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Deposition of TiC-Cu composite coating on AISI 304 stainless steel by EDC process using powder compact tool electrode

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In this work, powder compact tool electrode prepared with TiC-Cu powder mixture has been used to deposit TiC coating on AISI 304 stainless steel by electro discharge coating (EDC) process. Effects of peak current during EDC process have been analysed for the deposition rate, surface characteristics and micro-hardness value of the coating. Morphology of the deposited coating layers has been studied by the scanning electron microscopy (SEM) images while the compounds present in the coating layer have been analysed by X-Ray Diffraction (XRD) technique. Wear rate of the coated samples has been measured by ball-on-disc type sliding wear test against WC-Co ball. The experimental results revealed that higher peak current during the EDC process augmented the deposition rate and corresponding coating thickness. Micro-hardness value and wear resistance of the deposited coating has been found significantly improved than those of as received substrate material.

Keywords: Electro discharge coating, TiC coating, AISI 304 steel, Micro-hardness, Wear resistance

1 Introduction

Electro Discharge Coating (EDC) is an important aspect of electro discharge machine (EDM) that employs to improve the chemical and mechanical performance of the work-piece surface. Owing to electric spark prompted between the tool electrode and work piece, material from tool electrode gets eroded and deposited on the work piece surface by an incident of melting and solidification through a plasma channel[1]. With the simplicity in the equipment and process mechanism, EDC process has the potential to become an economical alternative, for the surface modification of various substrate materials. Numerous research works have been conducted in the field of surface modification by EDC process, to improve the surface properties of mild steel with copper-tin[2], HSS with tungsten carbide[3], AISI D2 steel with TiC/WC/Co[4], S45C steel with TiC[5], W-Cu coating on C-40 Steel[6]. It was reported that, with the deposition of these coatings, surface mechanical properties of the substrate materials *i.e.* hardness and wear resistance enhanced substantially.

AISI 304 stainless steel possesses unique combinations of mechanical and chemical properties, *i.e*. high strength, excellent corrosion and oxidation resistance, which made it most useful material for a structural component in chemical and food processing industry[7]. However, low hardness (200 HV), and poor tribological properties of AISI 304 stainless steel confine its application in the power transmitting devices or component those are prone to work in the abrasive environment. On the other side, TiC is a hard ceramic that has potential to improve the surface mechanical properties of the AISI 304 steel due to its high thermal stability, high hardness and excellent wear resistance along with the low coefficient of friction against standard counter material[8].

From the literature, it is revealed that the EDC process has a great potential for deposition of hard and wear resistance coating on a different type of substrate materials. However, no work on the surface modification of AISI 304 steel with TiC coating, by EDC process was reported. In this work TiC has been deposited on AISI 304 stainless steel by EDC process using powder compact tool electrode prepared with TiC and Cu. Effect of peak current during EDC process on the deposition rate and surface roughness of the coating has been evaluated. Compound phases present in the coating was identified by XRD analysis. The microstructure of the coating both from the top surface as well as from the cross-section was analysed by SEM. Dry sliding wear rate of the deposited coating was measured after ball-on-disc sliding wear test against WC-Co ball[9].

2 Experimental Planning and Procedure

Overall experimental procedure of this work includes, preparation of the tool electrode with TiC

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and Cu powder mixture, and then deposition of TiC coating by EDC process using EDM, measurement of the deposition rate, surface roughness value of the coated surface and micro-hardness value of the coating, and finally metallurgical characterization of the coating by XRD and SEM analysis.

The experimentation was performed by using a tool electrode prepared with TiC and Cu powder mixture. At first, TiC and copper powders were mixed (60:40 wt. %) thoroughly using a ceramic mortar-pestle and then the powder mixture was compacted under a press at a compression pressure of 300 MPa with a 15 mm diameter compaction die. Here, Cu powder acts as binding mediator and for enhancement of the strength of the compact. The melting as well as the sintering temperature of TiC is quite high. Hence, by using Cu as binder, at relatively low temperature sintering of TiC was achieved. Further, presence of Cu in the tool electrode increases its electrical conductivity, which is a basic requirement of a tool electrode[10]. Sintering of the prepared compacts was performed at 900 °C temperature under inert (Argon) gas. Detail of electrode preparation condition is depicted in Table 1. The prepared compacted pellet was then joined with tool extension part (made with mild steel) using electrically conductive silver based epoxy glue for proper holding of the tool electrode in the tool holding collet of EDM. Figure 1 shows the TiC-Cu tool electrode prepared by P/M method after joining with mild steel tool extension. AISI 304 stainless steel plate of 5 mm thickness with a dimension of 50 mm × 50 mm was taken as the substrate material. The chemical composition of AISI 304 stainless steel is shown in Table 2.

Using the TiC-Cu compacted tool, experiments were performed by varying the peak current (I_p) while keeping the other parameters (gap voltage, pulse on duration, duty cycle, and flushing condition) constant. Negative polarity *i.e.* tool electrode positive and workpiece negative was chosen for the present EDC process. The weight of the substrate was measured before and after each experimentation to obtain the amount of material deposited on the substrate surface and then deposition rate was calculated for per unit time. A detailed analysis of the compounds be present in the developed coating was executed by X-ray diffraction (XRD) technique. For the present experiment X-ray from Cu, Kα (λ=1.5418 A) radiation has been used and XRD pattern was analysed with the help of Phillip's X'pert high score software. The coated samples were cut at the cross-section of the coating layer and polished with finer graded polishing paper sequentially. After polishing, the cross-section of the coated samples was cleaned with acetone prior to SEM analysis and micro-hardness measurement of the coating. The macrostructure of the coating at the cross section was analysed using JEOL/EO scanning electron microscope. Micro-hardness value of the coating was measured at the cross-section with Vickers micro-hardness tester (Make: LECO, Model-LM810). For each sample, four measurements were considered and average micro-hardness value of the coating was deliberated.

Wear rate of the coated samples prepared by EDC process were measured after performing the sliding wear test with a ball-on-disc type tribometer (Make: Magnum). The coated specimens were slid against an 8 mm diameter WC-Co ball over 6 mm diameter track for 30 minutes and the weight loss of the coated

Fig.1 — TiC-Cu tool electrode prepared by P/M method after joining with mild steel tool extension.

Table 1 — Parameters of tool preparation by powder compaction method.

Size of the pellets	Powder composition	Compaction Pressure	Holding Time	Sintering Temperature
15mm diameter, 10mm height	TiC: Cu=60:40 wt.%	300MPa	2 minutes	900°C

Table 2 — Chemical composition of AISI 304 stainless steel.

Material	Fe	C	Si	Mn	P	S	Ni	Cr	Mo
AISI 304	69.62	0.07	0.75	1.731	0.045	0.031	8.554	18.97	0.224

Table 3 — Wear test condition used for ball-on-disc type sliding wear test.

Ball diameter	Load	Track diameter	Time for one test	Rotational speed	Sliding speed
8mm	20N	6mm	30min	400 rpm	125.66 mm/s

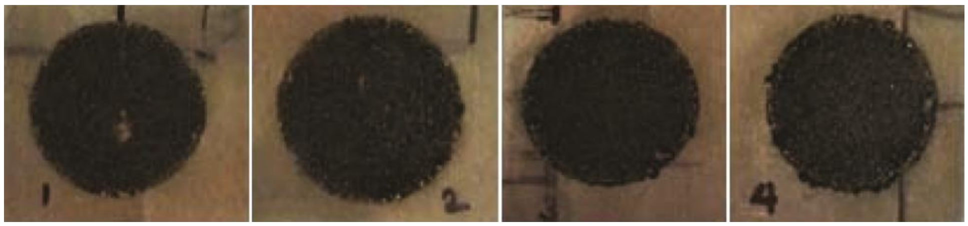

Fig. 2 — TiC-Cu coating deposited on AISI 304 stainless steel substrate by EDC process.

samples was measured using a precision balance with an accuracy of 0.1 mg. The wear rate was calculated using the Eq. (1). Details of wear test parameters are illustrated in Table 3.

$$\text{Wear rate} = (X_i - X_f)/T \quad \dots (1)$$

where, x_i and x_f are the weight of the coated specimen before and after the wear test and 'T' is the time of test.

3 Results and Discussion

During EDC process, TiC-Cu coating was successfully deposited on the AISI 304 stainless steel surface. Experimental data for deposition rate and surface roughness value of the coatings produced on AISI 304 stainless steel surface are shown in Table 3. Figure 2 shows the TiC coated stainless steel substrate that indicates a layer of coating material successfully deposited on the substrate surface during the EDC process.

3.1 Effect of Peak Current on Deposition Rate

Figure 3 shows the effect of peak current on the deposition rate of TiC-Cu coating on AISI 304 stainless steel substrate. From the graph, it is clearly observed that the deposition rate ranges from 8×10^{-4} to 1×10^{-3} g/min depending on the applied current; and as peak current increases, the deposition rate also gradually increases. However, the rate of change in deposition rate found higher when peak current changes from 2 to 4 A and this rate of change reduces for increasing the peak current from 4 to 5 A current. This may attribute to the fact that, with the increase in peak current, spark energy of electrical discharge becomes higher, which causes enhancement of dissociation of TiC-Cu powder from the tool electrode and consequent deposition of these powder particles on the work surface. During EDC process along with the deposition of tool material, due to spark energy, some amount of workpiece material removed also. When the deposition rate becomes higher than the removal rate, a coating is formed. Thus, it is expected that when the peak current becomes too high, *i.e.*, changing from 4 to 5 A, along with the deposition of the tool material effect of material removal becomes significant.

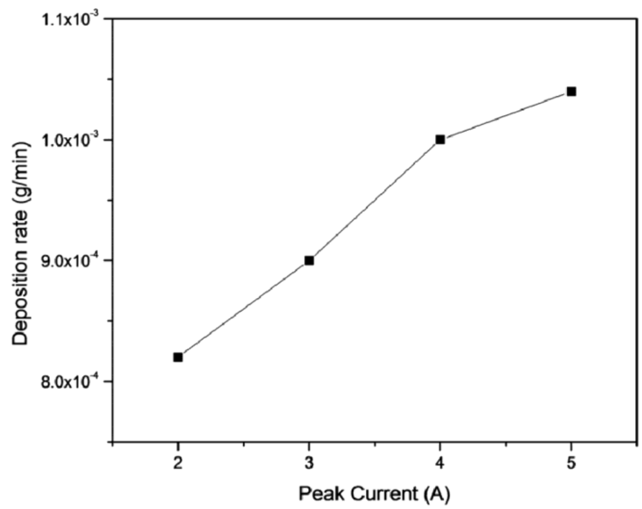

Fig. 3 — Effect of peak current on deposition rate of TiC-Cu coating on AISI 304 stainless steel substrate produced by EDC process.

3.2 Effect of Peak Current on Surface Roughness

Effect of peak current on the roughness value of the TiC-Cu coated AISI 304 steel surface is shown in Fig. 4. From the plot, it is observed that, at lower current, the average surface roughness value of the TiC coated stainless steel substrate is 5.16 µm and with the increase in peak current this value increases and extended up to 8.14 µm for the coating prepared with 5 A peak current. Roughness value of the coated surface mainly depends on the uneven deposition, crater formation and presence of cracks or pores. At higher peak current, due to high spark energy, dissociation of the tool electrode enhances and subsequently deposited on the substrate surface in bulk or at a faster rate. This phenomenon resulted in higher roughness on the coating surface. Further, at higher current, along with the formation of cracks or pores, partial removal of the pre-deposited coating layer leads to the formation of the crater, which further prompts a coarser surface.

3.3 XRD Analysis of TiC-Cu Coated Stainless Steel

The XRD pattern obtained from the top surface of the coating processed with 2A peak current is shown in Fig. 5. From the analysis of the pattern, it is revealed that TiC and Cu are the major phases present in the coated surface. In addition, the peaks of iron, which is overlapped with TiC, are also identified from the XRD plot. Apart from these, no peaks of other materials were detected. Thus, may conclude that TiC-Cu coating formed on the steel substrate after EDC process. During electrical discharge coating process TiC and Cu from the tool electrode eroded and deposited on the steel substrate. However, no dissociation of TiC phase or reaction between the TiC and steel substrate occurred.

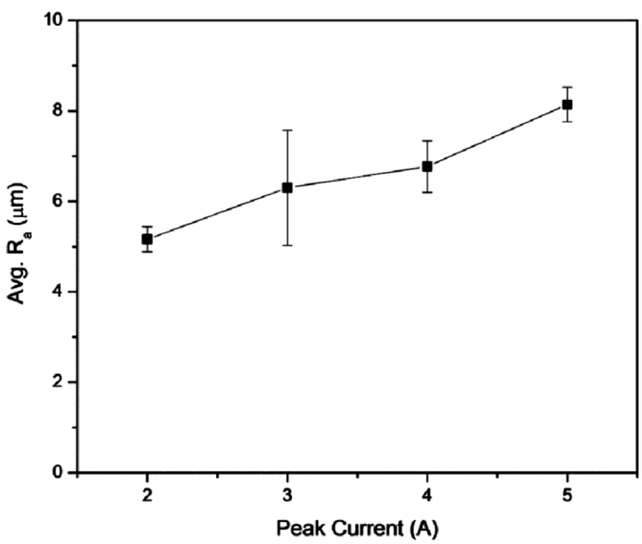

Fig. 4 — Effect of peak current on the average surface roughness of TiC-Cu coating on AISI 304 stainless steel substrate produced by EDC process.

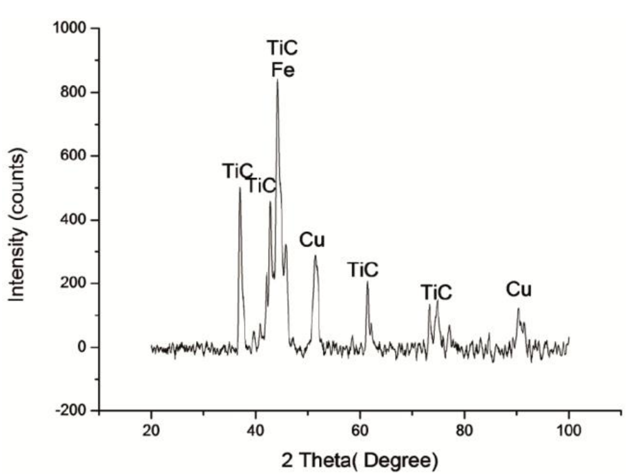

Fig. 5 — XRD graph of TiC- Cu coated AISI 304 stainless substrate by EDC process at 2 A peak current.

3.4 SEM Analysis of TiC-Cu Coated Stainless Steel

Figure 6 shows the SEM images taken at the cross-section of the TiC coated AISI 304 steel substrate. From the images, a layer of a composite structure consisting dark particles in a grey shaded matrix can be observed. The base metal can be identified from the white or light shaded phase. It is clearly observed from the images that when the peak current increases, the thickness of the coated layer also increases gradually. The average thickness of the coating was measured from the SEM images and plotted against the applied peak current which is depicted in Fig. 7. The plot indicates that the thickness of the coating increases from approximately 15 µm to 44 µm for increasing the peak current from 2 to 5 A, when all the other parameters remain constant. This may attribute to high spark energy at higher current, which causes the higher removal of the TiC particles from the tool electrode and deposition on the substrate at higher rate. The SEM images also revealed that the coating zone consisting dark particles along with some grey shaded matrix. Careful observation of the coating shows that no cracks have been formed at the coating and the substrate interface, which indicates a strong bonding between the produced coating and the substrate material. The images also show the formation of porosity on the coating, which increases with the increase of applied current during the coating process. These porosities are mainly formed in the coating due to entrapment of gas bubbles those are generated from the dissociation of the dielectric at a high temperature of the spark during the deposition process. With the increase in peak current, the probability of the gas formation and its entrapment also enhances, which resulted in an augmentation of the porosity.

Figure 8 shows the SEM images of the top surface of TiC-Cu coating deposited on AISI 304 steel and corresponding EDS analysis. Although the surfaces

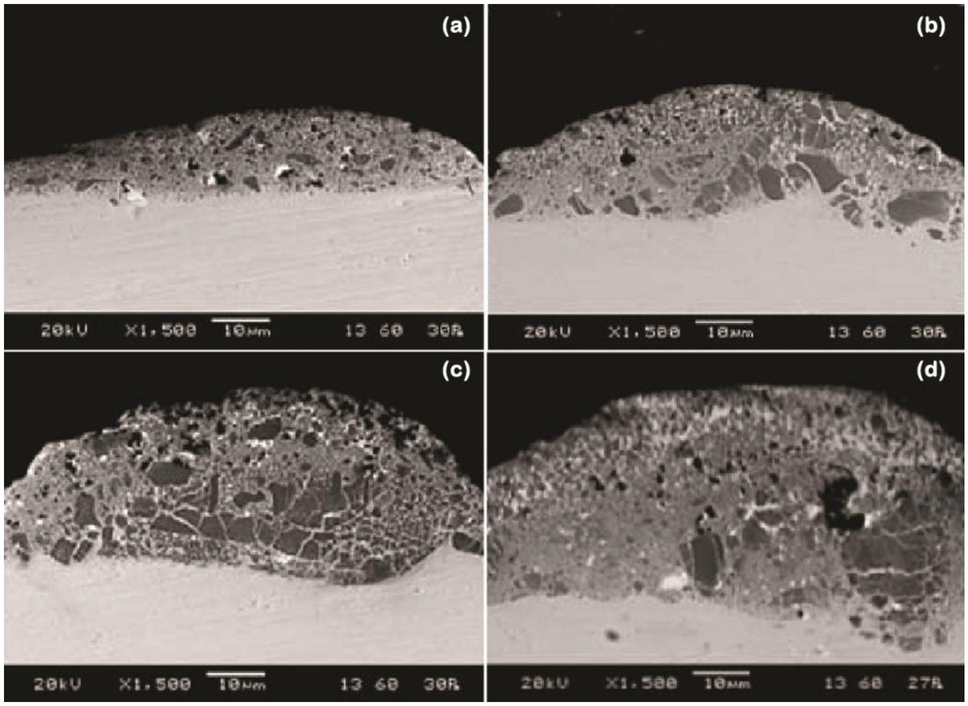

Fig. 6 — SEM images of the cross section of TiC-Cu coating on stainless steel substrate produced by EDC process with peak current of (a) 2 A, (b) 3 A, (c) 4 A and (d) 5A.

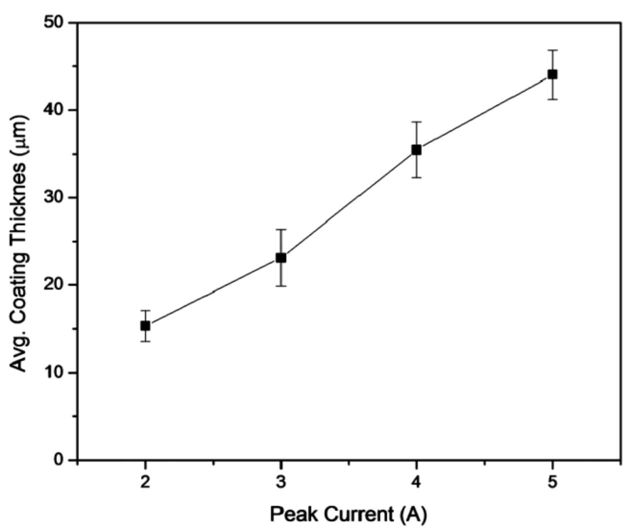

Fig. 7 — Variation of average thickness of the coating for different peak current during EDC process.

produced by EDC process are showing (at high magnification) uneven, however, careful observation of the images revealed that this unevenness increases with the increase of applied peak current. The unevenness on the coated surface mainly protruded due to the indiscriminate disintegration of TiC-Cu composite tool, its melting and deposition on the substrate and subsequent solidification of the molten powder composite along with some dissociated dielectric (mainly carbon particles). In the coating, some micro cracks were also observed on the surface of the coating. From the EDS spectra, the elements present on the coated surface are identified and it is revealed that with the increase in peak current, the percentage of Ti, C and Cu almost increases on the work surface. This may attribute to the deposition of higher amount of coating layer at higher peak current. Although a uniform coating has been formed by EDC process, some amount of Fe also detected from the base material, or due to partial dilution of the base material with the coating. Now, as the thickness of the coating increases with the increase of current, percentage of Fe becomes diminishing.

3.5 Micro-hardness

Figure 9 depicts the average micro-hardness value of TiC-Cu composite coating produced at different peak current condition by EDC process. The graph indicates that micro-hardness values of the coatings are in the range of 1340 to 1385 $HV_{0.025}$, which is almost six times higher than the hardness value of the AISI 304 steel substrate (220 $HV_{0.025}$). No specific difference or change in the average micro-hardness value of the coating has been observed for the change in peak current during the EDC process. However, a large variation in the standard deviation of the hardness value can be seen from the plot. Although with the increase in peak current the deposition rate and coating thickness increases, however since this hardness measurement taken randomly at the cross section of the coating layer, almost uniform average hardness value obtained with a large standard deviation. The hardness value of pure TiC is around

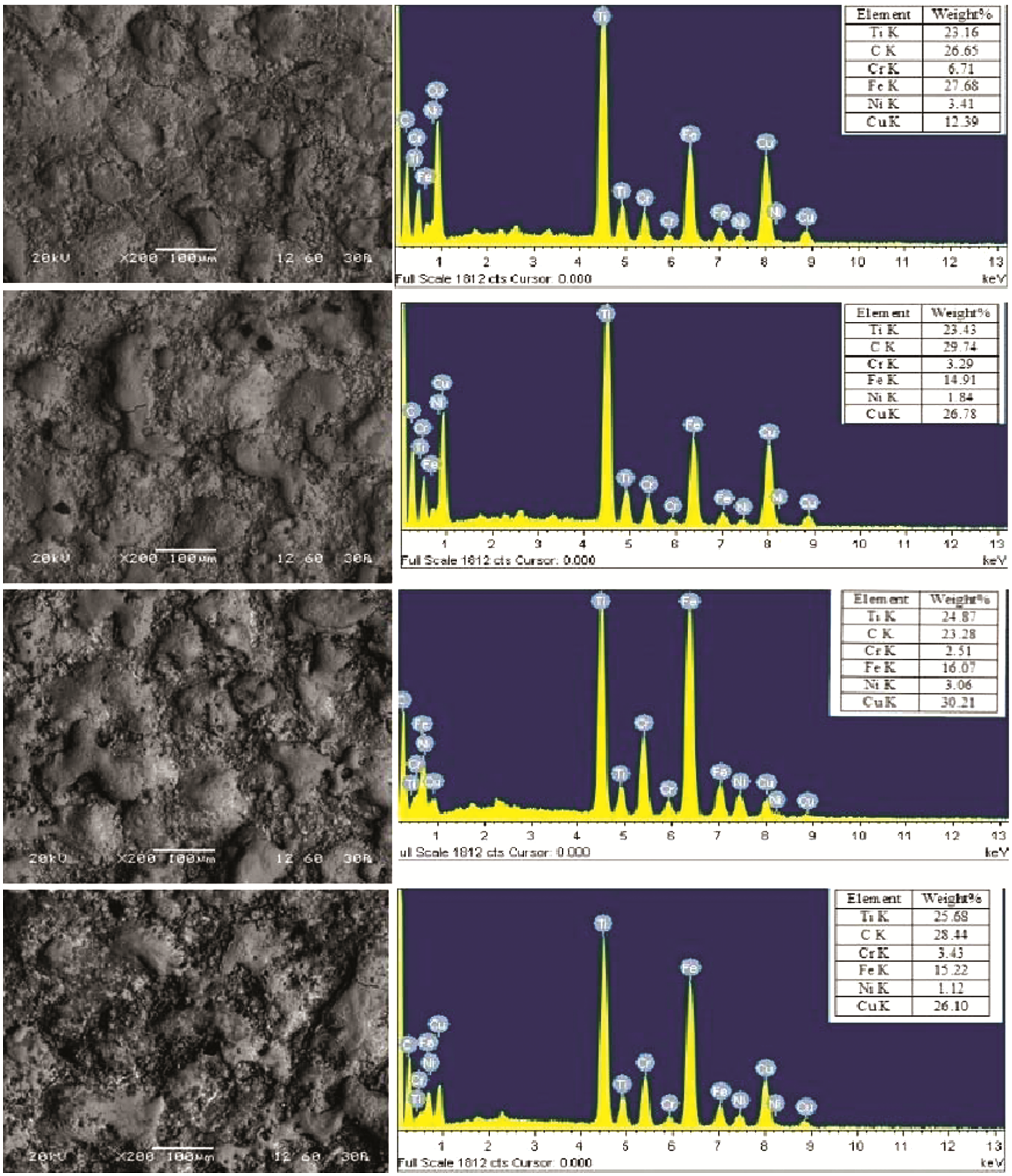

Fig. 8 — SEM images with EDS spectra of the TiC-Cu coated AISI 304 stainless steel produced at different peak current (a) 2 A, (b) 3 A, (c) 4 A and (d) 5 A.

3000 HV, with the deposition of TiC along with Cu and some other carbide particles those are formed during EDC process leads to a high value of hardness on the coating surfaces.

3.6 Sliding Wear Test

Sliding wear rate of the TiC coating produced on AISI 304 steel substrate was measured against WC-Co ball using a ball-on-disc type sliding wear tester. Wear rate was calculated in terms of weight loss per unit time (g/min) for the samples produced at the different current condition. Figure 10 shows the variation in wear rate for the coated samples produced at different peak current. The graph indicates that the wear rate of TiC coatings produced at different conditions is in the range of 2.33×10^{-5} to 3×10^{-5} g/min, which is almost 2 to 4 times time less than the wear rate of as received AISI 304 steel substrate under similar test condition. Careful observation of the plot also revealed that the wear rate decreases with the increase in peak current (up to 4 A) during the coating process. However, the wear rate agin increases for the coating produced with 5 A current. Sliding wear behaviour of any material usually correlated to its hardness value, as well as the thickness of the coating on the substrate surface. Although, the hardness value of the present TiC-Cu coating is almost uniform for different peak current applied during EDC process, however due to enhanced coating thickness, overall wear resistance of the coating increases when it produced at higher peak current. Essentially, during sliding wear test, with the sliding time, the WC-Co ball removes the coating material and penetrates towards the substrate. For the sample processed at low current, since the thickness of the coating is relatively less, after certain duration of wear test, the coating layer may remove out and the WC-Co ball comes in contact with the base material and wears out the softer base material at a faster rate. As a result, the overall wear rate of the coated sample becomes augmented. On the other hand, maintaining a higher coating thickness, sample processed at higher current condition exhibits lower wear rate. However, at maximum peak current (5 A), due to a higher deposition rate, a thicker layer of TiC-Cu coating produced on the substrate surface. Since in the EDC process, this coating material is deposited layer by layer, it is fairly possible that, after a certain value of coating thickness upper layer of the coating material not properly bonded and during sliding wear test, removed out rapidly.

Figure 11 shows the SEM image of wear track after the ball-on-disc sliding wear test performed on the TiC-Cu coated sample produced at 2 A current. The image indicates partial removal of the coating material and exposure of the substrate. From the high magnified image of the selected area, it is clearly seen that the coated layer removed due to the sliding action of WC-Co ball and substrate material uncovered. In contrast, for the sample produced at 5 A current it is seen that after the wear test the coating layer not removed from the substrate. Since the thickness of the coated layer is much higher for the sample produced at 5 A current than that of the sample produced at 2 A current, it is expected that removal of the coating layer is more prominent for the former.

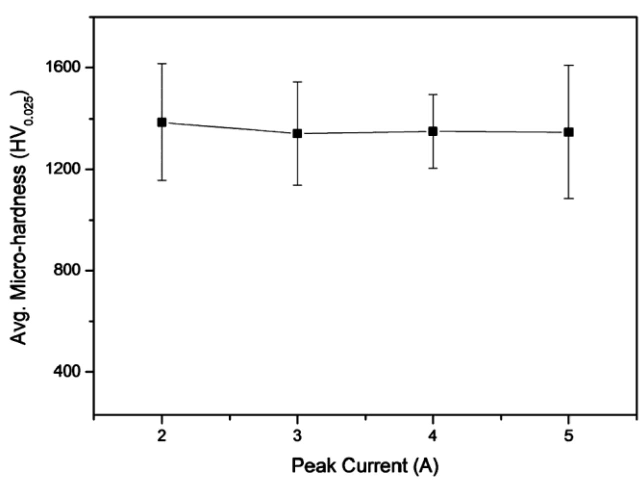

Fig. 9 — Variation of average micro-hardness value of the TiC-Cu coating produced with different peak current.

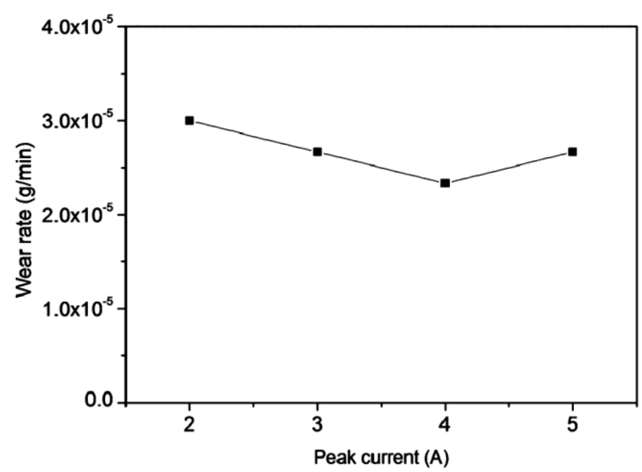

Fig. 10 — Wear rate of the TiC-Cu coating produced at different current condition by EDC process during ball-on-disc type sliding wear test.

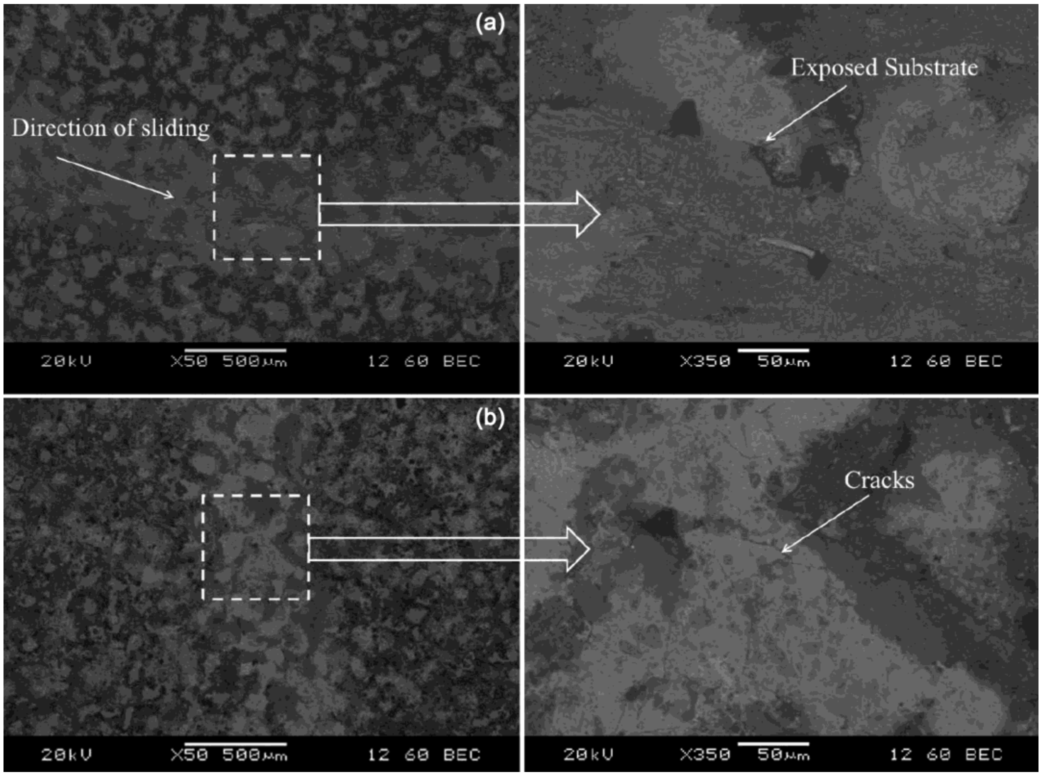

Fig. 11 — SEM images of wear track after the ball-on-disc sliding wear test performed on the TiC-Cu coated sample produced with (a) 2 A and (b) 5 A current.

4 Conclusions

From the present experimental investigation, it may be inferred that, with the help of powder compact tool electrode, using electro discharge coating (EDC) process a composite coating of TiC-Cu successfully deposited on AISI 304 steel substrate. The experimental result also revealed that, with the increase in peak current, the deposition rate of the coating increases almost linearly. Micro-hardness value of the deposited coating found almost six times higher than AISI 304 steel substrate, without any significant variation for the alteration of peak current. However, wear rate of the coating, during ball-on-disc sliding wear test reduces, due to the enhancement of the coating layer when peak current as a coating parameter increases up to a certain value. Wear rate of the coated samples was found in the range of 2.3 $\times10^{-5}$ to 3 $\times10^{-5}$ g/min, which is almost 2 to 4 times lower than the wear rate of AISI 304 steel substrate.

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