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Microstructures of TiN, TiAlN and TiAlVN coatings on AISI M2 steel deposited by magnetron reactive sputtering

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Abstract: In order to study the effect of the microstructure with Al and V added TiN coatings, TiN, TiAlN and TiAlVN coatings were deposited on AISI M2 high-speed steels by magnetron reactive sputtering. The microstructures of all the coatings were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The results indicate that the addition of Al into TiN coatings reduces their lattice constant, but a further addition of V into TiAlN coatings increases their lattice constant. Moreover, the growth morphologies for TiN, TiAlN, and TiAlVN indicate that adding Al and V has a tendency to improve the columnar structure. The (111) and (200) orientations of TiN, TiAlN, and TiAlVN are identified. The $\varepsilon(Fe_3N-Fe_2N)$ phase occurs because a small amount of Fe is present in the coatings. The interlayers of TiAlN and TiAlVN have the preferred $(01\bar{1}0)$ orientation. The texture (columnar) structure of the (111) and (200) orientations is observed in the TiAlN and TiAlVN coatings. An orientation relationship of $(01\bar{1}0)_{\alpha-Ti}//(110)_{T.M}$ occurs between the interlayer and tempered martensite (T.M) in TiAlVN.
Key words: coating; TiAlVN coating; sputtering yield; crystallographic relationship

1 Introduction

Nitride coatings of transition metals are commonly used in the tribological [1−3], decorative, and biomedical applications [4] because of their high hardness, high abrasion resistance and attractive golden-yellow color. However, TiN coatings oxidize rapidly at temperatures above 500 °C and form a layer of rutile TiO_2, which determines the limit of their high-temperature applications. The large difference between the molar volumes of TiO_2 and TiN causes stress in the oxide. Its magnitude increases with increasing oxide thickness till the critical thickness, which occurs when the oxide spalls and the unoxidized nitride below is exposed to further oxidation. Further improvements in properties can be realized in multicomponent Ti-based nitride coatings. For example, TiAlN coatings on tool steels are demonstrated to have excellent high-temperature stability and abrasion resistance [5−8]. TiAlN coatings have been deposited on tool steels by magnetron sputter ion plating [9], and physical vapor deposition (PVD) [10−12].

Although TiAlN hard coatings show successful performance because of their excellent wear and thermal resistance compared with TiN coatings, a low coefficient of friction or self-lubrication properties for high-speed and dry abrasive applications is required. The addition of V as a TiAlVN multicomponent or TiAlN/VN multilayer structure [13−16] is becoming an alternate option for high-temperature use. However, the effects of V addition on the basic crystalline structure and growth morphology are not investigated although some researches about the applications of TiAlVN coatings are found.

In this study, TiN, TiAlN and TiAlVN coatings were deposited by magnetron reactively sputtered on steels from targets of pure Ti, Ti−6Al (6%) and Ti−6Al−4V (6% and 4%). The effects of Al and V on the microstructure of TiN coatings were investigated.

2 Experimental

2.1 Deposition process

AISI M2 high-speed steels used as substrates were quenched and tempered before deposition. The hardness

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of the tempered M2 is HV 780 (HRC 63). Deposition processes were undertaken using the sputtering system of CSU550-1, Shengyang Piotech Co., Ltd.. Three materials were used as targets in this study: Ti, Ti−6Al and Ti−6Al−4V. The discharge power was controlled to be 220 W and the substrate temperature was kept at 300 °C. The argon and nitrogen flow rates during deposition were 28 and 3 mL/min, respectively, with a specific ratio of 9.3. The working pressure of the chamber was maintained at 0.8 Pa. Pre-sputtering was performed by plasma for 15 min to clean the target surface. The deposition duration for growing the films to approximately 2 μm in thickness was 1 h, but the metallic interlayer was grown within the initial 5 min.

2.2 Micro-Vickers hardness testing

The surface hardness of the nitride layers was measured using a micro-Vickers hardness tester with 0.147 N load and 10 s load-holding time (Mitutoyo, MVK-H1).

2.3 Microstructure characterization

The cross-sections of each nitride film were examined using an SEM (JEOL 6400) through backscattered electron images (BEI) and secondary electron images (SEI) to identify their columnar structure. The microstructure of the coatings was analyzed by X-ray diffraction (XRD, Siemens D5000) using Cu K_α radiation operated at 30 kV and 25 mA, with a scanning speed of 1.5 (°)/min. Moreover, the microstructure was investigated by a transmission electron microscope (TEM, JEOL JEM−3010, operating at 300 kV).

3 Results

3.1 Micro-Vickers hardness

The micro-Vickers hardnesses are HV1560, HV1640 and HV1690 for TiN, TiAlN and TiAlVN coating on M2, respectively.

3.2 XRD

The major phases of the nitride coatings on M2 identified by XRD are shown in Fig. 1. Figure 1(a) shows the strong (111) and (200) TiN peaks and several weak peaks, which were possibly caused by the ε(Fe_3N−Fe_2N) phase or Fe by Joint Committee on Powder Diffraction Standards (JCPDS) file 3−0924. Moreover, the (111) and (200) peaks are also present in TiAlVN/M2 (Fig. 1(c)), indicating that Al and V may dissolve in TiN. The most prominent peak of TiAlN/M2 in Fig. 1(b) may be contributed by the ε (Fe_3N−Fe_2N) phase according to the JCPDS files. The strong peak combined with the numerous related weak peaks may have occurred because of the Fe-containing phases in the nitride coatings, which were not observed in previous studies of TiAlN/M2.

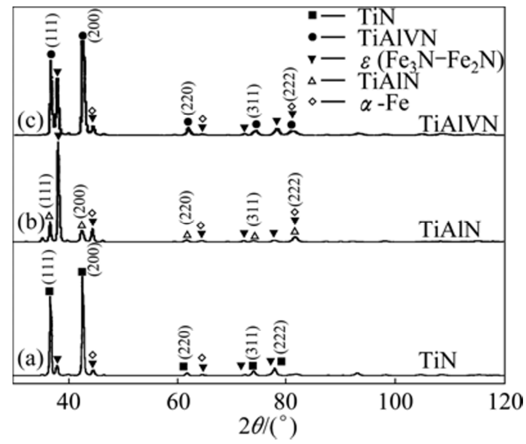

Fig. 1 XRD patterns of TiN/M2, TiAlN/M2 and TiAlVN/M2

Furthermore, the lattice constants were measured by extrapolation. The values of the lattice constant calculated from the diffraction peaks are plotted against $\cos^2\theta/\sin\theta+\cos^2\theta/\theta$, and the straight line is extrapolated to zero; thus, the exact lattice constant (a_0) can be obtained. Data for the TiN, TiAlN, and TiAlVN are shown in Fig. 2. The aluminum-containing nitride films evidently have a smaller lattice constant, but the addition of vanadium to TiAlN increases the lattice constant of the TiAlN coatings.

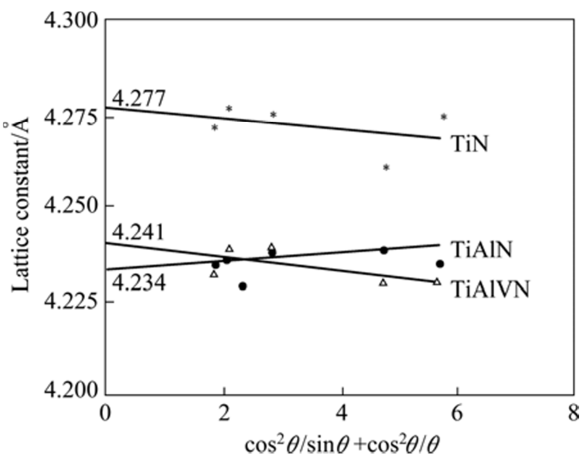

Fig. 2 Extrapolation of measured lattice parameters of TiN, TiAlN and TiAlVN coating versus $\cos^2\theta/\sin\theta+\cos^2\theta/\theta$

3.3 SEM

The cross-sectional SEM morphologies of the TiN, TiAlN and TiAlVN coatings on M2 steel are shown in Fig. 3. It indicates that the columnar structure of TiN/M2 is coarser than that of TiAlN/ M2 or TiAlVN/M2.

3.4 TEM

Figure 4 shows the cross-sectional TEM micrograph of TiN/M2. Moreover, the selected-area diffraction