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Effect of Temperature and Gas Flux on the Mechanical Behavior of TiC Coating by Pulsed DC Plasma Enhanced Chemical Vapor Deposition

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ABSTRACT

There are many factors in coatings process which are effective in changing coatings characteristic such as voltages, duty cycle, pressure, temperatures and gas flux. In this paper, in plasma enhanced chemical vapor deposition (PECVD) technique, temperature and gas flux are two important variants which affect the coatings structure and mechanical properties. All TiC coating deposited on a hot work tool steel (H13) had a thickness of 2-3 micrometer. The investigation of TiC coatings composition and structure were done with the grazing incidence XRD, the FTIR (Fourier Transformation Infrared Spectroscopy) and the Field Emission Scanning Electron Microscopy (FE-SEM). The mechanical properties of the coatings, such as hardness, wear resistance and surface roughness were studied with Vickers hardness indentation; pin on disk wear tests and atomic force microscopy, respectively. The best mechanical properties such as a high hardness (3100 VHN), wear resistance and fracture toughness (11.3MPa. $m^{1/2}$) and low surface roughness (18 nm) were related to the coating which was deposited in 450°C.

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1. INTRODUCTION

Hard coatings such as titanium carbide (TiC) and titanium nitrocarbide (TiCN) have been used in various industries such as microelectronics and aerospace due to the unique properties such as high hardness and Young's modulus, low friction coefficient, proper resistance to corrosion and wear, good electrical and thermal conductivity and high melting temperature [1, 2].

Thin coatings can be deposited by different methods such as physical vapor and thermal chemical vapor techniques. In the first method, due to the low temperature, the coatings adhesion strength decrease and in the second method, due to the high temperature, grain growth occurs and the quality of the coatings properties reduces [3, 4]. However, plasma enhanced chemical vapor deposition (PECVD) can be a proper technique deposition of thin coatings on different substrates, due to possibility to achieve the same properties in lower temperatures. Controlling the composition and the thickness of coatings by adjusting plasma parameters is easy. Moreover, rotating of the complex sample is not necessary [4-6]. Thus, PECVD is used to deposit various coating for casting and extrusion mold even with irregular geometry and complexity [7]. In TiC coating deposition via PECVD technique, the temperature, plasma power, duty cycle, CH_4 and $TiCl_4$ gas flux are the fundamental parameters, because the amount of excess carbon is a very effective factor on the coating properties. It was shown by Stokes et al. in 1998, increasing the level of excess carbon, can decrease coating hardness [4, 8, 9].

It is noteworthy that different researches have been studied the ratio of $TiCl_4$ to CH_4 flux from 3 to 10 and until now there is a lack of published information on the fewer ratios. Moreover, there are not any researches that showed minimum temperature which is needed to deposit a adhesive layer to substrate. In this study, TiC coating was deposited by PECVD on the tool steel using

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CH_4 as the reactive gas. The flux ratio of CH_4 to $TiCl_4$ was changed from 1.5 to 6. The deposition temperature was increased from 450 °C to 490 °C to deposit coating with thickness of 2-3 micrometer. Then, characteristics of coating were identified.

2. MATERIALS AND METHODS

H13 hot work tool steel samples with the composition listed in Table 1 were heat treated to increase the hardness. Then, samples were prepared by grinding up to 2000. Then, they were cleaned with an alkali solution in an ultrasonic bath at 100°C for 10 minutes. In order to increase the adhesion of coatings to the substrate and to increase the load capacity of coatings, a plasma nitriding operation with parameters listed in Table 2 was performed.

In the plasma nitriding process, the flux ratio of hydrogen to nitrogen was fixed about 3 to prevent the white layer formation. TiC coatings were deposited in a PECVD reactor, using a $TiCl_4$-Ar-CH_4-H_2-N_2 gas mixture. The plasma was triggered by a pulsed-DC power supply. The total pressure in the reaction chamber was 8-10 mbar. Fixed flux of argon, hydrogen and titanium chloride and methane were 500, 1600, 50 and 1200 sccm, respectively. The duty cycle for all specimens was 33%. A negative bias voltage was fixed at 600-650 V during the process in a chamber, with the height and the diameter of 70×50 cm. The process time was 80 min. The substrate temperature was controlled by an auxiliary heating system in addition to the intrinsic sputtering effect. Other experimental conditions were listed in Table 3.

The thickness of layers was from 2 to 3 μm. In this paper, the grazing incidence X-ray diffraction (GIXRD) with test specifications (α = 2 ° and radiation 1.5418 Å: Cu Kα) was used. To identify the types of created bonds, Fourier transformation infrared spectroscopy (FTIR) was also used. The cross section of coatings was observed by the FE-SEM (Philips XL-30).

In order to determine the hardness and the fracture toughness of the samples in each situation, we perform first Vickers indentation for loads ranging from 0.1 to 1000 N and at least five indentations for each load. The hardness of the coatings was measured on hot work tool steel substrates. Wear test was also carried out using a WC–6% Co ball of 6-mm diameter at a linear speed of 0.1 m/s in 1000 meters distance with applied load of 10 N at room temperature (25º C) when trace diameter was 45 mm.

In order to study the topology of the surface of samples, the atomic force microscope (AFM: Nanoscope Version 3A, Digital Instruments) with Berkovich tip was used.

3. RESULTS AND DISSUSION

In this paper, the temperature and the ratio of effective gas flux has been studied as two variants. Other parameters such as voltage, duty cycle, and gas flux of nitrogen, argon, $TiCl_4$, and coating thickness were kept fixed. The GIXRD test results for samples are shown in Figure 1.

When the ratio amount of CH_4 to $TiCl_4$ was lower than a critical value (about 2), despite the presence of $TiCl_4$ in the reactor and high temperature, instead of TiC deposition, a black layer consisting of iron carbide was formed. The peaks appeared in 43.9° and 51.34° 2θ degree were related to austenitic iron carbide phase and the rest of the peaks were corresponding to the substrate. The standard TiC peaks were at 36.9, 42.6, 62, 74.48, and 78.18 degrees. Moreover, when the process temperature decreased to 420°C, the TiC coatings were not deposited in adhesive form and were peeled from the substrates surface.

From the GIXRD test results, these diffraction patterns show the orientation in the direction of crystalline planes (1 1 1), (2 0 0), (2 2 0), (3 1 1) and (2 2 2). The (2 0 0) plane was revealed to be the preferred structure and this plane is thermodynamically stable relative to the (2 2 0) and (1 1 1) planes [10].

The result test of FIIR is shown in Figure 2. In this pattern, three peaks were seen in degrees of 1044, 554 and 444 Cm^{-1} which was related to TiC phase. The peaks related to C-C, C-H and C-N bond have a peak in the range of 1500-3000 Cm^{-1}. However, there was no peak in our pattern.

The peak in the frequency range of 2900-3500 Cm^{-1} related to oxygen, hydrogen and nitrogen bond [11, 12] was also not observed in our test. When the flux ratio of $CH_4/TiCl_4$ was kept about 6, according to the bond energy in reflection spectroscopy [13], TiC coatings had cubic structure with a formula which can be $Ti_{14}C_{13}$. Therefore, the ratio of C to Ti atom was approximately 1 to 1.

TABLE 1. Chemical composition of the substrate

C	Si	Cr	Mo	V	Mn	Ni	Cu	Fe
0.45	0.69	5.72	1.14	2.23	0.27	0.15	0.24	balance

TABLE 2. Parameters in plasma nitriding process

Temp. (C°)	pressure (mbar)	time (min)	hydrogen (sccm)	nitrogen (sccm)	argon (sccm)
470	2	60	1600	500	500

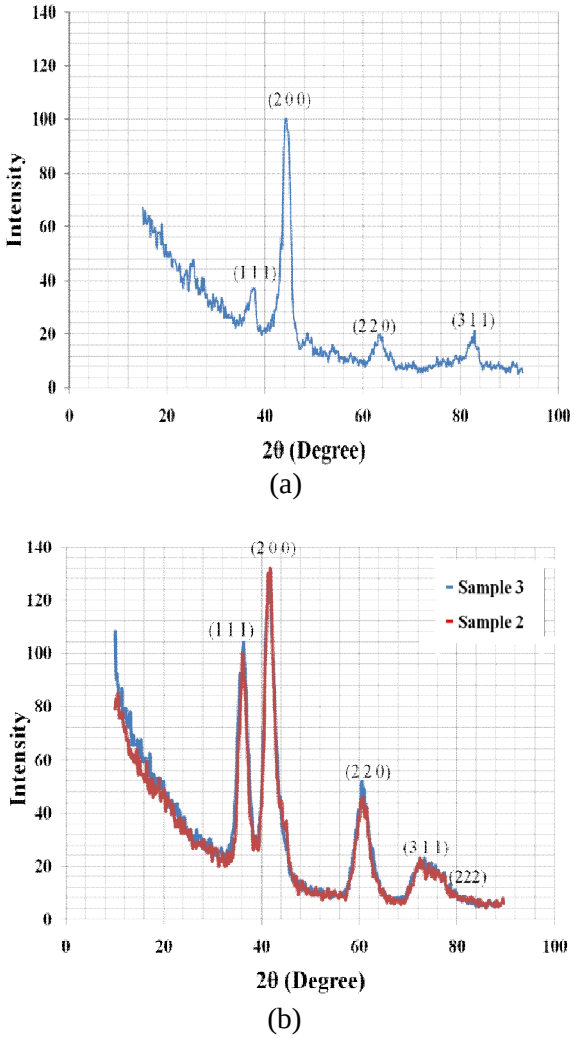


Figure 1. GIXRD test result for samples: (a) sample 1, (b) sample 2 ,3

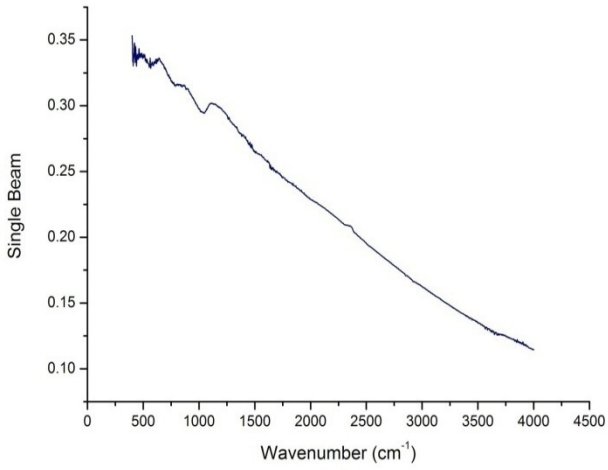


Figure 2. Reflective FTIR test result for sample 3

TABLE 3. Parameters in coating process

Sample no.	Methane(sccm)	Tem.(C°)	CH_4/ $TiCl_4$
1	80	490	1.6
2	100	470	2
3	120-300	450	2.4-6
4	100	450	2
5	120-300	470	2.4-6
6	120-300	420	2.4-6

The cross-section of coatings was observed by the FE-SEM, as shown in Figure 3. The total thickness of coatings was 2-3 µm, approximately. The investigation of the morphology in all coatings demonstrated a fine-grained structure. However, coatings which were deposited by the physical vapor deposition, had a columnar structure. Sample 3 seemed to have a more compact structure than Samle 5 due to the lower deposition temperature. A few spaces and defects were appeared in the coatings. As shown in Figure 3, it is demonstrated that when the substrate surface was not completely flat, coatings deposited even in down hills of the surface and a uniform coating was appeared. This is due to the high power throwing property in the PECVD process. This unique property is related only to the PECVD technique.

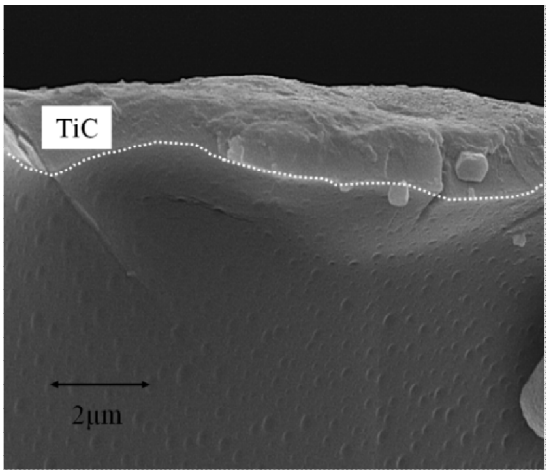
In this paper, the proper selection of ratio of effective gases prevented excess carbon to be precipitated as a separate phase [14]. Due to the Phase diagram, when the amount of carbon in the titanium network is 32-48.8%, TiC phase is formed as NaCl crystal structure. It is reported in a paper published in 1998 [9] that, when the flux ratio of $TiCl_4$ / CH_4 is more than 15, excess carbon will be precipitated in a separated phase. While the ratio of CH_4 to $TiCl_4$ gas flux be kept in proper level (more than 2 and less than 15), TiC phase with high hardness of 3000 VHN will be deposited. Microhardness changes will also just be depended on the deposition temperature. Microhardness test results are reported in Figure.4. The microhardness of sample 1 was 1200 VHN and the microhardness of other samples was about 3000 VHN. Sample of 3, 4 showed more microhardness than the sample of 2, 5 about (10%) due to the lower deposition temperature and more compact structure. When the deposition temperature was 450°C, the grain growth was minimized and the size of coating grain was decreased. As a result the microhardness was increased.

Notably, due to the papers published by other researchers [4, 10], the minimum temperature of TiC formation had been reported 470°C. It means that when this coating was deposited under the temperate of 470°C, the coatings was a non adhesive coating to the substrate. However, in this paper, the TiC coating coated at

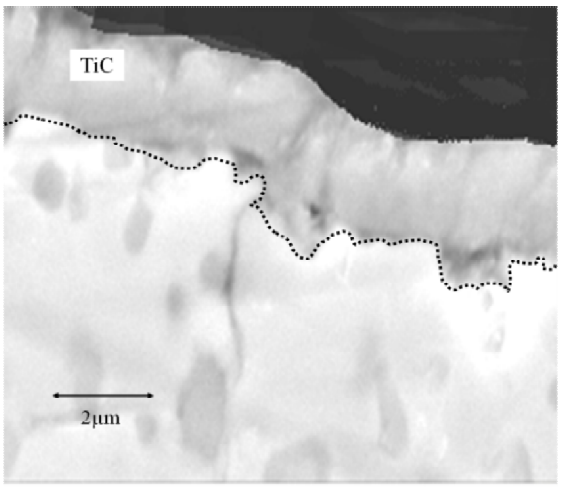
temperature of 450°C was a perfect and an adhesive coating with good mechanical properties. This result can reduce the cost of coating process. Furthermore, the substrate is not required to bear high temperature. According to our results, the flux ratio of CH_4 to $TiCl_4$ in the formation of TiC coating was more important than the temperature. Landcaster [15] proposed a simpler empirical wear formula as specific wear rate. It shows the resistance to abrasive wear:

$$k = \frac{V}{S.F} \quad (1)$$

In this formula, V is wear volume, S is wear distance and F is applied force. This wear coefficient k, with units of $m^3 .N^{-1} .m^{-1}$, has proven to be more useful for the comparison of the wear behavior of different materials than Archard's equation [15- 17].



(a)



(b)

Figure 3. FE-SEM cross section of samples: (a) sample 3 and (b) sample 5

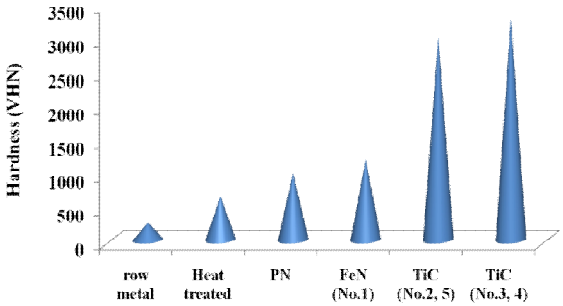


Figure 4. Harness result test

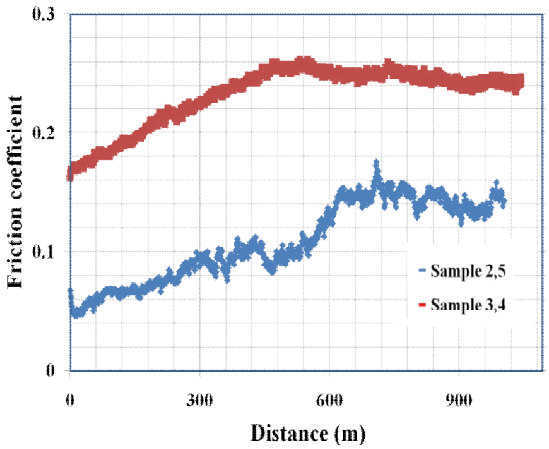


Figure 5. Friction coefficient verse wear distance diagram of samples

From the wear test result in Table 4, the wear rate of TiC coatings was not dependent on the temperature.

Changes in the coefficient of friction of the TiC coating determined during wear measurements are shown in Figure 5. For TiC coatings deposited in lower temperature, during the run-in 600 meter, the coefficient of friction was lower than 0.1 and then it increased to 0.15. Once the coefficient of friction reached to this value, it becames constant for the remaining distance. This result was in accordance with other papers independent from the method of deposition [8, 18]. When the deposition temperature increased to 470°C, coefficient of friction also increased and reached to the value of 0.2 to 0.3. Thus, the changes in deposition for 20°C caused to variation of friction coefficient about 50% due to the changing coating structure. However, the variation of the flux ratio could not lead to a change in the friction coefficient for TiC coating as reported by C. Jarms et al. [9]. During the running-in, owing to the interaction between the tip and the roughness of surface coatings, the friction coefficient (μ) rises. In the initial stage, the friction coefficient is controlled by the film roughness and the build-up of a transfer layer (tribolayer).

In the second stage, the friction and the wear are controlled by the nature of the tribolayer [19]. The micrographs of wear path for the samples are shown in Figure 6. The wear mechanism of samples was different from each other. The FeN coating was easily separated from the surface by the pin. The wear track for this coated sample was empty of debris and the wear edges were smooth. As TiC coating had a higher hardness than the iron carbide coating, the abrasive wear mechanism could be cutting. The total wear observed in the ceramic can be divided into two main broad categories such as (a) mechanically activated wear which includes abrasion, adhesion, plastic deformation, and fracture and (b) chemically activated wear also called tribochemical wear which includes diffusion or dissolution wear [20]. At higher speeds (to produce higher temperature), under dry or semi-dry conditions, chemical wear becomes the predominant mode of wear [21]. Thus, the speed of the mechanical mechanism is dominant mode of wear for all coated samples. The mechanical wear process is also divided into three stages for single layer of coating of TiC: crack nucleation, crack propagation along a given direction under applied force and forming of wear debris and damages on the rubbing surface. The image for TiC coating (Figure 6) shows that the wear track was full of debris. Debris was observed to pile up along the outer edges of the wear track. They were small near the wear edges but the debris size increased when we were far from the wear edges and near to the center. The Wear areas were separated by broken lines from other areas and this due to the compact structure. When the deposition temperature was increased to 470°C, the debris size in wear track was increased and gathered to holes but abrasion boundary was separated by curved lines. Due to the increasing of the debris size in wear track and formation of holes in TiC coated with higher deposition temperature, the wear rate increased.

Figure 7 shows AFM images for coatings that were analyzed in 2×2 μm. Lighter areas of AFM images represent those regions with a higher height. Their surface roughness is summarized in Table 4.

Ra is defined as the mean value of the surface height relative to the center plane and *RMS* is the root mean square roughness profile of the surface height within the scanned area. The surface roughness was about 21 nm for sample 2, 5. However, the surface roughness of sample 3, 4 with lower temperature was lower and was about 18 nm. In other papers, the amount of TiC coating surface roughness was reported less than 10 nm which this may be related to the higher temperature (500°C) with different mechanism [5, 22, 23]. The effect of temperature on the grain growth was very severe with respect to the gas flux. In this paper, the AFM images and Ra and RMS values showed that a lower deposition temperature and compact structure decreased the surface roughness, because the grain growth occurs when the deposition temperature increased.

TABLE 4. AFM, farcture toughness and wear result test

Sample no.	R_a (nm)	RMS (nm)	Toughness ($MPa.m^{1/2}$)	wear rate ($(m^3/Nm)* 10^{-13}$)
1	25±1	31±1	5.5	10.1
2,5	21±1	27±1	10	3.70
3,4	18±1	22±1	11.3	3.25

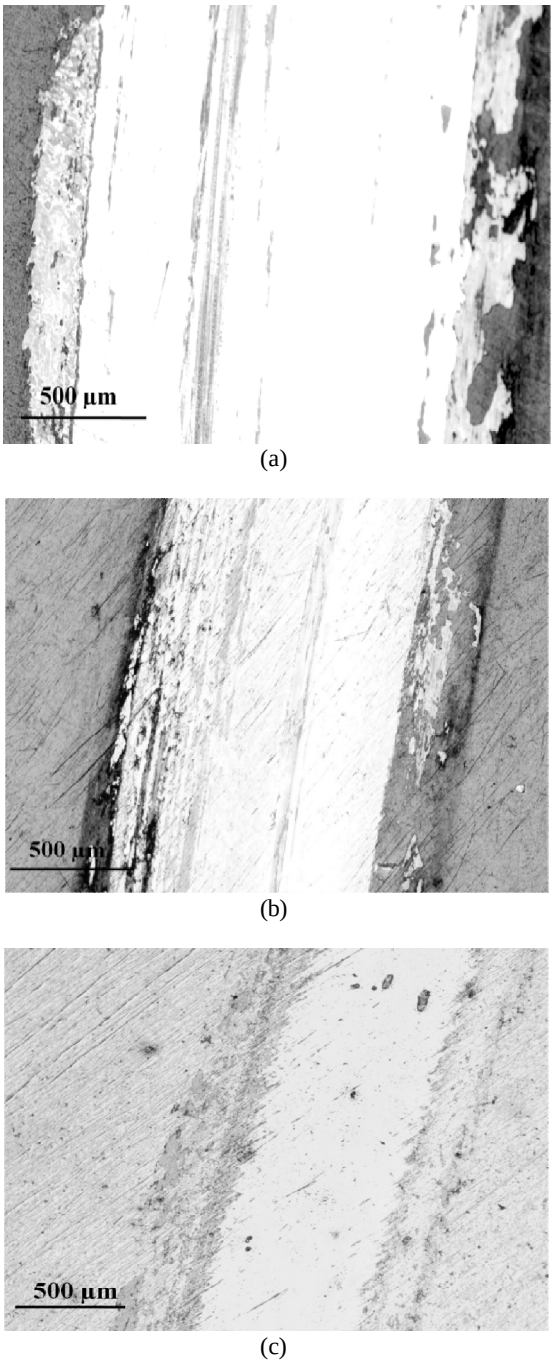
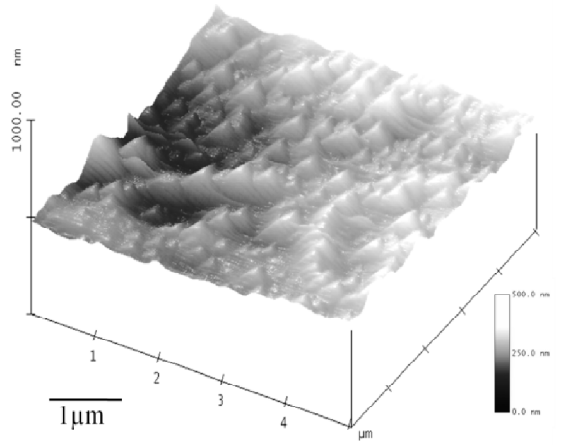


Figure 6. Wear path micrographs of samples: (a) sample 1, (b) sample 3, (c) sample 5

The surface roughness was also independent of flux ratio of important gas when this ratio is proper to deposit TiC coating. Higher coefficient of friction was seen for TiC coatings with high deposition temperature. This result was related to the lower surface roughness and grain growth. For thin films, the fracture toughness measurement remains difficult due to the thickness limitation. However, until now, unlike bulk materials, there is neither standard procedure nor commonly accepted methodology to follow [24]. In such cases, the indentations fracture (IF) technique might be considered as the most suitable technique [25]. The fracture toughness has been obtained using Equation (2) as it is appropriate for the ceramic coating (especially when the crack mode is Palmqvist cracking) [26].

$$K_C = 0.0889 \left(\frac{H.P}{4l}\right)^{1/2} \quad (2)$$

where, H is the Vickers hardness, P is the applied load, and l is the radial crack length.



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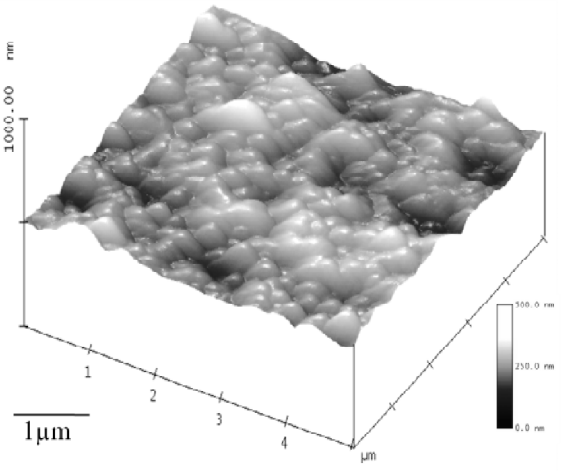

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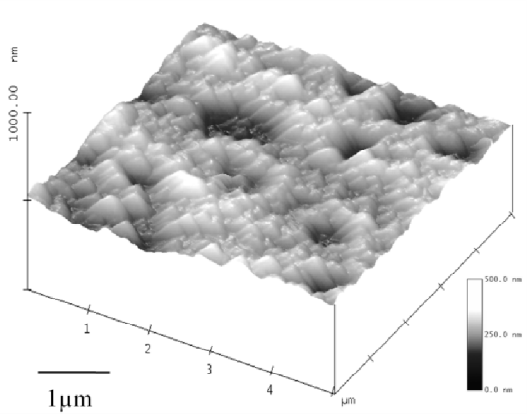

(c)

Figure 7. Topography of surface samples: (a) sample 1, (b) sample 3, (c) sample 5

As the fracture toughness depended on the applied force, the slopes of the load (P)-crack length ($l^{1/2}$) diagram were considered for comparing coatings fracture toughness. As shown in Table 4, the sample 3 had maximum fracture toughness (11.3 MPa.m$^{1/2}$) due to the higher hardness than sample 5.

4. CONCLUSION

The result showed that, when TiC coating was deposited in 420°C, the coatings were not adhesive to substrate. When the deposition temperature increased to 450°C, adhesive coatings were deposited on the H13 hot work tool steel. The temperature had also important role in coating characteristic changes like structure, hardness, wear resistance, friction coefficient, and surface roughness. Moreover, selecting the correct flux ratio of gases (2.4-6) had an effect on creating uniform TiC coatings. Hardness of TiC coating was dependent on the temperature due to compact structure. However, it is independent from flux ratio of gases. On the other hand, wear mechanism was changed abruptly when the deposition temperature changed from 450°C to 470°C. Minimum friction coefficient and surface roughness and higher hardness, fracture toughness and wear resistance for TiC coating with deposition temperature of 450°C was observed.

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Wear.

چکیده

بسیاری از فاکتورها در فرایند پوشش‌دهی همانند ولتاژ، سیکل کاری، فشار، دما و فلاکس گازها بر روی تغییر خصوصیات پوشش‌ها تاثیرگذار هستند. در این مقاله در تکنیک ترسیب از فاز بخار شیمیایی به کمک پلاسما، دما و فلاکس گازی دو پارامتری مهمی هستند که بر روی خصوصیات ساختاری و مکانیکی پوشش تأثیرگذار می‌باشند. تمامی پوشش‌های کاربید تیتانیم بر روی فولاد ابزار با ضخامت 3-2 میکرومتر ترسیب شد. بررسی ترکیب و ساختار پوشش کاربید تیتانیوم با روش‌های پراش اشعه ایکس، اسپکتروسکوپی عبوری مادون قرمز فوریه و میکروسکوب الکترونی روبشی گسیل میدانی مورد بررسی قرار گرفت. خواص مکانیکی پوشش مانند سختی، مقاومت به سایش، چقرمگی شکست و زبری سطحی به ترتیب با دستگاه فرورونده ویکرز، دستگاه سایش پین روی دیسک و میکروسکوپ نیروی اتمی مطالعه شده است. بهترین خواص مکانیکی مانند سختی بالا (3100 ویکرز)، مقاومت به سایش و چقرمگی شکست بالا (11/3مگاپاسکال) و زبری سطحی کم (18 نانومتر)، به پوششی مربوط است که دمای فرایند آن 450 درجه سانتی‌گراد باشد.

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