRD) technique. The Response Surface Methodology (RSM) based on Central Composite Design (CCD) was used to evaluate the effect of moving speed and impact angle as input variables on the erosive wear mass loss as the response. It was established that moving speed of the samples and the impact angle of the abrasive particles are statistically significant factors, while their interaction does not have significant effect on the wear process. The mathematical model of the investigated wear process was derived as well.

Keywords: solid particle erosion, vanadium carbide (VC), thermo-reactive diffusion (TRD), duplex treatment, response surface method (RSM)

1. INTRODUCTION

Solid particle erosion at low speeds is common by working parts of industrial machinery and equipment whose surfaces are in direct contact with the hard particles (abrasives) and moving relative to them during operation (agricultural, mining equipment, tunneling and construction equipment, etc.). In studies of such wear form various laboratory devices (tribometers) are used, which are developed to investigate the wear of tools for tillage [1, 2], tunneling [3-5] or mining [6, 7]. Usual abrasives for these devices are different soil types or mixtures of soil and stone in dry or wet condition. The wear protection of surfaces exposed to erosion is mainly carried out by hard coatings produced by various coating methods like thermal spraying [8-10], laser cladding [8, 11, 12] or thermo-reactive diffusion (TRD) processes [8, 13, 14].

Transition metal nitrides and carbides, such as vanadium carbide (VC), are commonly used as tribological coatings because they have excellent wear resistance, very high hardness, low friction coefficient, good corrosion resistance and very high melting point [15]. Vanadizing is a thermal-chemical process of diffusion of vanadium (V) in the surface of the treated steel, which, by combining vanadium and carbon from the steel substrate, creates a VC coating. Vanadizing can be performed by chemical vapor deposition (CVD), physical vapor deposition (PVD) and thermo-reactive diffusion (TRD) process. TRD is a low cost process which requires relatively simple equipment, and it is environmentally friendly [15]. TRD process involves the immersion of material in a fused salt bath of special composition that contains the carbide-forming element

vanadium but might also contain some other elements, e.g. Cr, Ti, Ta and Nb. The bath temperature is ranging from 900 °C to 1100 °C and the immersion time is 1 h to 10 h. The different microstructures of a VC coating may be induced by the significant composition difference among substrates with a carbon content of 0.3 wt% or higher [16].

During the vanadizing process a VC coating is formed on the surface of the substrate wherein the material below the coating partially loses carbon content (decarburization) due to its diffusion from austenitic interior towards surface and reaction with vanadium. Therefore, immediately under the VC coating a carbide concentration is lower than in the substrate core. Since the quenching comes after vanadizing, lower carbon concentration under carbide coating leads to softer structure in this area. Such plunge in hardness of the substrate can be dangerous for the thin surface coating because it does not provide sufficiently hard base for harder operational conditions. Carburizing, i.e. carbon diffusion into the surface of the substrate before vanadizing, results in higher carbon content which enables achieving greater thickness of the vanadium coating as well as reducing the carbon depletion in the area below the coating. Such duplex treatment (previously carburizing and vanadizing) results with harder structure after subsequent quenching as well as the possibility of application the vanadizing for steels with lower carbon content [16, 17].

Since VC coatings have excellent wear resistance, they were investigated with regard to different wear types. Most studies have analyzed their abrasion resistance and the erosive wear has been less frequently investigated. According to earlier studies of VC coatings obtained in salt baths, such coatings have very good resistance to erosive wear [18]. Some studies of erosion resistance of metal matrix composites (MMC) with stainless steel matrix and reinforcing metal carbides as protective coatings have found a higher resistance of coatings containing vanadium carbides (VC) than coatings with wolfram (WC) and titan

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carbides (TiC), where the erosion resistance increased with the increasing of VC content [19]. However, as is pointed out, there is a lack of comparable published data about erosion resistance for vanadized steels, as well as for carbonitrided and borided steels [14].

Numerous studies have established that moving speed of samples through the abrasive mass significant influence on their wear. Most of the studies found that an increase in speed causes increased wear rate [20-23]. This wear increasing mostly is not linear [21, 23]. Also, that effect of speed on erosive wear increasing depends on material hardness, i.e. this increasing is greater for soft materials than for hard materials [21, 22].

In many studies was established that effect of the impact angle on wear of material is dependent on the hardness of the material. With ductile materials the wear is greater at smaller impact angle. For some low carbon steels maximum wear rate occurs at impact angles under 22,5°, and with further increasing of impact angle the wear rate decrease [24, 25]. According to [26], with the same ductile carbon steel in non-heat treated state the maximum wear rate occurs at impact angle of 30° and in the quenched state at 45°. Wear tests on Ni-based coatings [23] and hard carbide layers [27, 28] shown that maximum wear occur at impact angles over 45°.

The aim of this paper is to contribute to the research of solid particle erosion of VC coatings caused by motion through the mass of free solid particles and to determine the effects of the moving speed and the impact angle.

2. MATERIALS AND METHODS

2.1 Samples for experiment

The samples for experiment were made in the form of tiles with dimensions 40 mm × 20 mm × 5 mm. The basic material (substrate) is medium carbon steel C45E (EN) with maximum Brinell hardness of 207.

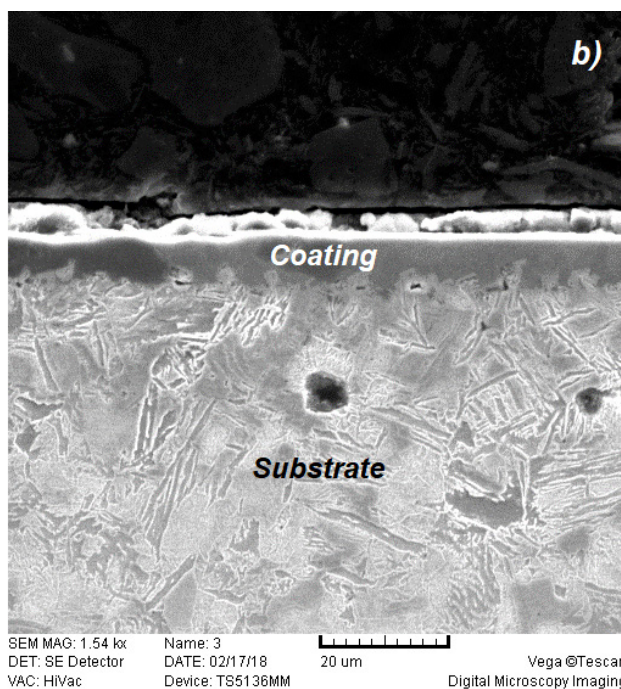
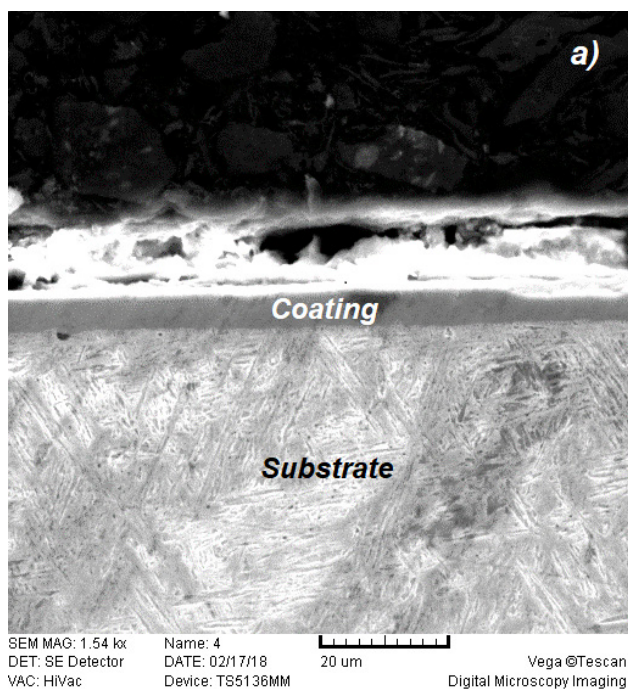


Figure 1. Microstructure of conventional vanadized (a) and duplex treated (b) samples

The chemical composition of the substrate was determined by optical emission spectrometry (OES). Microanalysis of the coatings was performed by scanning electron microscope (SEM) Tescan Vega TS5136LS with energy dispersive X-ray spectrometry (EDS). Surface hardness of coatings and cross-section hardness of the substrate was measured by Vickers microhardness tester Shimadzu HMW-2T.

Table 1. shows the substrate chemical composition.

Table 1. Substrate chemical composition

Element	wt, %	Element	wt, %
C	0.46	Cr	0.024
Si	0.29	Mo	0.08
Mn	0.68	Ni	0.020
P	0.026	Fe	balanced
S	0.029	-	-

Processes for coating substrate were performed as:

- conventional TRD vanadizing process,
- duplex treated TRD vanadizing process.

The conventional TRD vanadizing process was performed by immersing samples into the salt bath with added vanadium at temperature of 950 °C. Samples were kept in the salt bath for 4 h, after that immersed in rinsing agent and then quenched in water and rinsed with running water.

The duplex treated TRD process was performed as vanadizing with previously carburizing. The carburizing process of the samples was carried out in annealing box with carburizing granulate at a temperature of 950 °C and a time of 3 h. The samples were then transferred to a salt bath for the formation of the carbide layer. The process in the salt bath was identical to the conventional process. Figure 1 shows the microstructures of both VC coatings and Figure 2 the EDS point analysis to determine their chemical composition.

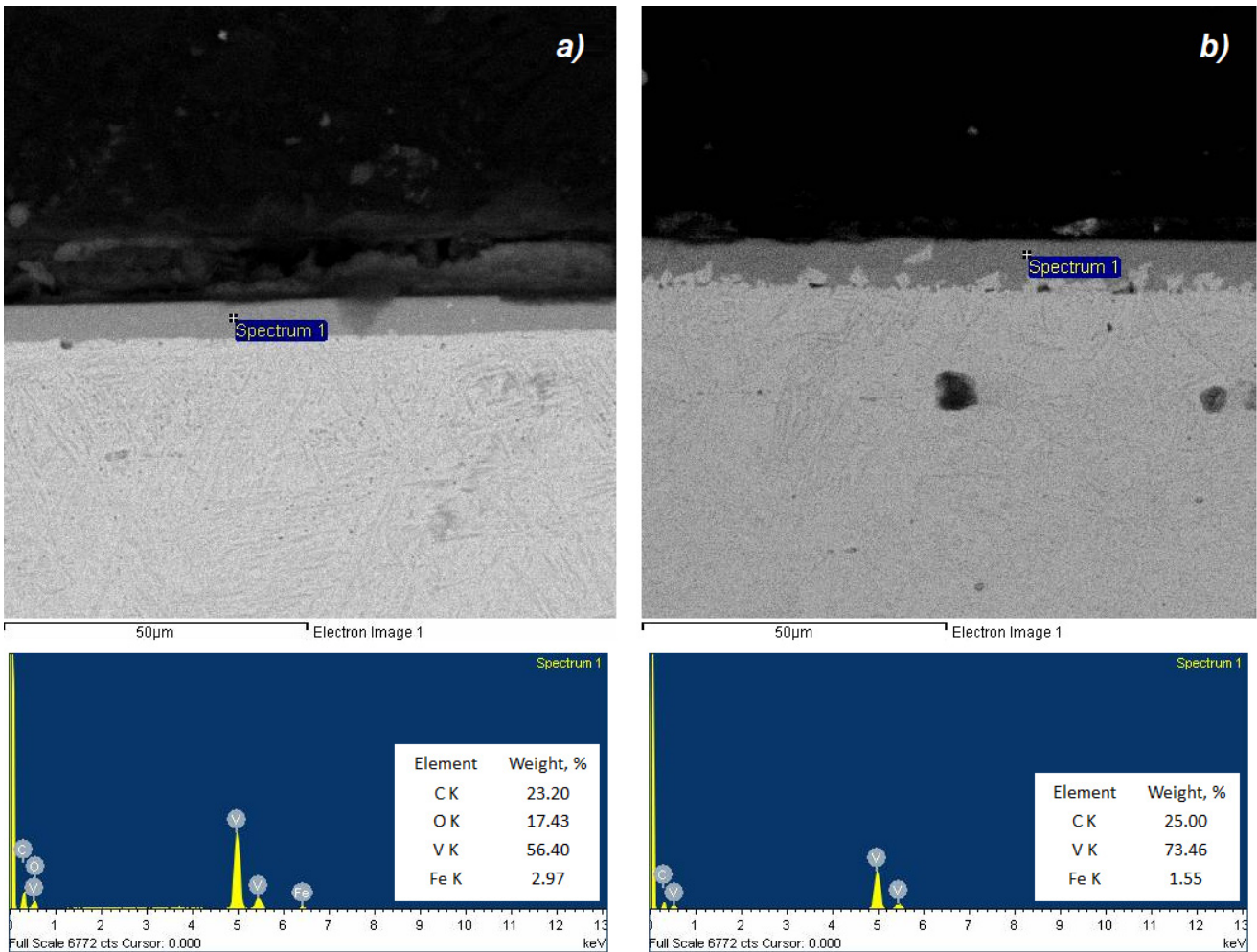


Figure 2. EDS point analysis of conventional vanadized (a) and duplex treated (b) samples

In the Figure 1 the uniform thickness of VC coatings with clear boundaries between coatings and the substrate can be observed.

The thickness of the obtained conventional VC coating was about 5 μm , with the mean surface hardness of about 2100 $\text{HV}_{0.03}$. The thickness of obtained duplex VC coating was about 7.5 μm with the mean surface hardness of about 2150 $\text{HV}_{0.03}$.

The substrate hardness under the VC coatings was measured and compared in depth by Vickers $\text{HV}_{0.05}$ test. The results of measuring are shown in Figure 3.

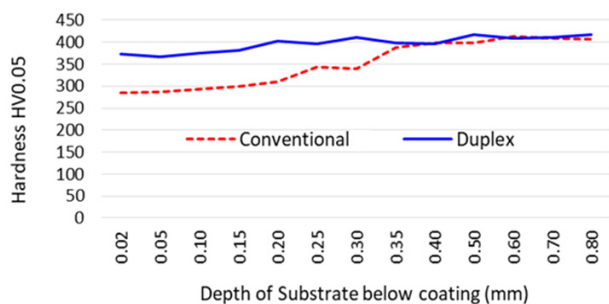


Figure 3. Substrate hardness below VC coatings

Just below the conventional VC coating, there is a drop of the hardness relative to the deeper quenched part of the substrate due to decarburization during the vanadizing process. This drop of the hardness in the substrate of VC coating with previously carburization is substantially smaller due to absence of decarburization.

2.2 Wear experiment

The experiment includes conducting of wear tests on adequate testing device (wear tester) with VC coating samples which moves in mass of free solid particles.

The input variables of the wear test are the moving speed of the sample (v) ranging 1.0 m/s to 3.0 m/s and the impact angle of the abrasive particles (α) relative to the worn surface of samples ranging 0° to 90° . The wear path was 50 000 m long. The results (responses) in the wear test are the amounts of the wear of the coatings, expressed as mass loss of the samples (Δm).

In the design of experiment, Central Composite Design (CCD) was selected to provide a qualitative analysis of the individual and interaction effects of the input variables [29]. According to the selected design of experiment, for each wear test of coating minimum and maximum levels of input variables were defined as well as 4 replications in the center point of the experiment. Also, for the purpose of qualitative statistical analysis of the results, tests were performed in 3 repetitions for each coating in each state of experiment and mean values of results (response) were processed.

The wear tests were conducted on a specially designed wear tester. The wear tester is in the form of a container (pot), 1.0 m in diameter and 0.4 m deep, with about 250-300 kg abrasives. This device enables:

- wear test with different abrasives,
- testing of different tribological coatings,

- moving speed of sample in the range 0.5 m/s to 3.5 m/s,
 - impact angle in the range 0° to 90°,
 - repetition of the state of the experiment.
- Figure 4 schematic shows the wear tester [17].

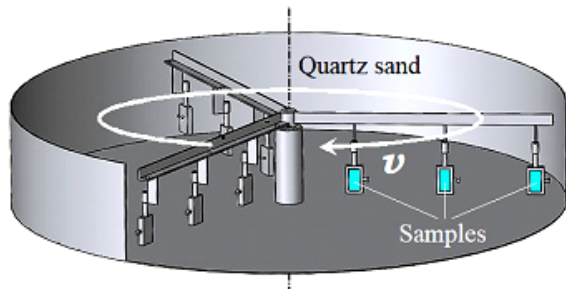


Figure 4. Schematic presentation of the wear tester

During the wear test, only one surface of the sample was exposed to the wearing by abrasive particles, because the samples were placed in holders which prevent wearing of the other sample surfaces.

Rounded quartz sand Ottawa AFS 50/70, with grain size between 212 μm and 300 μm , was used as an abrasive in the wear test.

The sample carrier rotates at 66 min^{-1} . The moving speeds of samples (v) are calculated as the peripheral speeds on the corresponding peripheral circles of the calculated diameters and they are determined by setting on a calculated diameter (or radius).

The impact angle of abrasive particles relative to the worn surface (α) was achieved by placing the sample at selected angle relative to tangent line of the peripheral

circle of a diameter belonging to the selected peripheral speed of the samples (v), as shown in Figure 5.

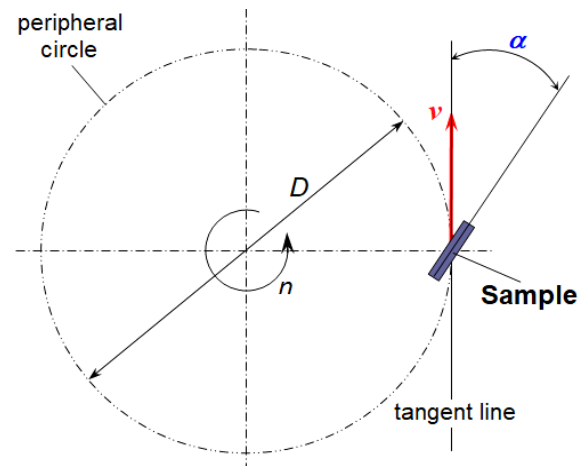


Figure 5. Scheme of placing the samples at selected angle

Mass losses of the samples during the wear test are calculated as the differences of measured sample masses before and after the test by analytical scale Mettler B5C 1000 with precision of 10^{-4} g. Before each measuring, the samples were thoroughly washed under water jet, cleaned from fine particles and dried with warm air.

3. RESULTS AND DISCUSSION

The analysis of the experimental results was carried out by using the software Design-Expert. Measured sample mass losses (response) of both coatings, according to the CCD experiment, are given in Table 2.

Table 2. Wear test results of conventional and duplex VC coatings

Run.	Factor A: Speed of samples (m/s)	Factor B: Impact angle (degree)	Response: Mass loss (10^{-4} g)	
			Conventional VC	Duplex VC
1	1.00	90.00	23.00	23.00
2	3.00	0.00	28.00	23.00
3	1.00	0.00	11.00	17.00
4	3.00	90.00	63.00	43.00
5	2.00	45.00	36.00	28.00
6	3.41	45.00	77.00	60.00
7	2.00	45.00	33.00	26.00
8	2.00	108.64	18.00	12.00
9	2.00	45.00	32.00	30.00
10	2.00	-18.64	5.00	3.00
11	2.00	45.00	37.00	33.00
12	0.59	45.00	25.00	27.00

Table 3. ANOVA for wear test of conventional VC coatings

Source	Sum of Squares	Degrees of Freedom	Mean Square	F-Value	P-Value	
Model	4385.12	5	877.02	34.27	0.0002	significant
A – speed	2130.06	1	2130.06	83.23	< 0.0001	
B – impact angle	534.40	1	534.40	20.88	0.0038	
A ²	435.60	1	435.60	17.02	0.0062	
B ²	846.40	1	846.40	33.07	0.0012	
AB	132.25	1	132.25	5.17	0.0634	
Residual	153.55	6	25.59			
Lack of Fit	136.55	3	45.52	8.03	0.0604	not significant
Pure Error	17.00	3	5.67			
Cor Total	4538.67	11				

The experimental results of both VC coatings were processed with Analysis of variance (ANOVA), which determines the accuracy of the proposed wear model and influence of the observed factors in the wear process based on their *P*-values. *P*-values less than 0.05 indicate factors with a significant effect.

Table 3 shows ANOVA for the wear test of conventional VC coatings. The accuracy of proposed second-order response surface model (equation 1) is confirmed with *P*-value of 0.0002, which means that this model can describe the actual wear process. In the observed case, the speed of the samples (*A*, *A*²) and the impact angle (*B*, *B*²) terms affect the wear significantly, while the interaction of variables (*AB*) is not significant. The model fit was evaluated by coefficient of determination *R*² = 0.9662, shown in Table 4, which confirms high level of compliance of measured data from the wear experiment and the predicted data from the developed model.

Table 4. Model statistics for conventional VC coating

Std. Dev.	5.06	R-Sq.	0.9662
Mean	32.33	Adj R-Sq.	0.9380
C.V.	15.65	Pred R-Sq.	0.7794
PRESS	1001.22	Adeq Prec.	20.7250

The mathematical model for the wear process of conventional VC coatings with actual variables was derived as the regression equation (1):

$$\Delta m = 20.69213 - 22.43261 \cdot v + 0.43718 \cdot \alpha + 8.25000 \cdot v^2 - 0.00568 \cdot \alpha^2 - 0.12778 \cdot v \cdot \alpha \quad (1)$$

The second-order response surface model was also proposed for duplex VC coatings and ANOVA for the wear test is shown in Table 5. The *P*-value of 0.0006 confirm the accuracy of the proposed model in description the actual wear process.

Table 5. ANOVA for wear test of duplex VC coatings

Source	Sum of Squares	Degrees of Freedom	Mean Square	<i>F</i> -Value	<i>P</i> -Value	
Model	2220.75	5	444.15	25.58	0.0006	significant
A – speed	660.10	1	660.10	38.02	0.0008	
B – impact angle	187.48	1	187.48	10.80	0.0167	
A ²	348.10	1	348.10	20.05	0.0042	
B ²	722.50	1	722.50	41.61	0.0007	
AB	49.00	1	49.00	2.82	0.1440	
Residual	104.17	6	17.36			
Lack of Fit	77.42	3	25.81	2.89	0.2030	not significant
Pure Error	26.75	3	8.92			
Cor Total	2324.92	11				

Table 6. Model statistics for duplex VC coatings

Std. Dev.	4.17	R-Sq.	0.9552
Mean	27.08	Adj R-Sq.	0.9179
C.V.	15.38	Pred R-Sq.	0.7427
PRESS	598.10	Adeq Prec.	18.9020

ANOVA shows that the speed of the sample (*A*, *A*²) and the impact angle of the abrasive particle terms (*B*, *B*²) are significant. The interaction of the variables (*AB*) does not significantly affect the wear process. The coefficient of determination *R*² = 0.9552, shown in Table 6, confirms high level of compliance of measured and predicted wear data. The mathematical model for the wear process of duplex VC coatings with actual variables was derived as the regression equation (2):

$$\Delta m = 32.11675 - 23.91637 \cdot v + 0.42424 \cdot \alpha + 7.37500 \cdot v^2 - 0.00525 \cdot \alpha^2 - 0.07778 \cdot v \cdot \alpha \quad (2)$$

The derived mathematical models of wear experiments are graphically presented as response surface plots, showing the effects of variables (moving speed and impact angle) on wear result (mass loss) for both VC coatings, as shown in Figure 6.

From the surface plots in Figure 6, an exponential increase in wear rate when increasing the moving speed from 1 to 3 m/s can be seen. The similar was shown in studies [21, 23]. The increase in wear rate at increasing the moving speed can be attributed to the fact that the

kinetic (impact) energy of the particles has a key role in wear mechanisms with abrasive particles, as similar indicated in the study [22]. The energy is the product of the speed of particles and their mass, wherein the influence of speed is in exponential dependence.

The derived wear models of coatings are defined as second order polynomials with a high coefficient of determination, also consistent with previous claim.

By comparing the response surfaces plots, it can see that the increase of wear rate by increasing of speed is slightly higher with conventional VC coating than with duplex VC coating. This can be attributed to the slightly higher surface hardness of the duplex coating, which is consistent with research [21, 22].

The effect of the impact angle on wear of coatings is also significant. Since the maximum wear rates in the experiment were generated at speed of 3 m/s, the effect of impact angle was also analyzed at the same speed. The response surface plot for a conventional VC coating shows that the highest wear rate occurred at an impact angle of about 71°. With the duplex VC coating, the highest wear rate occurred at an angle of 67°. Such results are consistent with data from studies [23, 27, 28], which indicated that for hard coatings the highest wear occurs at impact angles above 45°.

The tendency of the ascent of wear curve to change of impact angle for both coatings is very similar, which is expected given the small difference in the surface hardness of the coatings.

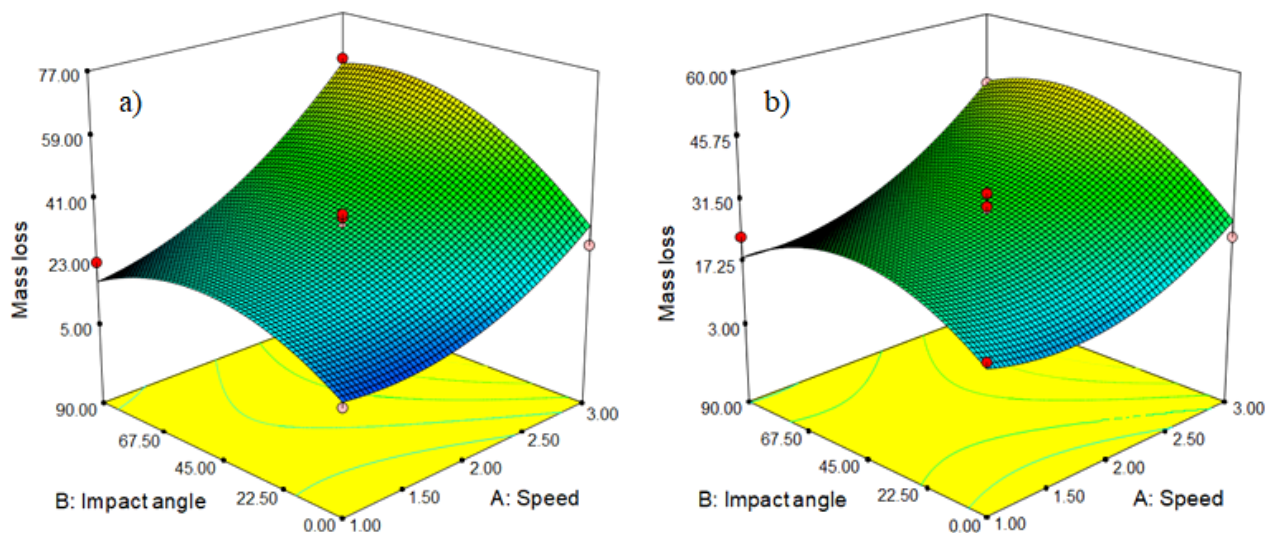


Figure 6. Response surface plots of mass loss for VC coatings – conventional (a) and duplex (b)

4. CONCLUSION

The wear experiment conducted in this research has shown that the tested VC coatings have high resistance to the solid particle erosion, primarily due to their high hardness.

Vanadizing with previous carburizing (duplex treatment) resulted in a harder VC coating with a greater thickness than the conventional obtained VC coatings.

Measuring of substrate microhardness of the conventional vanadized samples indicated significantly hardness reducing (up to 30 %) directly below the coating with respect to the hardness of deeper substrate, while with the previous carburizing such a phenomenon was significantly less expressed (up to 10 %).

Statistical analysis of the wear experiment results of both vanadized coatings established that the speed of the samples (v) and the impact angle of abrasive particles relative to the coating surface (α) have a significant individual effect on the observed wear process, while their interaction is not significant.

A mathematical response surface models in form of second-order polynomials have been derived for both vanadized coatings. The applicability of the models in predicting the actual wear processes of vanadized samples has been confirmed by ANOVA tests with high levels of significance and high coefficients of determination R^2 of the regression models. That confirms high compliance of measured data from the wear experiments and predicted data from the derived wear models.

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**УТИЦАЈ БРЗИНЕ И УДАРНОГ УГЛА НА
ЕРОЗИЈУ КРУТИМ ЧЕСТИЦАМА ВАНАДИЈУМ-КАРБИДНИХ ПРЕВЛАКА ИЗРАЂЕНИХ
ТЕХНИКОМ ТЕРМО-РЕАКТИВНЕ ДИФУЗИЈЕ**

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У раду је приказано истраживање ерозије крутим честицама конвенционалних и двоструко третираних (дуплекс) ванадијумско-карбидних (VC) превлака, израђених на подлози челика С45Е техником термо-реактивне дифузије (ТРД). Метода одзивних површина (PCM), утемељена на централно композитном плану експеримента (CCD), коришћена је за процену утицаја брзине кретања ударног угла, као улазних варијабли, на губитак масе ерозијским трошењем као одзивом. Утврђено је да су брзина кретања узорака и ударни угао абразијских честица статистички значајни фактори, док њихова интеракција нема значајан утицај на процес трошења. Изведен је и математички модел испитиваног процеса трошења.