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Process and properties of $TiC_{0.7}N_{0.3}$-based cermet coating deposition on steel by electrospark deposition technique

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Abstract

Electrospark deposition (ESD) method is a promising process to produce hard and wear-resistant coatings on metallic substrates. In this study, ESD has been successfully applied to deposit $TiC_{0.7}N_{0.3}$-based cermet coatings on steel substrate. It has been possible to obtain a fully dense and strong adherent coating layer with a thickness of 20 μm. The microstructures of the deposited coatings were characterized by X-ray diffraction (XRD) and scanning electron microscopy. In addition, the friction and wear properties of the coatings and substrates were investigated systematically by conducting ball-on-disc sliding wear tests. The experimental results demonstrate that the wear resistance of the steel substrate, which contains a TiCN coating deposited via the ESD process, is noticeably enhanced under the dry sliding wear conditions. In addition, while the microhardness of substrate is 232 HV on average, the microhardness of the coating reaches 870 HV onto the surface.

Key words: electrospark deposition (ESD), titanium carbonitride, micro-hardness, sliding wear

1. Introduction

The electrospark deposition (ESD) process is a surface treatment technique to produce hard and wear-resistant coatings on various metallic materials. It uses high current/low voltage electrical pulses of short duration to coat an electrode material (anode) with a substrate (cathode) [1]. During the process, stored energy from high voltage capacitors is discharged through an electrode of the material to be deposited. Thus, a small molten part of the material is removed from the electrode and coated to the substrate in the form of a sudden spark [1, 2].

The ESD coating process offers some unique advantages relative to electrodeposition and other surface modification techniques. In the ESD process, the total heat input to the workpiece is relatively small and the bulk substrate remains near ambient temperature. Thus, the bulk metallurgical structure of the substrate is relatively unchanged, and thermal distortions are minimized. The heat rapidly dissipates leading to rapid solidification. This results in an extremely fine-grained coating of high density, hardness and strength. The ESD process allows the use of any electrical conductor as the electrode; therefore, a variety of surface types can be formed. The process can be used to deposit even ultra hard ceramic coatings on a metallic substrate. It has originally been used to generate wear-resistant and corrosion-resistant coatings. Additional advantages of the ESD process are that it is relatively inexpensive and that portable equipment can be used [1, 3].

Cermet-based coatings are extensively used not only to fulfil the requirements of enhanced wear resistance for many applications of surface engineering, but also to enhance the service life of working tools and machine parts. TiCN-based cermet coatings combine the advantages of the high hardness of TiC, the ductility of TiN, and high adhesion strength, giving TiCN-based cermet coatings significantly improved mechanical properties relative to TiC or TiN coatings. Therefore, TiCN coatings are highly promising for various wear applications [4–8].

Several high-energy coating technologies have been

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*Corresponding author: tel.: +90-262-605-1784; fax: +90-262-653-8490; e-mail address: korkmazk@gyte.edu.tr used to produce TiCN coatings, however, the ESD process is a simple and cost-effective low-energy technique. This method produces metallurgical bonding between the coatings and substrates, resulting in a much better coating adhesion compared to other low-energy coating processes, such as detonation-gun, plasma-spray, and electrochemical plating [1, 2]. In view of the potential demand for wear-resistant coatings deposited at low substrate temperatures, it is important to study the preparation and properties of these TiCN coatings in detail. In this paper, we report the results of the analysis of the structure-property relationship in these coatings and their dependence on the deposition conditions when synthesized by the ESD process.

The aim of the present research is to coat $TiC_{0.7}N_{0.3}$ on the St37 steel substrate by using the ESD process and to analyse the morphology, microstructure, and sliding wear behaviour of the coatings. Friction and wear measurements were carried out in a ball-on-disc tribometer using alumina balls.

2. Experimental procedure

2.1. ESD device

A special ESD machine was employed in the experiments. The ESD coating system was described in previous work [9]. Its power consumption was 180 W and the output of stabilized voltage was constant at 40 V.

2.2. Materials

In the ESD process, a number of different treating electrode materials can be used, but these materials must conduct electrical current. In this study, titanium carbonitride ($TiC_{0.7}N_{0.3}$) produced by a conventional powder metallurgy technique was selected as a treating electrode material. The powder mixture, consisting of TiC(70)-TiN(30) alloy powder and a small amount of Al powder as a liquid-phase additive for sintering, was prepared in a turbula mixer for 3 h at $60\,\mathrm{Hz\,s^{-1}}$. The mixture was then uniaxially pressed into bar shapes (4 mm thick × 5 mm wide × 15 mm long) at a pressure of 450 MPa. The pressed compacts were sintered in vacuum at 1400 ℃ for 1 h. To deposit a coating on the St37 steel workpiece ($10 \times 10 \times 10\,\mathrm{mm^3}$), the sintered electrodes of TiCN containing Al were used.

2.3. Coating process

The ESD process is performed using a hand-held applicator in air at room temperature. In the system, the voltage drops at the interelectrode gap (17 V), and the amount of electricity (3 C) is kept constant. The ESD process was conducted in air with a series of rectangular pulses of a duration of 200 μs, a repetition rate of 100 Hz, and amplitudes of current of 400 A.

2.4. Wear-resistance and friction tests

A standard ball-on-disc tribometer (CSM Instruments) was employed to investigate the wear behaviour of TiCN-coated and uncoated samples. The tribometer was controlled by its PC software, which allowed to follow the evolution of the friction coefficient. The dry sliding wear test experiments were performed in air at room temperature and at a relative humidity of 50 ± 2 %. An alumina ball with a diameter of 6 mm was loaded with constant normal loads of 5 N and 10 N, while the sliding speed was $10\,\mathrm{cm\,s^{-1}}$, and the sliding distance was 200 m. After the wear tests, in order to find the wear coefficient, the lost volume of the wear track was measured by using an optical profilometer (DEKTAK8, Advanced Development Profilometer Veeco).

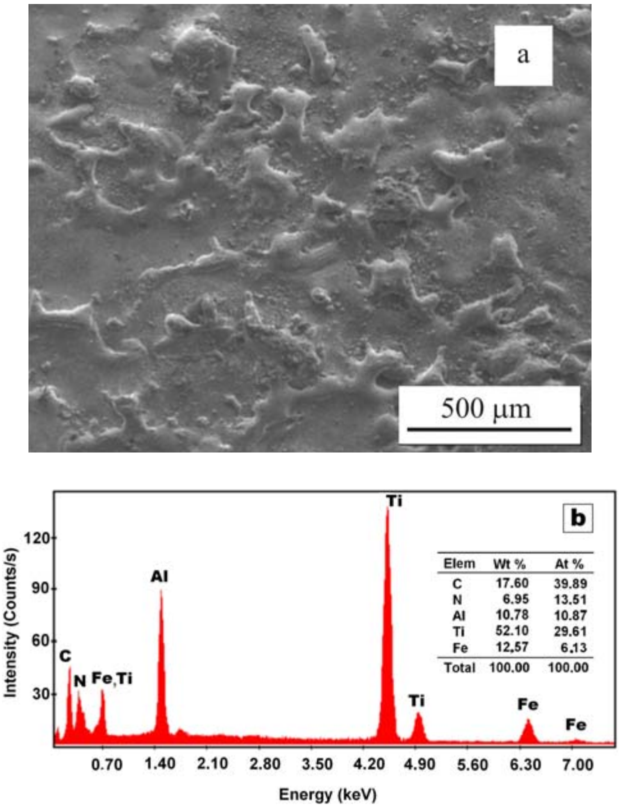
2.5. Characterization

Both the surface morphology on the coated samples and thickness of the deposit layer were investigated using a scanning electron microscope (SEM, Philips XL 30 SFEG). Phase composition, microstructure, and elemental distribution analyses of the deposit layer were performed using an X-ray diffractometer (Bruker D8 Advance) and an embedded EDX digital controller (EDX detector operating 15 kV) attached to the SEM. X-ray diffraction pattern measurements were performed in the range of 20° to 90°, at a scanning speed of $2^\circ\,\mathrm{min^{-1}}$, in Cu K$\alpha$ radiation, and with the value of λ at 0.1504 nm.

Vickers microhardness (HV) measurements were taken on polished cross-sections of the coatings. These measurements were made with a microhardness tester (MHT-10 Microhardness Tester, Anton Paar) at a load of 50 g and a loading time of 10 s. Metallographic analysis was carried out using an optical microscope (Zeiss Avio, Imager) equipped with VideoTest Structure software. Roughness measurements were conducted by using a profilometer device (SJ-400 Mitutoyo) with a precision of 0.01 μm. The thickness of the deposit layer was measured by a thickness meter DualScope MP-40 (Fischer Inst.). Mean value and error variance were calculated based on 20 random measurement points onto the coating of the surface.

3. Results and discussion

The top surface morphology of the coating deposited on the St37 steel substrate by the ESD process



Elem	Wt %	At %
C	17.60	39.89
N	6.95	13.51
Al	10.78	10.87
Ti	52.10	29.61
Fe	12.57	6.13
Total	100.00	100.00

Fig. 1. The morphology (a) and EDX analysis (b) of the coating surface obtained by using the ESD process.

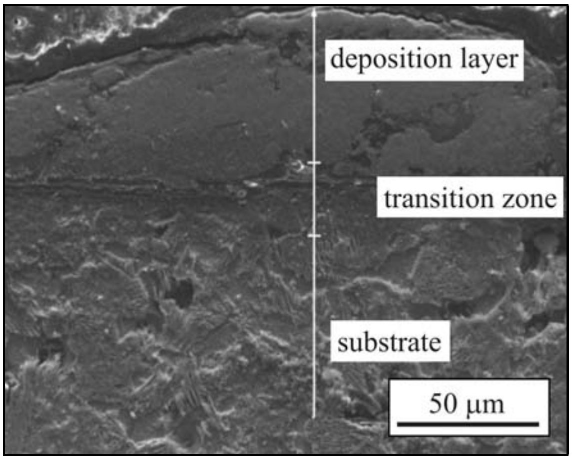


Fig. 2. SEM micrograph of a cross-section of ESD deposition TiCN coatings onto St37 steel substrate.

and the EDX analysis are illustrated in Figs. 1a,b. The presence of Ti, N, C, Al, and Fe elements proves the alloy formation between the coating and the substrate. As can be seen, the coating surface is characterized as irregular and rough view, due to the globular mass transfer mechanism during the ESD process from the electrode to the substrate. This is a characteristic feature of the ESD process [2, 3]. A molten globular droplet forms at the end of the TiCN electrode tip, and it is moved by discharge plasma channel towards the substrate surface, which leads to a "splash" appearance. Similar results were reported in earlier studies [10, 11]. This shows the roughness and uniformity of the coating. The average roughness (of fifteen measurements) was reported to be approximately $R_a = 7.8 \pm 3.2$ µm for the surface of the coating. Some random results were taken by using a DualScope thickness meter on the surface of the coatings, and then an average coating thickness of these results was calculated to be 27.9 ± 17.2 µm.

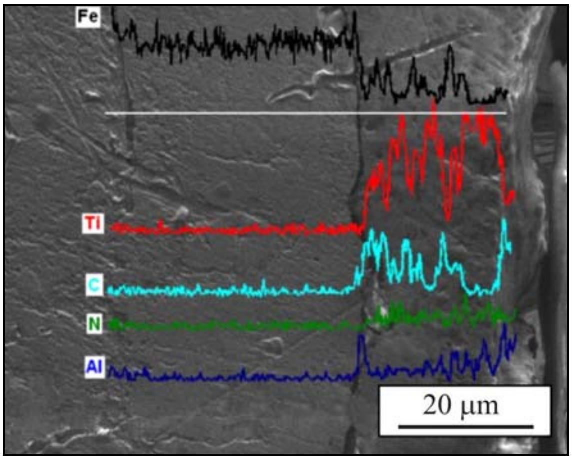


Fig. 3. Graphical representation of the elemental distribution of Ti, C, N, Al, and Fe along the cross-section.

Figure 2 presents a typical SEM micrograph for a cross-section of the electrospark deposited TiCN coatings on the steel substrate. It can be seen that there are three continuous zones (deposition layer, transition zone, and substrate) throughout the cross-section. There is a continuous interface between the coating and the substrate. The excellent metallurgical bonding between the coating and the substrate was established by alloying. However, the coating thickness is not uniform, as seen by the presence of a few micro-cracks and micro-pores. This indicates that both the deposited material (TiCN) and the substrate must have been molten during the process, and some gas bubbles and solidification cracks had been formed.

The distribution of elements, such as Ti, C, N, Al, and Fe, in the coating and the substrate was determined by EDX (Energy Dispersive X-ray Spectrum) elemental line scanning technique, and these data are presented in Fig. 3. According to the elemental graph, the amount of Ti, C, N, and Al decreases from the coating surface to the substrate, while Fe increases. As a result, there is a short-range interdiffusion (transition) zone, and this indicates the existence of a narrow mixing and remelting layer between the substrate and the coating material during the deposition process.

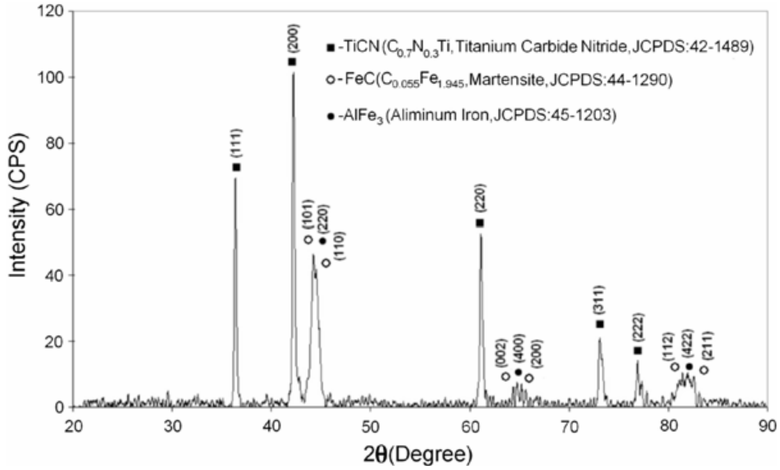


Fig. 4. X-ray diffraction spectrum of TiCN coating on steel.

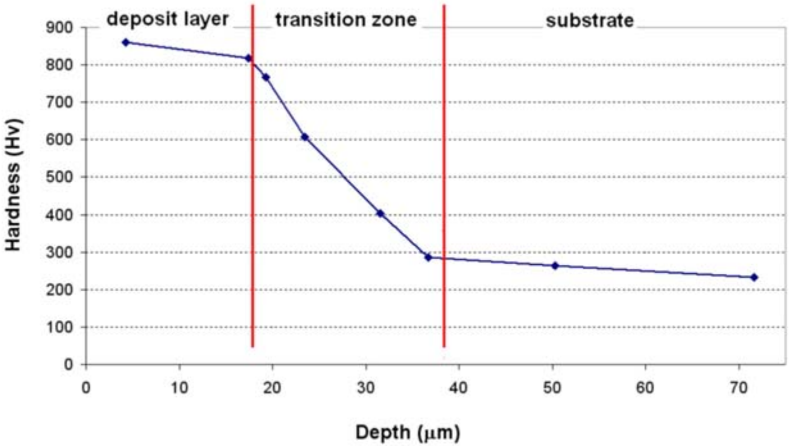


Fig. 5. Microhardness depth profile of TiCN coating on steel.

The X-ray diffraction spectrum for the coated surface is presented in Fig. 4, which reveals the present phases in the coating. According to the spectrum, the coating layer is mainly composed of titanium carbide nitride ($TiC_{0.7}N_{0.3}$), martensite ($Fe_{1.945}C_{0.055}$), and aluminium iron (Fe_3Al) phases. The presence of the martensite and aluminium iron phases is related to the alloying of the coating and the substrate and its rapid solidification.

Figure 5 shows the microhardness distribution along the depth of the electrospark deposited TiCN coating on the steel substrate. The peak value for microhardness, HV, was found to be 870, which appears on the top surface of the deposit layer, and is approximately four times higher than that of the substrate (232 HV). There is a gradual decrease in the microhardness values with depth into the deposit layer toward the substrate, and this situation is related to the decreasing amounts of both the $TiC_{0.7}N_{0.3}$ phase and martensite phase through the transition zone and the main substrate zone.

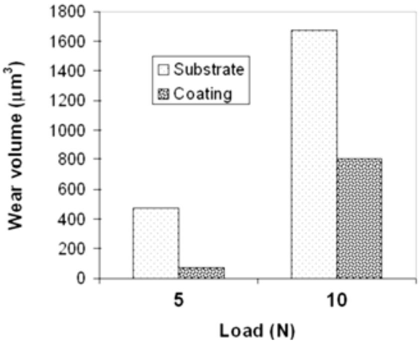


Fig. 6. Wear volumes of TiCN-coated and uncoated steel after sliding wear test against alumina ball.

Wear test results of the uncoated and ESD processed TiCN-coated St37 steel samples under dry sliding wear test conditions are presented in Table 1 and Fig. 6 (units of specific wear rate, worn volume per unit, sliding distance per unit, and normal load). It

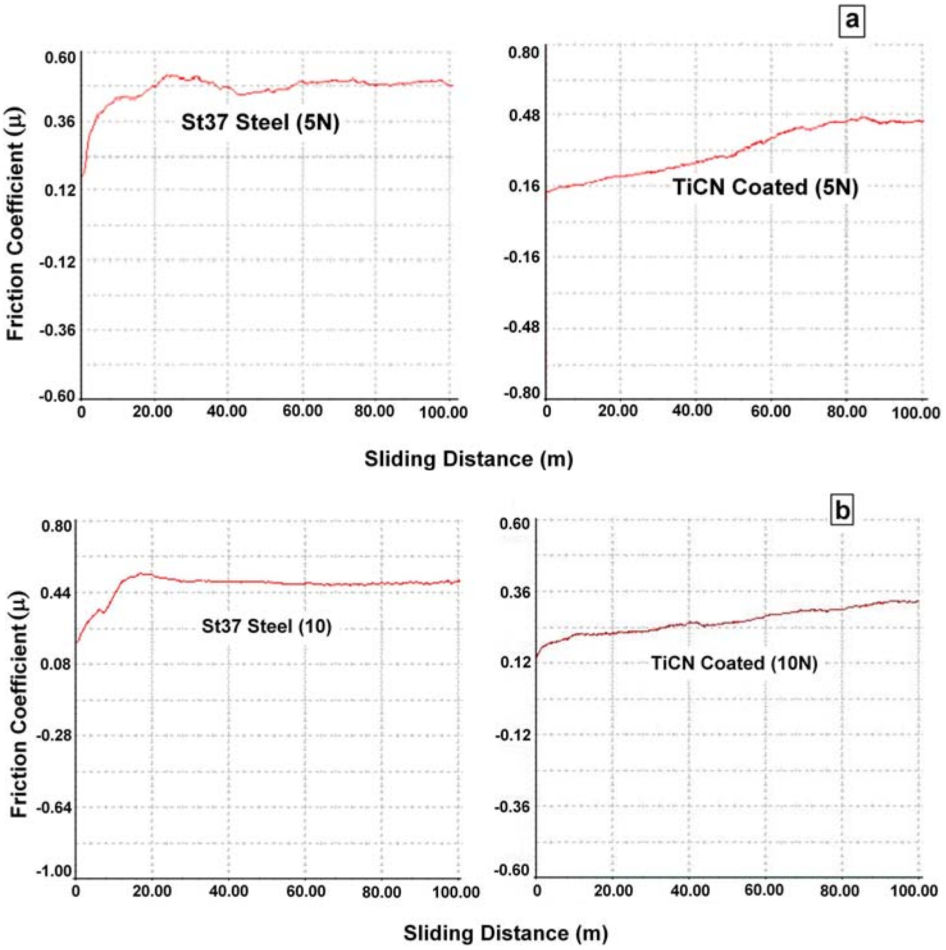


Fig. 7. Evolution of the friction coefficients against sliding distance (less than 100 m) for 5 N (a) and for 10 N (b) loads.

Table 1. Sliding wear test results

Samples (load)	Wear rate ($\mathrm{mm^3\,N^{-1}\,m^{-1}}$)	Friction coefficient mean
St37 steel (5 N)	1.614×10^{-5}	0.465
TiCN coating (5 N)	2.455×10^{-6}	0.431
St37 steel (10 N)	2.482×10^{-5}	0.488
TiCN coating (10 N)	1.360×10^{-5}	0.328

is evident from Fig. 6 that TiCN electrospark depositing on the steel significantly improves the wear resistance. Under 5 N and 10 N loads, the wear resistances of the coated samples are, roughly, up to 7 times greater and up to 2 times greater, respectively, than those of the uncoated samples. The variation of the friction coefficient with the sliding distance for the electrospark deposited TiCN coating and St37 steel substrate are also presented in Figs. 7a,b. The data generally indicate nonlinear increases in the friction coefficient during the sliding wear process until stable levels are reached. It is noted that the average friction coefficients for the coated and uncoated samples are $\mu_c = 0.431$ and $\mu = 0.465$, respectively, for a 5 N load. For a 10 N load, the friction coefficient average values are $\mu_c = 0.328$ and $\mu = 0.488$ for coated and uncoated samples, respectively. For TiCN-coated samples, friction coefficients were observed to be lower than those of the uncoated samples. From the results of the wear resistance and friction behaviour and microhardness of the samples, it can be said that it is possible to obtain very hard coatings that show wear resistant with lower friction on the steel via the ESD process.

4. Conclusions

The electrospark deposition (ESD) process has been successfully applied to deposit a $\mathrm{TiC_{0.7}N_{0.3}}$-10Al layer onto an St37 steel substrate in air. As the microscopic observations reveal, the coating has an irregular and rough surface due to the globular mass transfer mechanism occurring during the process. However, the coating is fully dense with a minimal quantity of micro-cracks and micro-pores, and the coating is strongly adherent to the steel substrate. The thickness of the coating is typically more than 20 µm.

The coating is primarily composed of titanium carbonitride ($TiC_{0.7}N_{0.3}$) and martensite ($Fe_{1.945}C_{0.055}$) phases. The presence of these phases effectively improves the tribological performance of the coating. Thus, the wear resistance of the St37 steel substrate with TiCN deposition via the ESD process is noticeably enhanced under the dry sliding wear conditions. In addition, the hardness of the coating reaches 870 HV onto the surface. Via the ESD process, we have been able to obtain very hard coatings showing high wear resistance with low friction values.

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