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TiN coating on wall of holes and stitches by pulsed DC plasma enhanced CVD ①

MA Sheng-li(马胜利)[1, 2], XU Ke-wei(徐可为)[1], JIE Wan-qi(介万奇)[2]
(1. State Key Laboratory for Mechanical Behavior of Materials, Xi′an Jiaotong University, Xi′an 710049, China;
2. State Key Laboratory for Solidification Processing, Northwestern Polytechnical University, Xi′an 710072, China)

Abstract: TiN coating samples with narrow-stitch or deep-hole of different sizes and real dies with complex shape were processed by a larger-scale pulsed plasma enhanced CVD(PECVD) reactor. Scanning electron microscopy, optical microscopy, Vicker′s hardness and interfacial adhesion tests were conducted to find the relation between the microstructure and properties of TiN coating on a flat and an inner surface. The results indicate that the inner-wall of holes (d > 2 mm) and inner surface of narrow-stitches (d > 3 mm) can be coated with the aid of pulsed DC plasma in an industrial-scale reactor. The quality of coatings on different surfaces is almost the same. The coating was applied to aluminum extrusion mould, and the mould life was increased at least by one time.

Key words: pulsed DC plasma; moulds; PECVD; TiN coatings

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1 INTRODUCTION

Hard coatings deposited by plasma enhanced chemical vapor deposition have obtained more interest recently. So far, wear and corrosion-resistant coatings such as TiN, TiCN, TiC, TiBN, TiAlN can be deposited on various substrates by PECVD[1-5]. Heim et al[6] investigated the deposition of TiAlN and TiAlNC coatings on tool steel and hard metal substrate in industrial PECVD-plant. Their results showed a fine morphology and high microhardness up to 3 000 Hv as well as strong adhesion of 30－40 N measured in scratch test compared with PVD TiN. In recent years, the synthesis of hard coatings can also be performed using CVD enhanced by plasma generated by microwave source or radio frequency self-biasing voltage[7, 8]. However, pulsed DC voltage for plasma generation during the CVD process has an advantage of suppressing abnormal arcs that could damage workpieces. Another important advantage of pulsed DC plasma in CVD is the possible homogeneous deposition on the inner-wall of small-hole[9]. Therefore, the relation between process parameters and the coatings properties has been investigated for a long time to understand the deposition process[10-12]. However, the coatings were often fabricated in laboratory scale reactors and the samples were usually made with a simple shape. It was questioned whether the results from a small reactor or a sample with simple shape could be transferred to a large-scale reactor or to a real component with complex geometry. This is of great interest for industrial application.

In this paper, samples with narrow-stitch or deep hole of different sizes and real dies with complex shape are coated with TiN deposited by a pulsed DC plasma CVD in industrial-scale reactor, and the quality of coatings on different surface is demonstrated.

2 EXPERIMENTAL

Schematic diagram of the industrial-scale pulsed DC plasma enhanced CVD reactor with size of d450 mm × 600 mm, designed and manufactured by the authors, is shown in Fig. 1. Detailed information about the equipment and its deposition

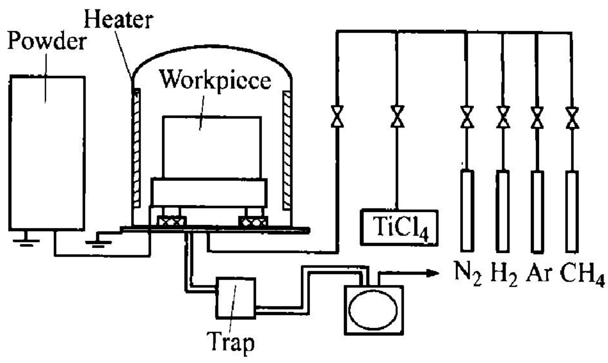

Fig. 1 Schematic diagram of PECVD system

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Correspondence: MA Sheng-li, PhD; Tel: +86-29-2668614; Fax: +86-29-3237910; E-mail: slma@mail.xjtu.edu.cn

Table 1 Plasma nitriding and PECVD process parameters for TiN coating

Process	Pulsed voltage/ V	Pulse-on time/ μs	Pulse-off time/ μs	Temperature/ ℃	Pressure/ Pa	Gas flow/ (mL·min⁻¹)				Time/ h
						N_2	H_2	Ar	$TiCl_4$	
Plasma nitriding	1 000	25	25	520	1 000	200	600	–	–	2
PECVD TiN	650	25	25	520	350	350	600	70	75	2

process can be found in Ref. [13]. AISI H13 steel, a material of mould and die, was quenched and tempered as a substrate with a hardness of 45HRC. The steel samples with narrow-stitch or deep-hole of different sizes were designed in this study. The width of the stitches and the diameter of the holes were 1 mm, 2 mm, 3 mm, 4 mm, 5 mm, 6 mm and d1 mm, d2 mm, d3 mm, d4 mm, respectively. The depth of the stitch and the hole was fixed at 18 mm and 10 mm. A real aluminum extrusion mould (d240 mm × 50 mm) made of H13 steel was also selected as sample.

Hard TiN coatings were deposited on the in-situ nitrided steel samples and the aluminum extrusion mould. The main plasma nitriding parameters and PECVD deposition parameters used are shown in Table 1. Scanning electron microscopy (S2700) was used to examine the surface morphology and cross-section microstructure of TiN coatings. The Vickers's hardness was determined on the surface of the coating using a load of 0.5 N. Interfacial adhesion tests for evaluation of the bonding between the coating and the substrate were conducted in two methods. The first was indentation adhesion test using a modified Rockwell hardness tester[13]. The indentation force was applied with a continuous load, and the adhesion was denoted by a critical load corresponding to the initiation of coating spallation monitored by acoustic emission. The second was rolling contact fatigue test[14]. A number of balls in bearing race were pressed on the coated sample. The test was interrupted periodically to check failure of the coatings. A plot of load vs number of cycles can be obtained. The shear stress aptitude at the interface, after 5×10^6 cycles with a detachment area of less than 5% of the total coating, was taken as the measure of the adhesion strength.

3 RESULTS AND DISCUSSION

The results show a homogeneous deposition for narrow-stitch at a ratio of width to depth of more than 3∶18 and for deep-hole with diameter, depth of more than d2 mm × 10 mm. It can be seen in Fig. 2 that the micrographs of TiN coatings, deposited on outer-surface and inner-surface, are almost identical, the thickness (1.3 μm) of the coating is the same. These results suggest that the complex geometry with narrow-stitch (> 3 mm in width) or deep hole (> 2 mm in diameter) can be successfully deposited with TiN using the novel pulsed DC PECVD technology.

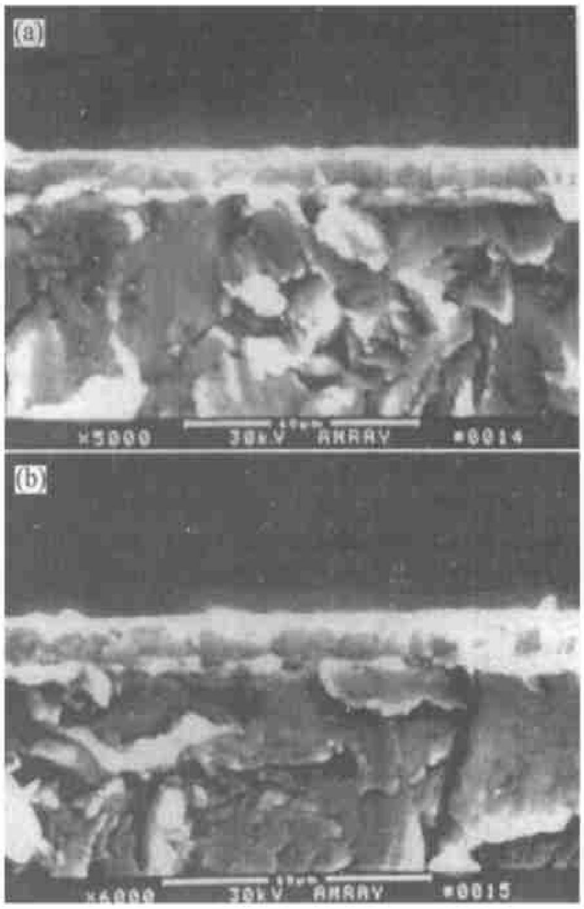

Fig. 2 Cross-sectional microstructures of TiN coatings deposited on outer (a) and inner (b) surface

Fig. 3 shows the surface hardness of TiN coatings at inner and outer surface on nitrided H13.

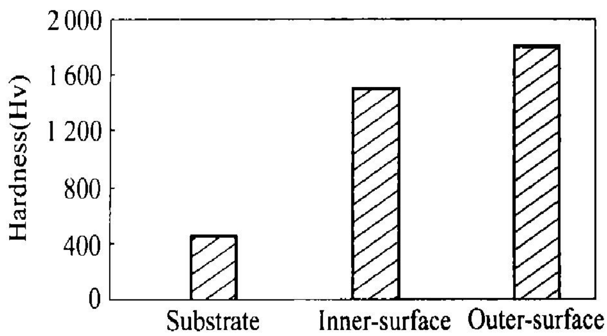

Fig. 3 Surface hardness of plasma nitrided and PECVD TiN coated H13 steel

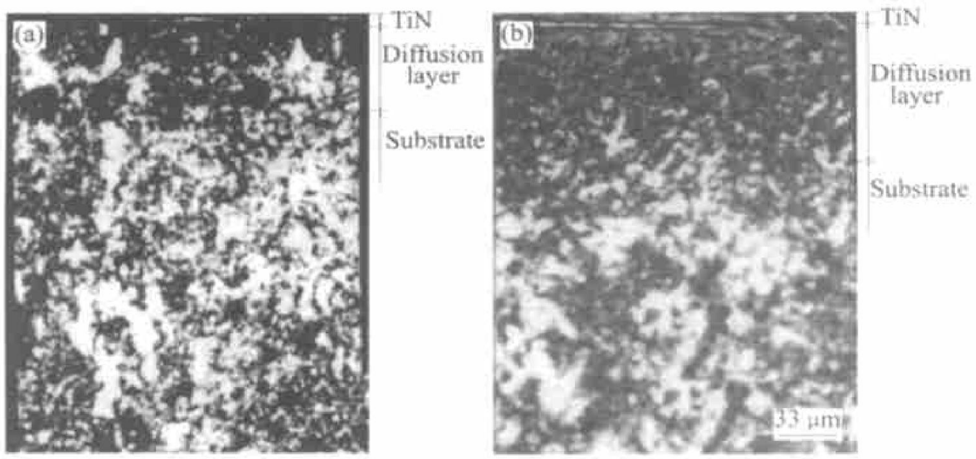

Fig. 4 Cross-sectional microstructure of plasma nitrided and PECVD TiN coated at inner (a) and outer (b) surface of H13 samples

Hardness of the coatings is increased remarkably compared to the substrate. The small hardness difference between the outer and the inner surfaces could be resulted from the nitrided layer. The nitrided layer at the inner-surface is darker than that at outer-surface as seen in Fig. 4, possibly due to the weak plasma intensity within the stitch or the hole.

However, Fig. 5 indicates that the interfacial adhesion strength measured by both indentation test and rolling contact fatigue test have high values. A small decrease of F_c value for the inner-surface coating is probably due to the low load-carrying capacity of the thin nitrided layer. It has been showed that the $\Delta\tau_c$ in the rolling fatigue test is one of the best parameters for characterization of the bond strength of hard coatings and F_c in the indentation test is a measure of the load-carrying capacity of coating/substrate system[13].

An aluminum extrusion mould was first nitrided and than coated with TiN by the pulsed DC plasma treatment. Pictures of the coated mould are shown in Fig. 6. The mould was tested under the condition of working temperature of 400 − 500 ℃ and working pressure of 200 − 400 Pa when aluminum alloy 6063 was extruded . The result shows that the service life of the mould was increased at least by one time.

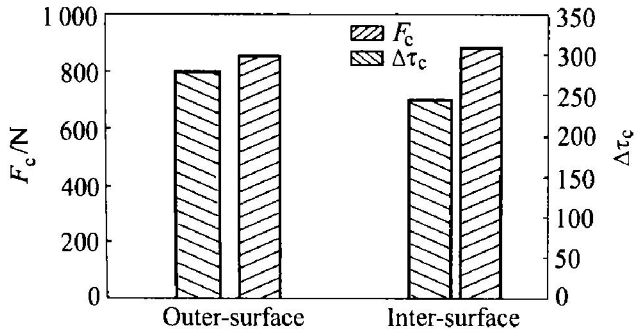

Fig. 5 Interfacial adhesion comparison between inner and outer surface

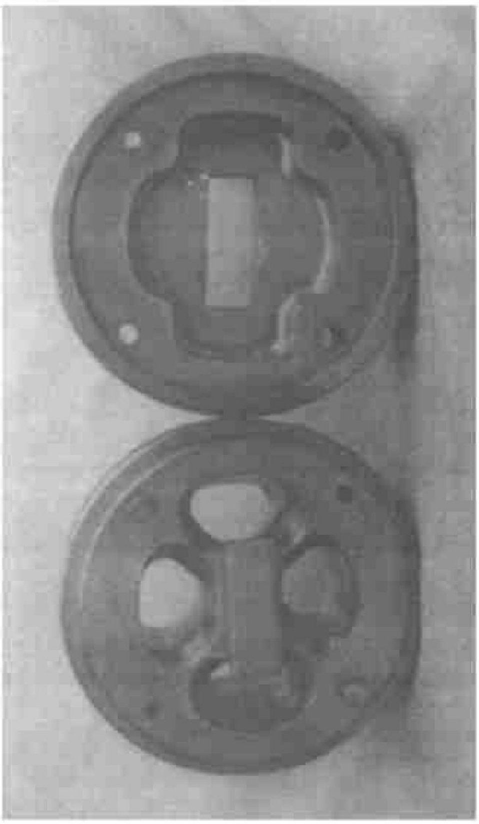

Fig. 6 Pictures of duplex treated aluminum extrusion mould by plasma nitriding and plasma enhanced CVD

4 CONCLUSIONS

The inner-wall of holes (d> 2 mm) and the inner surface of narrow-stitch (> 3 mm) can be coated with the aid of pulsed DC plasma in an industrial-scale reactor. The quality of the coatings on different surfaces are almost the same. The pulsed DC plasma enhanced CVD has a great potential in applying to die and mould industry. The duplex processing of the first plasma nitrided and then coated with TiN by plasma enhanced CVD can double the service life of aluminum extrusion mould.

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