Rare Metal Materials and Engineering
Volume 51, Issue 1, January 2022
Available online at www.rmme.ac.cn

Science Press

Cite this article as: Iram Sidra, Cai Fei, Wang Jianming, et al. Mechanical and Wear Properties of AlCrN/AlCrVN Multi-layer Coatings with Different Numbers of Bilayers[J]. Rare Metal Materials and Engineering, 2022, 51(01): 74-82.

ARTICLE

Mechanical and Wear Properties of AlCrN/AlCrVN Multi-layer Coatings with Different Numbers of Bilayers

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Abstract: AlCrN/AlCrVN multi-layer coatings with different numbers (1, 2, 4, 6) of bilayers were deposited by arc ion plating method. The effects of multi-layer structure on microstructure, mechanical properties, tribological properties, and cutting performance were investigated. The results show that the deposited AlCrN/AlCrVN multi-layer coatings are mainly composed of solid solution (Al, Cr)N with preferred growth orientation of [111] crystal orientation. Compared with other multi-layer coatings, the lowest coefficient of friction (~0.46), the lowest wear rate of 0.15×10^{-11} $m^3/N\cdot m$, the highest hardness of $HK_{0.05}$=38 000 MPa, and coating-substrate bonding strength of L_{C2}=53±1 N can be achieved for AlCrN/AlCrVN multi-layer coating with 6 bilayers at high temperature. The improvement in hardness and wear resistance is due to the formation of more interfaces between adjacent layers. Cutting test results reveal that AlCrN/AlCrVN coating with 6 bilayers has the longest cutting length of 7.4 m under the cutting wear standard condition of flank wear VB=0.2 due to its relatively higher hardness and better wear resistance.

Key words: AlCrN/AlCrVN multi-layer coatings; microstructure; mechanical properties; tribological behavior; cutting performance

Many nitride coatings deposited by physical vapor deposition (PVD) method, especially the chromium nitride (CrN) coating, are employed as protective coatings because of their outstanding mechanical and tribological properties[1-4]. AlCrN coating formed by doping Al into CrN coating is an improved tool-coating system due to its high hardness, high wear resistance, good thermal stability, and fair oxidation resistance[5]. However, the growing trend of high-speed dry cutting in the machining industry significantly reduces the tool life. Coated tools usually suffer abrasive and adhesive wear and have various types of coating defects, such as thermal cracking, structure failure, and built-up edges, which cannot satisfy the industry requirements.

The self-lubrication mechanism is employed recently to overcome these problems. The influence of self-lubrication caused by different transition metal oxides, such as oxides of W, Mo, and V, during the turning process was investigated[6,7]. These transition metals can be oxidized easily to form a lubricant layer at higher temperature which is beneficial to lower coefficient of friction (COF) [8,9]. Various Mo/V-based nitride coatings are used to achieve low COF and improve wear properties of substrates due to the formation of the lubricious oxide layer on the contact surface[10]. However, the formation of lubricious oxides on the contact surface is not always helpful in decreasing the wear rate or COF which also depend on the morphology and chemical properties of the resulting oxides. The multi-layer coating system is another appropriate method to enhance the wear resistance and adhesion for improving tool life[11]. Multi-layered interfaces provide a certain extent of crack inhibition, thus ameliorating the fracture performance and wear resistance[12-14]. Compared with that with single-layer coatings, the substrate with multi-layer coatings exhibits excellent wear resistance and mechanical strength owing to the specific interfaces. Many

Received date: January 19, 2021
Foundation item: Science Foundation of Anhui Province (1808085QE131); Tribology Science Fund of State Key Laboratory of Tribology (SKLTKF18B13); Key Research and Development Plan of Anhui Province (1804b06020370)
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CrAlN-based multi-layer coating systems, such as CrN/W_2N[15], CrAlN/ZrN[16], CrAlSiN/CrAlN[17], and TiN/CrAlN[18], have been widely studied to improve hardness and wear properties which depend on microstructure, interface adhesion, and the thickness of each individual layer[19].

Although the microstructure, adhesion, and wear resistance of VN-based multi-layer coatings were investigated, the number of bilayers of the AlCrN/AlCrVN coatings is rarely investigated. This research used arc ion plating method to create a new AlCrN/AlCrVN multi-layer coating and to study the influence of the number of bilayers on the microstructure, mechanical properties, tribological properties, and cutting performance.

1 Experiment

The AlCrN/AlCrVN multi-layer coatings were prepared on mirror-polished high-speed steel (HSS, M2 steel) substrates via arc ion plating technique using $Al_{30}Cr_{70}$ and $Cr_{70}V_{30}$ alloys (purity of 99.96%). Initially, all substrates were treated with Ar^+ ion to remove contamination from the surfaces under the Ar (purity of 99.999%) atmosphere at a pressure of 4.0 Pa and bias voltage of −400 V for 10 min. Then, the AlCrN/AlCrVN multi-layer coatings were deposited under the pure N_2 atmosphere at a pressure of 3.5 Pa. The temperature of deposition process was 450 °C. The number of bilayers in multi-layer coatings was 1, 2, 4, and 6, and the specimens were named as AlCrN/AlCrVN-1, AlCrN/AlCrVN-2, AlCrN/AlCrVN-4, and AlCrN/AlCrVN-6, respectively. The detailed operating parameters are listed in Table 1.

The surface and cross-sectional morphologies of the as-deposited AlCrN/AlCrVN multi-layer coatings were observed by scanning electron microscope (SEM, Phenom XL, Netherlands). Energy dispersive spectrometry (EDS) was applied for element composition analysis. The surface roughness was quantitatively analyzed using the Alpha-Step IQ surface profiler (KLA-Tencor P7, Netherlands). X-ray diffraction (XRD, Bruker D8, Germany, Cu Kα) was used to analyze the phase structure of the as-deposited coatings. Transmission electron microscopy (TEM, FEI Tecnai G2 F20, USA) was adopted to study the microstructures using the focused ion beam (FIB) method at 4.5 keV, and the test angle was 3°~8°. The hardness was measured by a microhardness tester under 0.49 N. The scratch test was performed by a scratch tester (acoustic emission method, HT-3001) with the load of 0~80 N. The tribological behavior was evaluated by the CSM HT1000 instrument (Switzerland) at room temperature and 500 °C with Al_2O_3 balls of 6 mm in diameter. The wear characterization was tested at the rotation speed of 400 r/min, load of 5 N, and sliding speed of 0.0732 m/s under the dry-sliding condition. The wear tracks were analyzed by step profiler, SEM, and EDS. The milling test using HSS cutters was conducted on a CNC VMC-85 Hoton (China) machine to examine the cutting performance through the standard wear value of flank wear VB at 0.20 mm. The cutting test was conducted at the linear speed (V_c) of 60 m/min and the spindle speed of 1910 r/min with the feed per tooth (f_n) of 0.2 mm/r, axial depth (a_e) of 2 mm, and axial depth (a_p) of 2 mm.

Table 1 Deposition parameters of AlCrN/AlCrVN multi-layer coatings

Deposition parameter	Value
Bias voltage/V	−80
N_2 flow rate/mL·min^{-1}	100
Temperature/°C	450
Pressure/Pa	3.5
Arc current of AlCr target/A	130
Arc current of CrV target/A	120
Number of bilayers	1, 2, 4, 6
Total deposition time/min	180

2 Results and Discussion

2.1 Cross-section and surface morphologies

Fig.1a~1d illustrate the SEM cross-section morphologies of as-deposited AlCrN/AlCrVN multi-layer coatings. The AlCrN/AlCrVN multi-layer coatings contain the dense columnar structures and adhere to the substrate. The coating thickness is 3.59, 3.81, 3.61, and 3.45 μm for AlCrN/AlCrVN-1, AlCrN/AlCrVN-2, AlCrN/AlCrVN-4, and AlCrN/AlCrVN-6 coatings, respectively. As the number of bilayers is increased, the sharp interfaces gradually disappear in AlCrN/AlCrVN multi-layer coatings, thereby improving the adhesion strength of coatings[20].

Fig. 2a~2d show the surface morphologies of AlCrN/AlCrVN-1, AlCrN/AlCrVN-2, AlCrN/AlCrVN-4, and AlCrN/AlCrVN-6 multi-layer coatings, respectively. The main concern of multi-layer coatings deposited via arc ion plating is the macro-particles (MPs) formed by the evaporation of metal targets[21]. The three-dimensional surface morphologies of AlCrN/AlCrVN multi-layer coatings are shown in Fig.2e~2h. It can be observed that the roughness of multi-layer coatings is decreased with increasing the number of bilayers. The AlCrN/AlCrVN-1 coating has a high surface roughness R_a of 0.34±0.02 μm, while other AlCrN/AlCrVN multi-layer coatings have a relatively lower surface roughness R_a of 0.20±0.02 μm. Improvement of surface roughness can be ascribed to the increase in internal stress with increasing the number of interfaces[22].

2.2 Microstructure analysis

Fig.3 presents XRD patterns of AlCrN/AlCrVN-1, AlCrN/AlCrVN-2, AlCrN/AlCrVN-4, and AlCrN/AlCrVN-6 multi-layer coatings. All coatings have the face centered cubic (fcc) (Al, Cr, V)N solid solution with the diffraction peaks of (111), (200), (220), and (311) crystal planes. All these multi-layer coatings show similar XRD patterns with strong diffraction peak of the (111) crystal plane, indicating the preferential growth direction along (111) plane during deposition. The average crystal size of multi-layer coatings calculated by Scherer's formula is listed in Table 2, which is decreased from

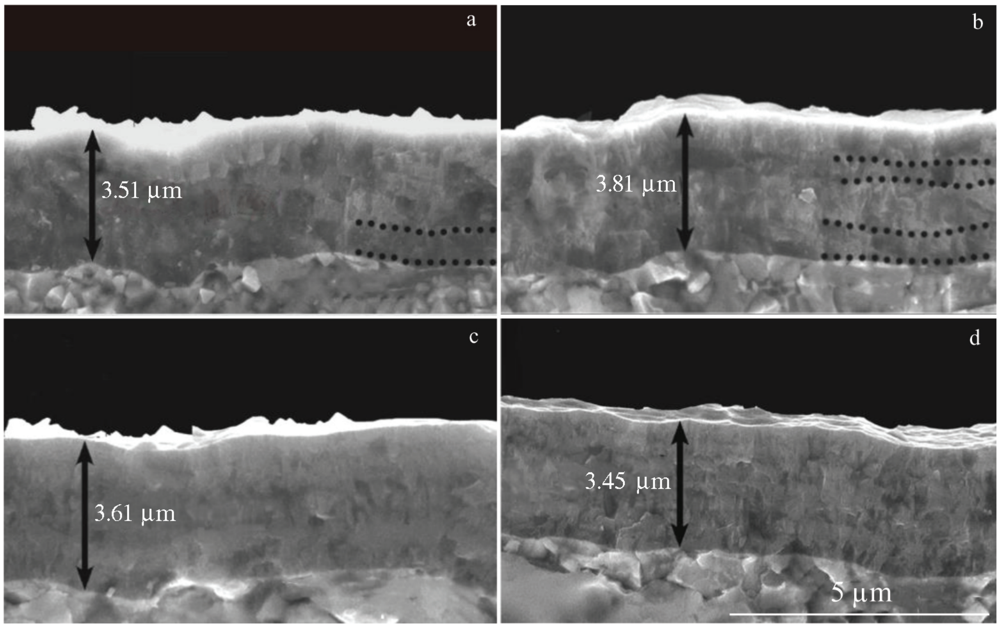

Fig.1 SEM cross-section morphologies of AlCrN/AlCrVN-1 (a), AlCrN/AlCrVN-2 (b), AlCrN/AlCrVN-4 (c), and AlCrN/AlCrVN-6 (d) multi-layer coatings

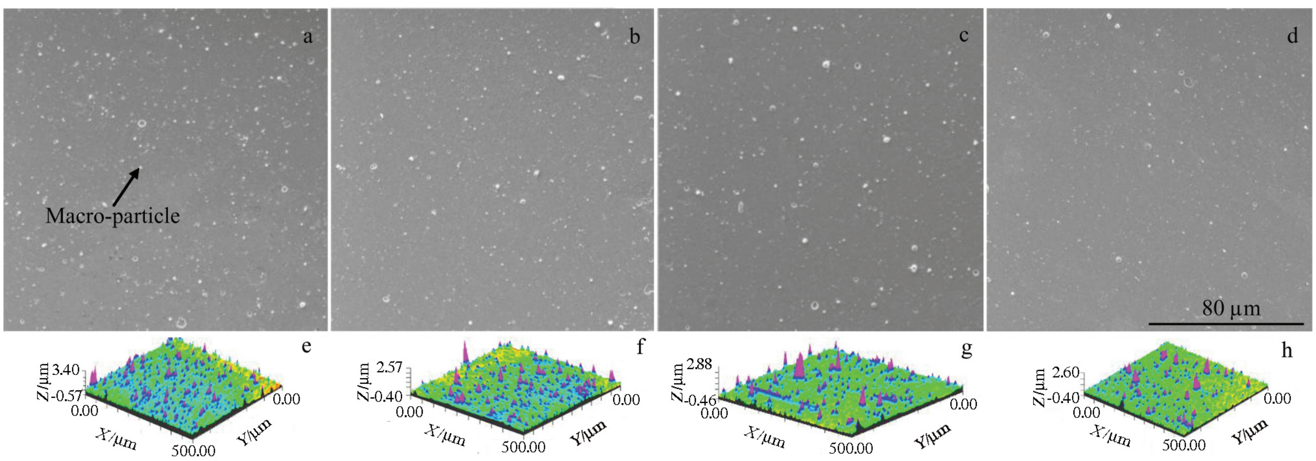

Fig.2 SEM surface morphologies (a~d) and 3D surface morphologies (e~h) of AlCrN/AlCrVN-1 (a, e), AlCrN/AlCrVN-2 (b, f), AlCrN/AlCrVN-4 (c, g), and AlCrN/AlCrVN-6 (d, h) multi-layer coatings

25.14 nm to 18.08 nm with increasing the number of bilayers, suggesting that the increased interfaces can interrupt the grain growth and change the preferred orientation and thus the multi-layer coatings with more bilayers have fine grain size.

The microstructure of AlCrN/AlCrVN-6 coating was investigated by TEM, EDS, high resolution TEM (HRTEM), inverse fast Fourier transform (IFFT), and selected area electron diffraction (SAED), as shown in Fig.4. Fig.4 shows the light and dark layers (Fig.4a) representing the AlCrN and AlCrVN sub-layers, respectively, and the grain growth direction (Fig. 4b), which indicates the existence of the columnar microstructure. The AlCrVN layers are much thicker than the AlCrN layers; the ratio of thickness of AlCrN

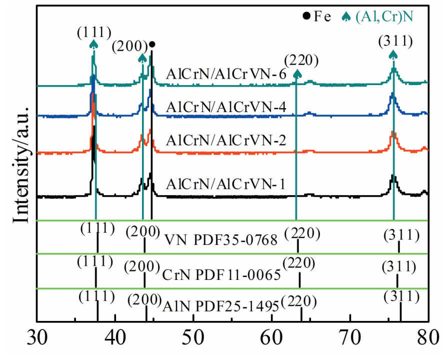

Fig.3 XRD patterns of AlCrN/AlCrVN multi-layer coatings

sub-layer to that of AlCrVN sub-layer is about 1: 2 in the AlCrN/AlCrVN multi-layer coatings (Fig. 4c). The element composition of each layer can be obtained by TEM-EDS line-scanning method. The EDS spectra (Fig. 4d) of AlCrN and AlCrVN layers confirm the presence of Cr, Al, V, and N in the coating. Both the AlCrN and AlCrVN sub-layers show a crystalline grain structure without amorphous regions, as shown in Fig. 4e. Two lattice planes with d=0.24 nm and d=0.21 nm can be identified as the CrN (111) plane and VN (200) plane, respectively, indicating the existence of VN phase in AlCrN/AlCrVN multi-layer coatings. The IFFT measurement precisely confirms a correlation between the AlCrN and AlCrVN sub-layers: the interplanar distance d of the AlCrN layer is longer than that of the AlCrN layer, as shown in Fig.4f. Different lattice orientations at the interfaces of AlCrN/AlCrVN-6 multi-layer coating can also be observed. These interfaces can generate a stress field to block the movement of dislocations at interfaces, thereby enhancing the hardness[23]. In addition, SAED patterns in Fig. 4g and 4h reveal that the AlCrN layer contains a CrN crystal plane and the polycrystalline planes of CrN and VN can also be observed in AlCrVN layers. These phases play a positive role in improving the mechanical properties and tribological performance.

2.3 Mechanical properties

The hardness $HK_{0.05}$ of AlCrN/AlCrVN multi-layer coatings is shown in Fig.5. In AlCrN/AlCrVN multi-layer coatings, the average hardness is increased with increasing the number of bilayers. The AlCrN/AlCrVN-6 multi-layer coating has the maximum hardness of 38 000 MPa. As the number of bi-layers is increased, the periodic interfaces between adjacent layers block the dislocation movement, which increases the hardness[24].

The adhesion strength of the AlCrN/AlCrVN multi-layer coatings was evaluated by the scratch test, as shown in Fig.6. Generally, the scratch track can be characterized into three phases: L_{C1}, L_{C2}, and L_{C3} (L_{C2} refers to the adhesion strength of the coatings)[25]. Adhesion strength (L_{C2}) of 33±1, 35±1, 47±1, and 53±1 N is achieved for AlCrN/AlCrVN-1, AlCrN/AlCrVN-2, AlCrN/AlCrVN-4, and AlCrN/AlCrVN-6 multi-

Table 2 Surface roughness R_a, average crystal size, coefficient of friction, and wear rate of AlCrN/AlCrVN multi-layer coatings

Specimen	Roughness, R_a/μm	Average crystal size/nm	COF at RT	COF at HT	Wear rate at RT/ $\times10^{-17}$ $m^3 \cdot N^{-1} \cdot m^{-1}$	Wear rate at HT/ $\times10^{-11}$ $m^3 \cdot N^{-1} \cdot m^{-1}$
AlCrN/AlCrVN-1	0.34	25.14	0.46	0.55	0.43	3.26
AlCrN/AlCrVN-2	0.24	22.02	0.42	0.57	0.23	0.24
AlCrN/AlCrVN-4	0.23	20.67	0.41	0.49	0.20	0.18
AlCrN/AlCrVN-6	0.20	18.08	0.39	0.46	0.24	0.15

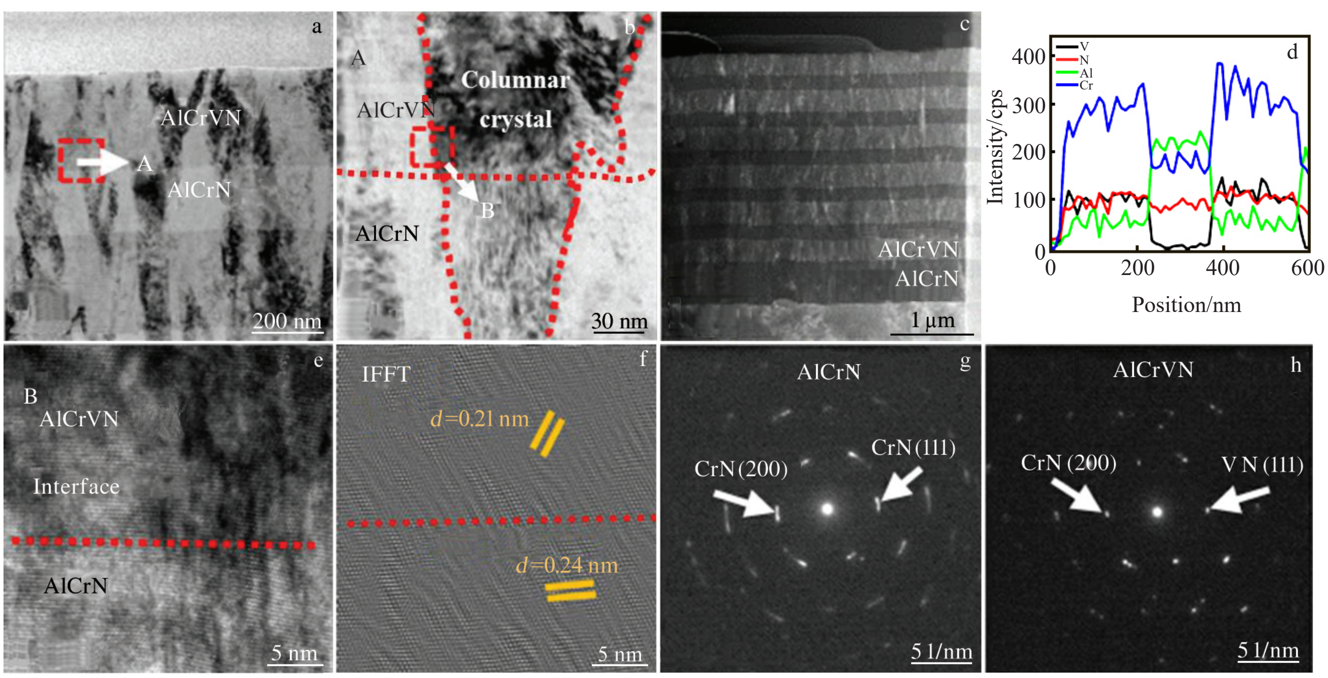

Fig.4 TEM cross-section morphology (a) and magnified TEM image of area A in Fig.4a (b) of AlCrN/AlCrVN-6 multi-layer coating; TEM cross-section morphology (c) and corresponding EDS spectra (d) of AlCrN/AlCrVN-6 multi-layer coating; HRTEM image of area B in Fig.4b (e) and corresponding IFFT image (f) of AlCrN/AlCrVN-6 multi-layer coating; SAED patterns of AlCrN layer (g) and AlCrVN layer (h)

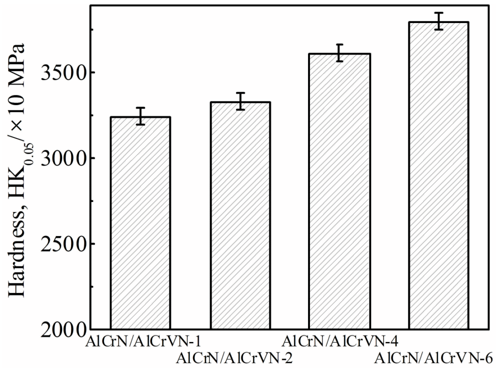

Fig.5 Hardness of AlCrN/AlCrVN multi-layer coatings

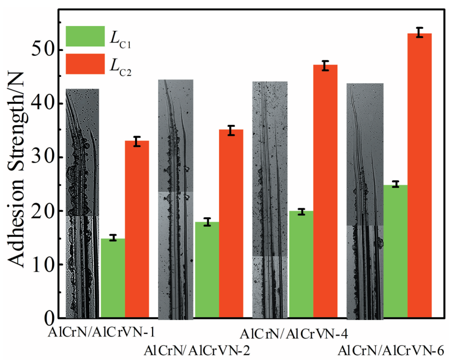

Fig.6 Scratch images and adhesion strength of AlCrN/AlCrVN multi-layer coatings

layer coatings, respectively. The AlCrN/AlCrVN-6 multi-layer coating shows less delamination in the coating surface under the applied load, indicating a higher adhesion strength. Therefore, the scratch test results reveal that the adhesion strength of AlCrN/AlCrVN multi-layer coatings is significantly improved with increasing the number of bilayers; multi-layer coatings therefore achieve a better resistance to crack initiation[26].

2.4 Tribological properties

COF of AlCrN/AlCrVN multi-layer coatings against Al_2O_3 balls at room temperature (RT) and high temperature (HT) of 500 °C is illustrated in Fig.7a and 7b, respectively. All AlCrN/AlCrVN multi-layer coatings have a similar trend of COF during the dry-sliding test. In Fig. 7a, COF of the AlCrN/AlCrVN-4 and AlCrN/AlCrVN-6 multi-layer coatings is slightly higher firstly, then decreased after 400 s, and finally stabilized, which demonstrates a better result compared with AlCrN/AlCrVN-1 and AlCrN/AlCrVN-2 multi-layer coatings at RT. This increase in COF from the beginning can be a result of the variation in contact stress and the surface roughness[27]. In general, COF ranges in 0.39~0.46 for all coatings at RT. The maximum COF of AlCrN/AlCrVN-1 and AlCrN/AlCrVN-2 multi-layer coatings at around 0.55 and 0.57 can be obtained, respectively. COF of AlCrN/AlCrVN multi-layer coatings is decreased with increasing the number of bilayers, and the minimum COF value of 0.49 and 0.46 can be obtained

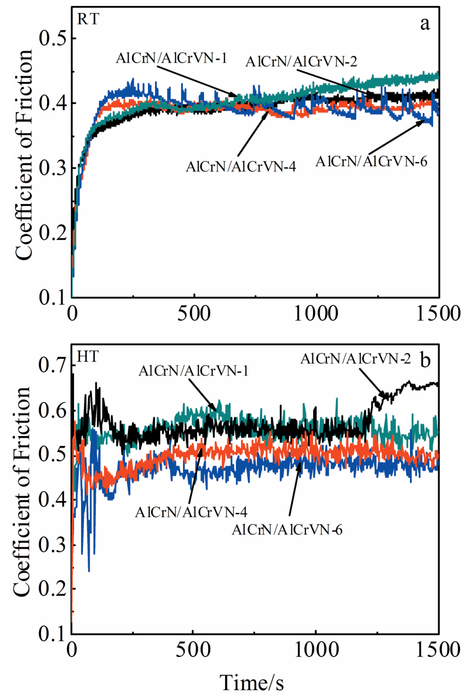

Fig.7 COF of AlCrN/AlCrVN multi-layer coatings at room temperature (a) and high temperature (b)

for the AlCrN/AlCrVN-4 and AlCrN/AlCrVN-6 multi-layer coatings, respectively, at HT. A similar trend can also be found for the CrN-based coatings containing vanadium[28].

The calculated wear rates of different multi-layer coatings are shown in Table 2. AlCrN/AlCrVN-1 multi-layer coating exhibits the largest wear rate at RT of 0.43×10^{-17} m^3/N·m, while AlCrN/AlCrVN-2, AlCrN/AlCrVN-4, and AlCrN/AlCrVN-6 multi-layer coatings have the wear rate of 0.23×10^{-17}, 0.20×10^{-17}, and 0.24×10^{-17} m^3/N·m, respectively. As the testing temperature is increased from RT to HT of 500 °C, the wear rate of the AlCrN/AlCrVN-1 multi-layer coating is increased to 3.26×10^{-11} m^3/N·m. The wear rate at HT of AlCrN/AlCrVN-2, AlCrN/AlCrVN-4, and AlCrN/AlCrVN-6 multi-layer coatings is 0.24×10^{-11}, 0.18×10^{-11}, and 0.15×10^{-11} m^3/N·m, respectively. Therefore, the increasing bilayer number is conducive to improving the wear properties at elevated temperature due to the better adhesion strength and higher hardness of multi-layer coatings and the formation of oxide layer. In addition, more interfaces have multiple barriers against the propagation of cracks. Wang et al[29] found that CrAlN/VN multi-layer coatings with more interfaces always provide positive effects through stress relaxation and crack deflection for reducing wear rate, compared with the single-layer coatings[22].

Fig. 8a~8d show wear track morphologies of AlCrN/AlCrVN multi-layer coatings at RT. The worn morphology of AlCrN/AlCrVN-1 and AlCrN/AlCrVN-2 multi-layer coatings shows abrasion and adhesion wear parallel to the relative sliding movement due to the lower hardness; however, no obvious abrasive wear can be found on AlCrN/AlCrVN-4 and AlCrN/AlCrVN-6 multi-layer coatings, which can be ascribed

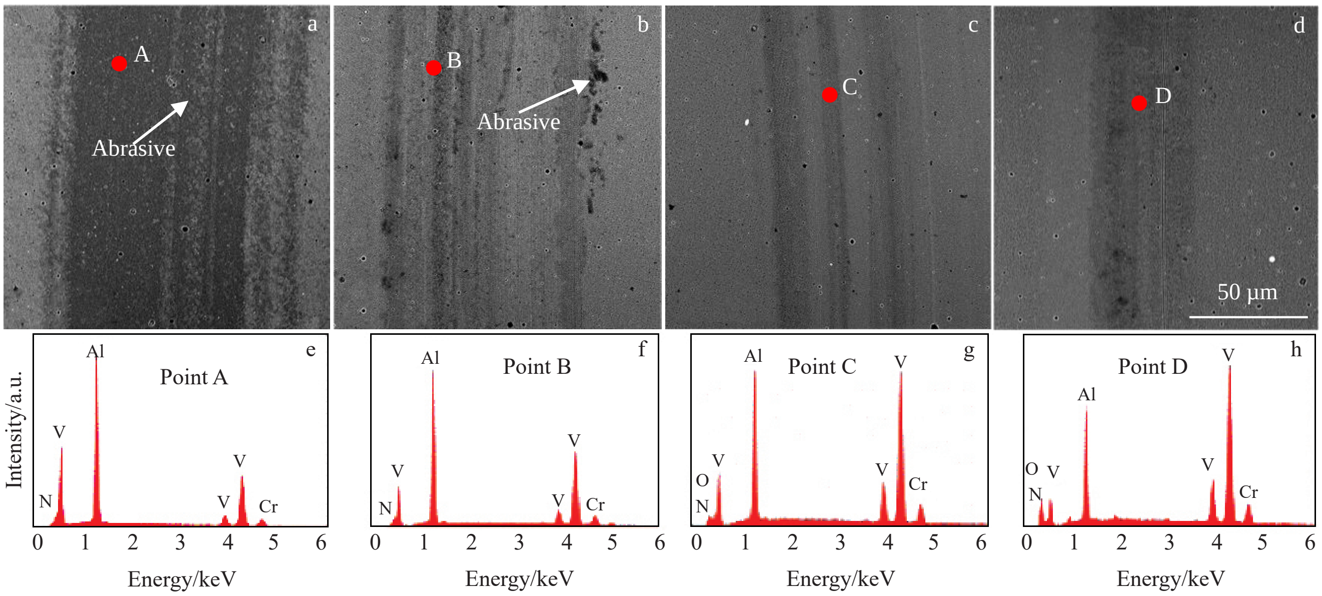

Fig.8 Wear track morphologies (a~d) and EDS spectra of red points A~D in Fig.8a~8d (e~h) of AlCrN/AlCrVN multi-layer coatings at room temperature: (a, e) AlCrN/AlCrVN-1, (b, f) AlCrN/AlCrVN-2, (c, g) AlCrN/AlCrVN-4, and (d, h) AlCrN/AlCrVN-6

to the improved hardness and strong interaction of interfaces. Tribological behavior was further studied via EDS analysis, as illustrated in Fig.8e~8f. Al, Cr, O, N, and V can be observed as major elements on the wear tracks. The surface morphologies and EDS spectra of the wear tracks at HT are shown in Fig. 9. At HT, the abrasion and adhesion wear is more obvious on the surface of all multi-layer coatings. The wear track of AlCrN/AlCrVN-1 coating is wider, indicating that the protection against friction is weakened at HT. Meanwhile, AlCrN/AlCrVN-4 and AlCrN/AlCrVN-6 multi-layer coatings have narrow wear tracks and thus possess better wear properties under lower abrasion condition. This phenomenon is due to the improved hardness for resisting abrasion. The corresponding EDS analysis shows that there are high concentrations of O, V, Cr, and Al elements. The high

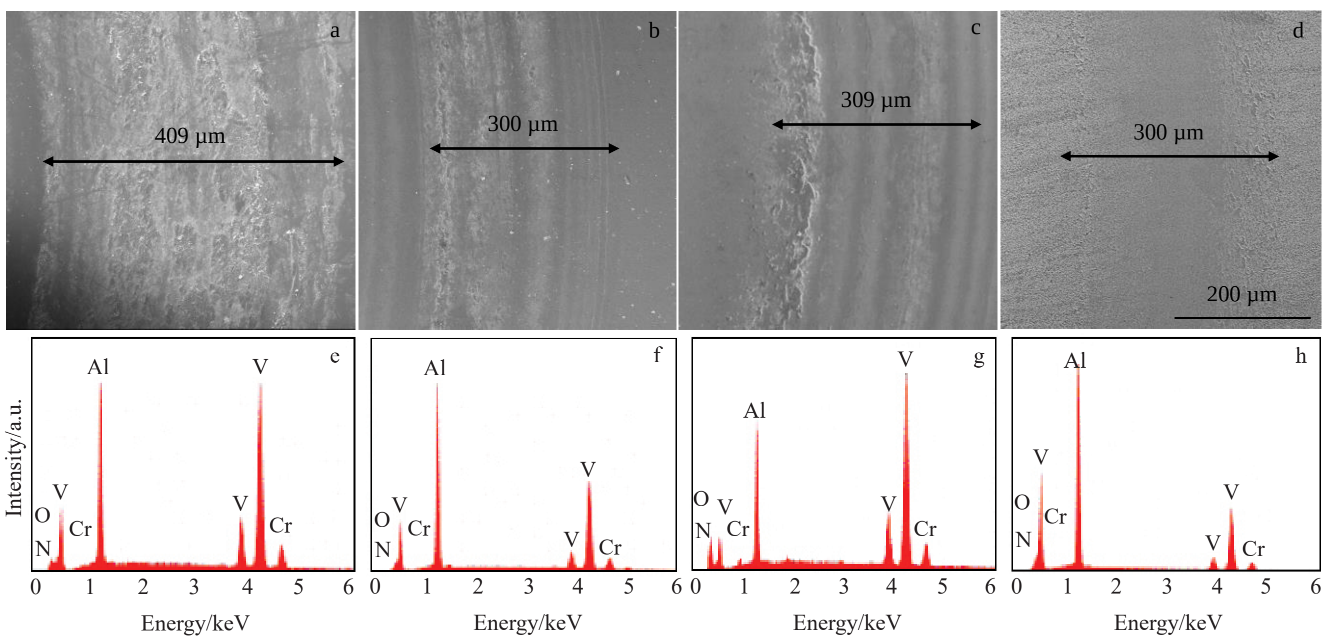

Fig.9 Wear track morphologies (a~d) and EDS spectra (e~h) of AlCrN/AlCrVN multi-layer coatings at high temperature: (a, e) AlCrN/AlCrVN-1, (b, f) AlCrN/AlCrVN-2, (c, g) AlCrN/AlCrVN-4, and (d, h) AlCrN/AlCrVN-6

content of O element on the surface of the AlCrN/AlCrVN-4 and AlCrN/AlCrVN-6 multi-layer coatings indicates the formation of vanadium oxides (V_2O_3) during the sliding test[30,31].

2.5 Cutting performance

The flank wear VB standard was set as 0.20 mm in this research. As shown in Fig.10, when VB=0.20 mm, the cutting lengths of AlCrN/AlCrVN coated cutters are 5.6, 5.4, 6.2, and 7.4 m for AlCrN/AlCrVN-1, AlCrN/AlCrVN-2, AlCrN/AlCrVN-4, and AlCrN/AlCrVN-6 multi-layer coatings, respectively. The AlCrN/AlCrVN-6 coated tools have the longest service life, which is associated with its high adhesion strength, great hardness, and low COF.

The worn morphologies and EDS spectra of the coated cutters are shown in Fig. 11. The flank surface morphologies can be divided into three areas: the fully worn area (substrate), delaminated (partially worn) area, and area without wear. Typical adhesion, chipping, and abrasive wear can be detected as main components on the flank surface of the AlCrN/AlCrVN-1 and AlCrN/AlCrVN-2 coated tools. With

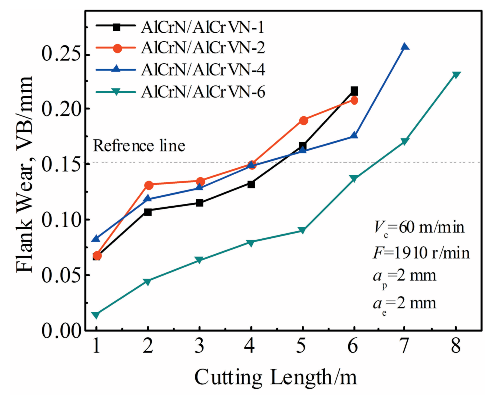

Fig.10 Cutting life curves of different AlCrN/AlCrVN end milling cutters at cutting speed of 60 m/min

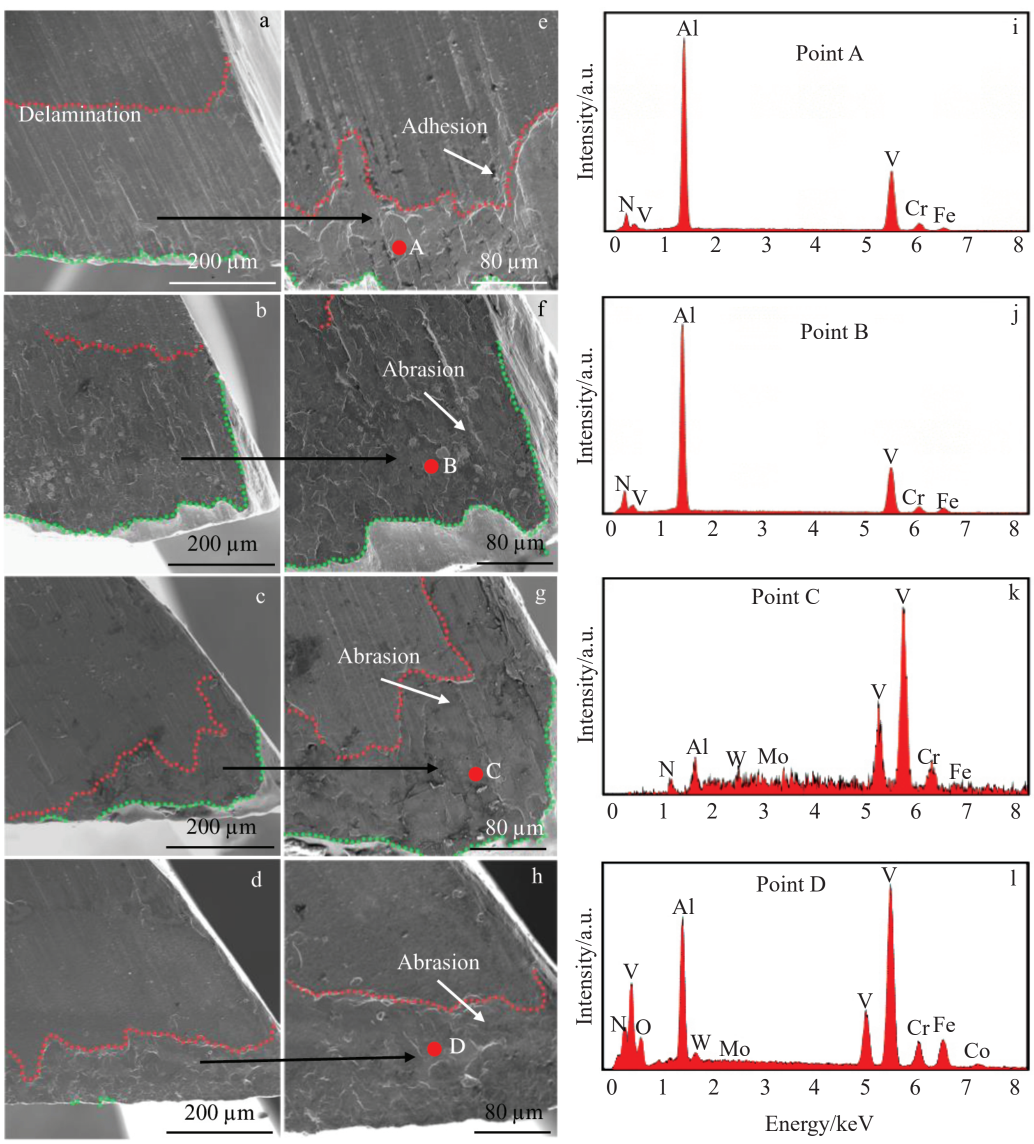

Fig.11 Flank surface morphologies (a~h) and EDS spectra of red points A~D in Fig.11e~11h (i~l) of AlCrN/AlCrVN end milling cutters: (a, e, i) AlCrN/AlCrVN-1, (b, f, j) AlCrN/AlCrVN-2, (c, g, k) AlCrN/AlCrVN-4, and (d, h, l) AlCrN/AlCrVN-6

increasing the number of bilayers, more interfaces are introduced into the coating, which hinders the dislocation and propagation of cracks and enhances the mechanical strength and wear resistance of the coatings[32,33]. No obvious adhesive wear and chipping phenomena can be observed on AlCrN/AlCrVN-4 and AlCrN/AlCrVN-6 coated tools, and only a small amount of abrasion can be observed on the sides of the tool due to the higher hardness of coating and lower COF between workpiece and tool[34]. EDS analysis confirms the presence of oxygen on the surface. Thus, AlCrN/AlCrVN-6 coated tool with more interfaces has better mechanical properties and tribological performance.

3 Conclusions

1) The deposited AlCrN/AlCrVN multi-layer coatings primarily consist of face centered cubic solid solution (Al, Cr)N with [111] preferential orientation.

2) The lowest coefficient of friction around 0.46, the lowest wear rate of 0.15×10^{-11} $m^3/N\cdot m$, the highest hardness of 38 000 MPa, and adhesion strength L_{C2} of 53±1 N are achieved for AlCrN/AlCrVN multi-layer coating with 6 bilayers at high temperature .

3) Owing to the greater hardness and lower wear rate, AlCrN/AlCrVN multi-layer coating with 6 bilayers also has the longest cutting life of 7.4 m under the condition of flank wear VB=0.2 mm.

4) For AlCrN/AlCrVN multi-layer coating with 6 bilayers, more interfaces can hinder the crack propagation, which positively affects the mechanical properties and tribological properties.

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不同双层结构AlCrN/AlCrVN多层涂层的力学和磨损性能

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摘　要：采用电弧离子镀的方法制备了不同数目（1、2、4、6）双层结构的AlCrN/AlCrVN多层涂层，并研究了多层结构对涂层微观结构、力学、摩擦学和切削性能的影响。结果显示，沉积态AlCrN/AlCrVN多层涂层主要由固溶(Al，Cr)N组成，优先生长方向为[111]晶向。与其他多层涂层相比，具有6层双层结构的AlCrN/AlCrVN涂层在高温下表现出较低的摩擦系数（约0.46）和磨损率（0.15×10^{-11} $m^3/N\cdot m$），以及较高的硬度（$HK_{0.05}$=38 000 MPa）和膜-基结合强度（L_{C2}=53±1 N）。多层涂层相邻层之间形成了较多的界面，有助于提高多层涂层的硬度和耐磨性。切削试验结果显示，当切削磨损标准VB=0.2时，AlCrN/AlCrVN-6涂层具有较高的硬度和耐磨性，最长的切削长度为7.4 m。

关键词：AlCrN/AlCrVN多层涂层；微观结构；力学性能；摩擦学性能；切削性能

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