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ARTICLE

Effect of Multilayered Structure on Properties of Ti/TiN Coating

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Abstract: Metal nitride hard coatings, such as TiN and TiAlN are widely used to protect materials because of their higher hardness and wear properties. However, these coatings always contain a high degree of internal stress which could arouse adhesion problems. It is hard to synthesize monolayer TiN or TiAlN coatings thicker than 10 μm by PVD (physical vapor deposition). The multilayer composite structure, offering efficient means of controlling residual stress, is a successful way to synthesize thicker hard coatings. In the present study, a series of multilayer composite Ti/TiN coatings with different composite periods were synthesized by plasma enhanced ion plating and the effects of multilayered structure mechanical properties of the coating was studied. The result shows that the mechanical properties of the coating are strengthened with increasing the periods of composite Ti/TiN layer. The micro hardness (HV0.25) can reach to about 27 500 MPa and better toughness and higher thickness (>50 μm) are obtained. The toughness of the coating is almost directly proportional to hardness. Meanwhile, the tribological performance of the coating is improved with a lower dry friction coefficient (about 0.35) and higher wear resistance by alternating 48 periods of Ti/TiN layer. However, the bonding strength of the coating is weakened with further increasing the period of Ti/TiN layer due to the weak interface of coating and substrate.

Key words: Ti/TiN coating; multilayered structure; micro hardness; tribological performance; toughness

Metal nitride hard coatings, such as TiN, TiAlN, have been widely used to protect the component because of their higher hardness, better tribological properties and oxidation resistance[1-6]. However, the applicability of these hard coatings has been constrained by limitations of the coating thickness. Hard coatings, synthesized by PVD technology always contains high degree residual stress owing to lattice distortion and thermal mismatch effects which is bad for ceramic coating's fatigue life, bonding strength and so on[7,8]. Therefore, it is hard to synthesized monolayer PVD hard coating thicker than 10 μm without adhesion problems. Many ways were proposed to minimize or eliminate that residual stress. Researchers[9,10] have revealed that ductile titanium interlayer in multilayer Ti/TiN coatings could accommodate the TiN internal stresses and was necessary to the residual stresses relaxation. Following this guideline, thick hybrid metal/ceramic coatings were produced by Ti interlayer accommodating the TiN residual stress[11-14]. According to their researches, a favorable residual stress gradient could be formed by that multilayered structure. Besides, by adjusting Ti interlayer's thickness, a significant improvement of mechanical properties such as wear resistance, toughness and adhesion was achieved. These improvements were related to some (or a combination) of the following mechanisms: crack deflection duo to weak interface, crack-tip shielding by plastic deformation in combination with strong interfaces, crack deflection due to differences in the elastic modulus of the individual layer materials, compared to monolayer TiN coating[15-18].

Although a lot of research pointed out that the multilayered structure was an effective way to improve PVD hard coating's thickness, and thick hard coatings have been synthesized with better performance, any deep study of the influence of periods of Ti/TiN layer on their mechanical properties has not been carried out. In the present research, a series of multilayered thick Ti/TiN hard coating with various periods was synthesized and the effect of periods on coating's hardness,

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toughness, adhesion and tribological performance was studied in detail.

1 Experiment

Multilayered Ti/TiN hard coatings with various periods were deposited onto TC4 alloys and Si (100) substrates by plasma-enhanced ion plating system using high purity Ti symmetrical targets at 300 °C. Technical principle and advantage of the plasma-enhanced effect has been shown in our earlier publication [19].

The TC4 and Si (100) samples were polished to mirror, washed in distilled water, dried in warm air and fixed on a turntable between the targets and the target-to-substrate distance was fixed at 50 mm as usual. Prior to deposition, the system was evacuated to a vacuum pressure of 1×10^{-4} Pa and Ti ion cleaning on samples at bias voltage of –1100 V was carried out to eliminate contamination and improve adhesion. Then a series of multilayered Ti/TiN coatings with various periods were deposited on the substrates at bias voltage of –100 V. In this study, the thickness of Ti interlayer was set at 100 nm and that of TiN was set at 500 nm. At the same time, a monolayer TiN was deposited in order to make comparison.

A scanning electron microscopy operated at 20 kV (JSM-6700F, SEM) was employed to study the coatings' cross-sectional morphology and thickness. The hardness was evaluated by micro indention measurement (MH-5 digital Vickers micro hardness tester) at load of 25 g and dwell time of 10 s. The adhesion was quantitatively evaluated using WS-97 automatism loading scratch instrument at a scratch speed of 40 N/min. And the tribological performance was characterized by HT-500 ball-on-disk abrasion apparatus. The test was carried out at room temperature and lasted about 40 min with Si_3N_4 ball (6 mm in diameter) as the frictional pair, extra load of 5 N and disk speed of 560 r/min.

According to Lawn, Evans and Marshall[20], the toughness of the coating bears the following relationship with the length of cracks and applied load using micro indention measurements.

$$K_{IC} = \delta\left(E/H\right)^{1/2}\left(P/C^{3/2}\right) \quad (1)$$

Where P is the applied indentation load, E and H are the elastic modulus and hardness of the film, respectively. C is the length of cracks, and δ is an empirical constant which depends on the geometry of the indenter [21,22].

2 Results and Discussion

2.1 Cross-sectional morphologies of Ti/TiN coatings

A typical columnar structure with micro cracks and through-coating pinholes can be seen clearly from Fig.1a. That structure often has a high degree interfacial residual stress which is bad for thick coating synthesis. With ductile Ti interlayer's accommodation, a multilayered structure can be formed which is necessary to the residual stress relaxation. As shown in Fig.1b, 1c and 1d, 6 periods, 24 periods and about 150 periods of multilayer composite Ti/TiN coatings were deposited and the maximum thickness could reach to over 100 μm without adhesion problems. It is confirmed that hard PVD coatings with high thickness can be achieved by adopting the multilayered composite structure.

The high thickness of multilayered composite Ti/TiN coating can be attributed to the modulated function of Ti interlayer. Firstly, the re-nucleation of the TiN phase is promoted by Ti interlayer and so the growth of TiN columnar crystals is restrained. As a result, the defects in the columnar structure

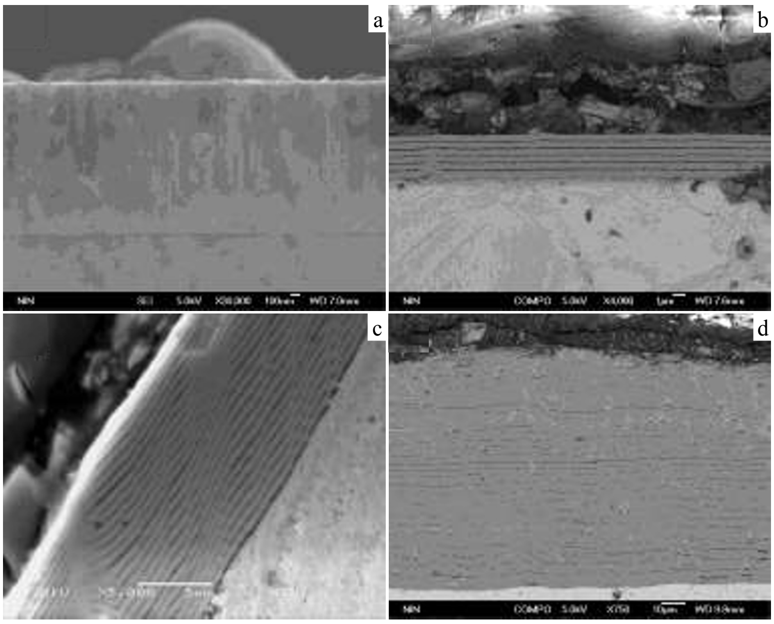

Fig.1 Cross-sectional morphologies of Ti/TiN coatings with different composite structures: (a) single TiN coating, (b) Ti/TiN coating with 6 periods, (c) Ti/TiN coating with 24 periods, and (d) Ti/TiN coating with 150 periods