

N. RADEK*, J. KONSTANTY**

CERMET ESD COATINGS MODIFIED BY LASER TREATMENT

POWŁOKI CERMETALOWE NANIESIONE ELEKTROISKROWO I MODYFIKOWANE OBRÓBKĄ LASEROWĄ

The main objective of the present work was to determine the influence of laser treatment on microstructure, microhardness, roughness, bonding strength, corrosion resistance and tribological properties of wear resistant coatings produced on C45 carbon steel by the electro-spark deposition (ESD) process. Consumable WC-Co-Al₂O₃ electrodes were prepared by the powder metallurgy route and transferred to the substrate surface using the EIL-8A apparatus. The cermet layers were subsequently melted by means of the BLS 720 Nd:YAG laser. The coating characterisation studies showed marked improvements in the bonding between the steel substrate and the cermet coating, and in corrosion resistance at the expense of higher surface roughness and minor drop in the Vickers microhardness.

Keywords: electro-spark deposition, laser treatment, coating, roughness, tribology, microstructure, corrosion resistance, microhardness, adhesion

Głównym celem pracy było określenie wpływu obróbki laserowej na mikrostrukturę, mikrotwardość, chropowatość, przyczepność, odporność na korozję i właściwości tribologiczne powłok odpornych na ścieranie naniesionych na stal węglową C45 w wyniku procesu obróbki elektroiskrowej (ESD). Zużywalne elektrody WC-Co-Al₂O₃ wykonano metodą metalurgii proszków, a materiał наносono na powierzchnie podłoża używając urządzenia EIL-8A. Powłoki cermetowe były następnie przetapiane laserem BLS 720 Nd:YAG. Badania powłok wykazały znaczną poprawę w wiązaniu między stalą a pokryciem cermetowym, a także poprawę odporności na korozję, ale za cenę wyższej chropowatości powierzchni i niewielkiego zmniejszenia mikrotwardości Vickersa.

1. Introduction

Application of protective coatings to a variety of machine parts and structural elements as well as cutting and shaping tools is often the most economical way of improving their surface properties, such as resistance to wear and corrosion, sliding properties, cosmetic appearance, etc. Coating deposition can either be included in the device manufacturing process (technological surface layers) or become a routine action which keeps the device in working order (functional surface layers).

A number of modern surface processing methods, including electro-spark deposition (ESD) and laser treatment, rely on energy transfer to the substrate surface. Although its history dates back to the 1960's the ESD process found industrial application to deposit refractory materials on metals and alloys, mainly steels, in the 1970's. As shown schematically in Fig. 1, the ESD consists in transferring an ionized electrode material to

the substrate surface utilising electric energy and involve chemical, electro-chemical and electro-thermal reactions [1].

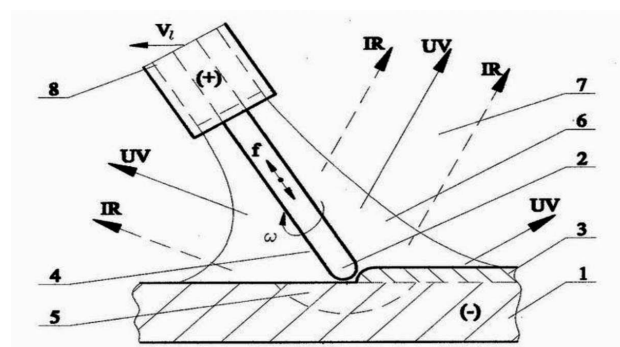


Fig. 1. Schematic view of coating formation by electro-spark deposition method: 1 – substrate material (cathode), 2 – consumable electrode (anode), 3 – coating, 4 – plasma, 5 – diffusion or reactive-diffusion zone, 6 – shielding gas, 7 – air, 8 – electrode holder with gas supply channels, IR – infrared radiation, UV – ultraviolet radiation

* TECHNICAL UNIVERSITY OF KIELCE, 25-314 KIELCE, ALEJA 1000-LECIA PANSTWA POLSKIEGO 7, POLAND

** AGH-UNIVERSITY OF SCIENCE AND TECHNOLOGY, 30-059 KRAKÓW, 30 MICKIEWICZA AV., POLAND

The electric spark locally generates very high pressure and temperature reaching $5,000\div 40,000^{\circ}\text{C}$ on the tip of the electrode [2].

To date a number of alternative ESD techniques has been developed to suit various coating deposition conditions and meet surface topography requirements [3-9]. ESD coatings may serve as both technological and functional surface layers and can be deposited using a portable, manually operated equipment or fully automated systems.

ESD coatings have some disadvantages, such as high roughness and presence of porosity and discontinuities but these can be eliminated by laser treatment. The laser beam is used for surface sealing, removing surface irregularities, homogenizing the chemical composition of the coating and changing its phase composition [10-13]. Therefore laser-treated ESD coatings show lower porosity, better adhesion to the substrate, higher resistance to wear and seizure, higher fatigue strength due to the presence of compressive stress, and good resistance to corrosion [14].

This paper reports on the effects of laser treatment on microstructure, microhardness, roughness, bonding strength, corrosion resistance and friction coefficient of ESD WC-Co-Al₂O₃ coatings.

2. Experimental procedure

The coatings were deposited on the C45 grade plain-carbon steel by the ESD method using a portable equipment shown in Fig. 2.



Fig. 2. Portable EIL-8A electro-spark deposition facility (TRIZ, Ukraine)

The electrodes, of composition 85% WC, 10% Co and 5% Al₂O₃, were produced using the powder metallurgy hot pressing route [15]. The main characteristics of the powders used in this work are included in Table 1.

The powders were mixed for 30 minutes in the chaotic motion *Turbula T2C* mixer. The mixture was then poured into rectangular cavities of a graphite mould,

each 6×40 mm in cross section, and consolidated by passing an electric current through the mould under uniaxial compressive load. A 3 minute hold at 950°C and under a pressure of 40 MPa permitted obtaining electrodes of porosity $<10\%$ and strength sufficient to maintain integrity when installed in the electrode holder.

TABLE 1
Powders used to manufacture cermet electrodes

| Powder | Particle Size, μm | Surface Area (B.E.T.), m^2/g | Producer |
|--------------------------------|------------------------------|--|--------------|
| WC | $\sim 0.2^{\dagger}$ | 1.8 | OMG |
| Co | $\sim 1.4^{\dagger}$ | – | Umicore |
| Al ₂ O ₃ | $18\div 60^{\dagger\dagger}$ | – | Sulcer-Metco |

[†] measured using Fisher Sub-Sieve Sizer

^{††} measured using Sympatec's *HELIOS* laser analyser

To deposit the WC-Co-Al₂O₃ coatings the EIL-8A apparatus was operated at voltage, current and capacitance of 230 V, 2.4 A and 150 μF , respectively. The coatings were afterwards treated with the *Baasel Lasertechnik 720* Nd:YAG laser run in pulsed mode at a power level of 20 W, spot size of 0.7 mm, pulse duration of 0.4 ms and pulse repetition frequency of 50 Hz. The sample movement rate and beam shift jump were set to 250 mm/min and 0.4 mm, respectively.

The cermet WC-Co-Al₂O₃ coatings were tested for surface roughness, friction properties, scratch adhesion, resistance to corrosion, microhardness, and subjected to metallographic examination and X-ray diffraction (XRD) analysis in both as-deposited and laser treated condition.

3. Results and Discussion

3.1. Profilometry

The roughness of the WC-Co-Al₂O₃ coatings was quantitatively assessed using the *Taylor-Hobson Form Talysurf-120L* surface profiler. Roughness profiles are routinely measured by dragging a stylus along the laser beam path whereas the maximum values of the arithmetic average departure from the surface plain are reported to occur in the perpendicular direction. Therefore in this study an average value of R_a was calculated for each coating from readings taken on evenly divided sampling lengths running parallel to the electrode/laser beam motion and on similar lengths at 90° .

It was found that the employed surface treatments increased the average roughness value (R_a) from $0.39\div 0.41$ μm for the C45 steel substrate up to $6.16\div 7.79$ μm and $8.12\div 9.03$ μm for the WC-Co-Al₂O₃ coatings in as-deposited and laser treated condition, respectively.

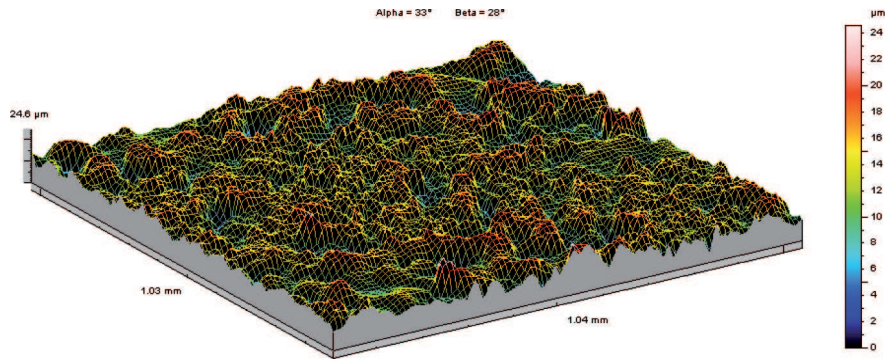


Fig. 3. Surface topography of the WC-Co-Al₂O₃ coating

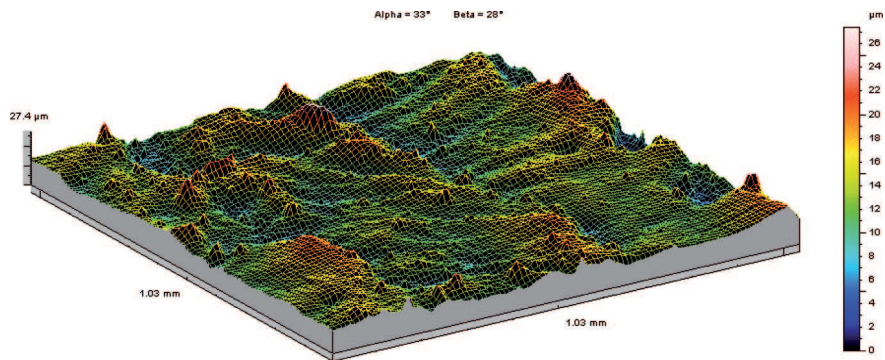


Fig. 4. Surface topography of the WC-Co-Al₂O₃ coating after laser treatment

Presumably the increase in *Ra* readings after laser treatment resulted from an unequal distribution of temperature in the laser beam, and consequently a non-uniform distribution of energy in the laser melted zone, which augmented geometric irregularities on the surface after solidification [16].

Typical surface topographies of as-deposited and laser treated coatings are shown in Figures 3 and 4.

3.2. Tribology

The *T-01M* pin-on-disc tribometer was employed to determine the dry friction behaviour of the WC-Co-Al₂O₃ coatings. A ball-on-flat contact geometry was chosen to measure the friction force between a 100Cr6 grade steel ball, 6.3 mm in diameter, and the tested coating. During each test cycle a new ball was pressed against the tested surface and set in sliding motion for 3600 s over the rotating sample as explained schematically in Figure 5.

P is the normal force applied on the ball (14.7 N),
R is the radius of the wear track (0.014 m) and
n is the rotational speed of the disk (427 rpm).

As can be seen from Figure 6, the 100Cr6

steel shows markedly lower friction coefficient on as-deposited coating than on its laser treated counterpart. Initially, the friction coefficient increases and after 500 and 1200 s stabilizes at a level between 0.35÷0.37 and 0.55÷0.58 for the as-deposited and laser treated WC-Co-Al₂O₃ coating, respectively. Interestingly, in the latter case the friction coefficient has lower value when the testing cycle starts but the red line (Fig. 6) slopes up very steeply and exceeds the black line after around 700 rotations.

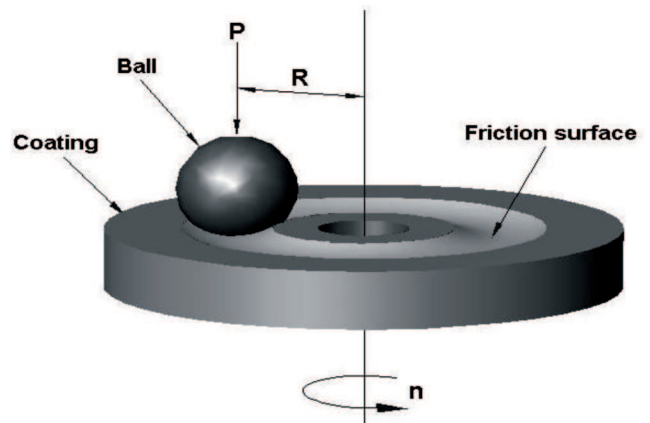


Fig. 5. The ball-on-disk setup used to monitor the friction coefficient

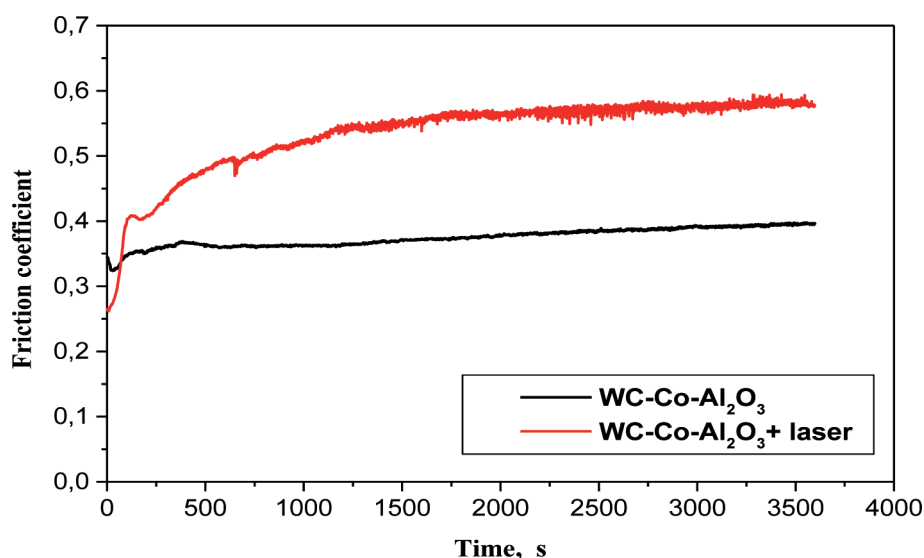


Fig. 6. Variation of friction coefficient over time between the steel ball and: as-deposited coating (black line); laser treated coating (red line)

3.3. Scratch adhesion testing

Scratch adhesion testing was conducted to assess the adhesion of the WC-Co-Al₂O₃ coatings to the C45 grade steel substrate using the *CSEM Revetest* scratch tester. The tests consisted of scratching a coated, or coated and laser treated, sample with a diamond stylus of 200 μm tip radius at a table speed of 9.77 mm/min under step-wise increasing normal stylus load of 103.2 N/min. Coating removal, and hence the critical stylus load, was identified through a combination of optical examination, changes in the tangential force and acoustic emission. The results are presented in Table 2.

TABLE 2

Results of scratch adhesion tests

| Coating condition | Critical stylus load, N | | | Mean value †, N |
|-------------------|-------------------------|------|------|-----------------|
| | Measurement | | | |
| | 1 | 2 | 3 | |
| As-deposited | 7.42 | 6.67 | 4.89 | 6.3 ± 3.8 |
| Laser treated | 8.92 | 9.26 | 8.65 | 8.9 ± 0.9 |

† scatter intervals estimated at 90% confidence level

From the obtained data it becomes evident that due to laser treatment it is possible to markedly improve adhesion of the WC-Co-Al₂O₃ coating to the C45 steel substrate. In addition, the low scatter of critical stylus loads indicate that the laser treatment presumably eliminate voids present at the coating/substrate interface.

3.4. Corrosion resistance testing

The corrosion resistance was assessed by estimating the corrosion current density (I_k) from current versus voltage data, using the *Atlas-Sollich Atlas'99* computerised system for electrochemical tests. The polarisation curves were determined by polarizing the samples with a potential shift rate of 0.4 mV/s over a range of about 800 mV. The measured data were then fitted to the theoretical model of the corrosion process [17]. The tests were performed at 21 ± 1°C on samples, 10 mm in diameter, which were exposed to a 3.5% NaCl solution for 24 hours. The results are summarised in Table 3.

TABLE 3

Corrosion current densities of the tested materials

| Material | Corrosion current density (I_k), μA/cm ² |
|--|---|
| C45 | 35.4 |
| As-deposited WC-Co-Al ₂ O ₃ | 16.8 |
| Laser treated WC-Co-Al ₂ O ₃ | 13.5 |

The obtained I_k values indicate over 100% increase in corrosion resistance of the ESD coated sample compared to uncoated C45 steel substrate. Further increase in corrosion resistance occurs after laser treatment which may be explained in terms of microcrack healing and thus preventing penetration of corrosive fluid to the substrate.

3.5. Microhardness testing

The microhardness tests were carried out with a Vickers indenter, under a load of 0.4 N, on polished

cross-sections at three different locations, i.e. in the coating, in the heat affected zone (HAZ) located close to the coating, and in the substrate at a certain distance from the coating. An uncoated substrate material was also tested. The hardness test results presented in Table 4 indicate minor decrease in microhardness of both the coating and HAZ after laser treatment, whereas the steel substrate remains unaffected.

TABLE 4

Results of microhardness tests

| Coating condition | HV0.4 † | | |
|-------------------|---------|---------|-----------|
| | Coating | HAZ | Substrate |
| As-deposited | 843 ±24 | 378± 37 | 141±6 |
| Laser treated | 784±26 | 342±17 | 143±8 |

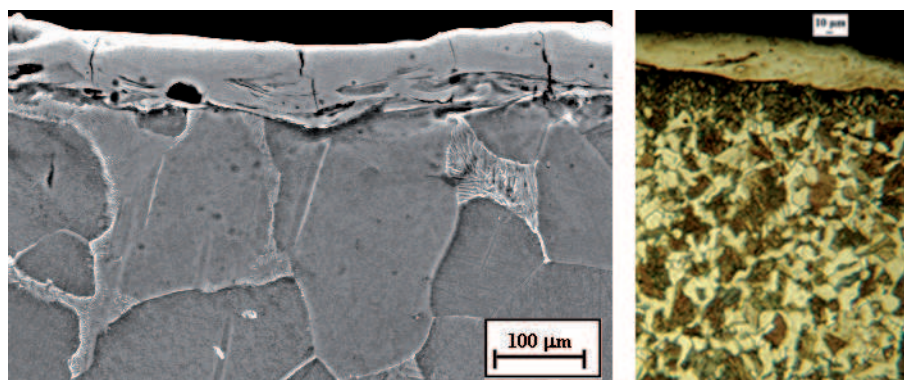
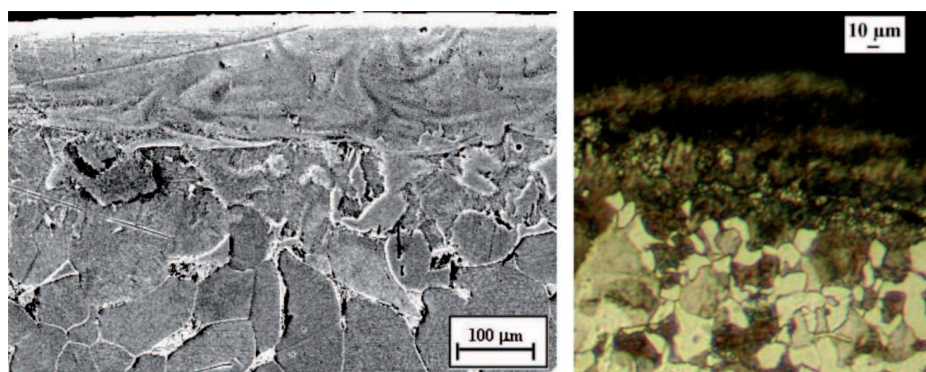
† scatter intervals estimated at 90% confidence level

3.6. Metallographic examination and X-ray diffraction analysis

Selected samples, representative of each coating condition, were also examined by means of scanning electron microscopy (SEM) and light microscopy (LM). Metallographic sections were prepared by cutting each sample at a right angle to its surface, mounting it in a conducting resin, wet grinding on SiC paper and polishing on cloths impregnated with diamond compounds of decreasing grit size.

The SEM examination revealed a distinct interface between the ESD WC-Co-Al₂O₃ coating and the substrate (Fig. 7).

The as-deposited coating has a thickness of between 60 and 70 μm, and shows interfacial shrinkage voids, internal porosity and cracks. Apparently the susceptibility to cracking can be attributed to the tensile thermal stress generated during solidification of the molten electrode material. As seen in Figure 8, it is possible to heal up the cracks and remove porosity and interfacial imperfections by laser re-melting of the coating.

Fig. 7. SEM (left) and LM (right) micrographs of the polished cross section through a WC-Co-Al₂O₃ ESD coating on C45 steel substrateFig. 8. SEM (left) and LM (right) micrographs of the polished cross section through a WC-Co-Al₂O₃ laser treated coating on C45 steel substrate

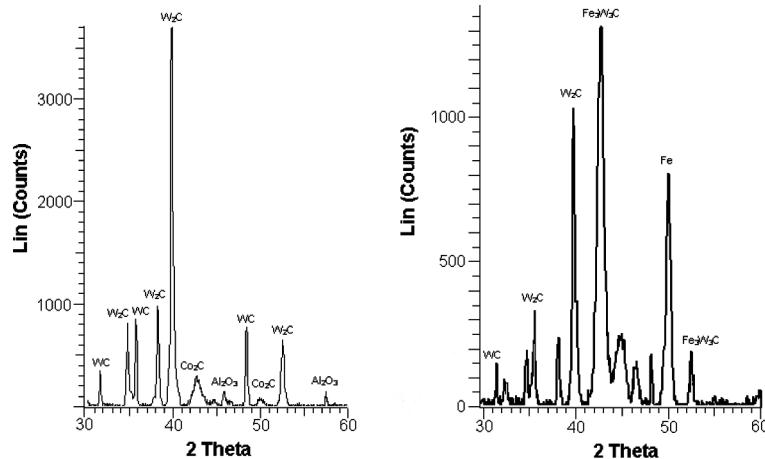


Fig. 9. XRD patterns indicating phases present in: as-deposited coating (left); laser treated coating (right)

The laser treatment also refines the grain size of the steel substrate within the HAZ and markedly increases thickness of the coating, to $90 \div 110 \mu\text{m}$, which indicates that the laser melted zone stretches to the substrate. A dissolution of the steel substrate material in the coating was corroborated by XRD analysis. The XRD patterns presented in Figure 9 reveal presence of Fe and $\text{Fe}_3\text{W}_3\text{C}$ carbides in the laser melted coating, whereas these phases were not found in the coating prior to laser treatment.

4. Conclusions

A concentrated laser beam effectively modifies functional properties of the ESD WC-Co- Al_2O_3 coatings through:

1. $\sim 50\%$ increase in the coating thickness.
2. Removal of cracks and internal porosity resulting in increased potential to combat corrosion.
3. Elimination of coating/substrate interface voids and thus marked increase in coating adhesion to the carbon steel substrate.
4. Minor increase in surface roughness and friction coefficient on a bearing steel.
5. $\sim 7\%$ decrease in the Vickers microhardness.

The paper was performed within N N503 150736 project financed by the Ministry of Science and High Education

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