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Thick CrN/TiN multilayers deposited by arc ion plating

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ABSTRACT

The thick multilayers, composed of one, two and three CrN/TiN bilayers, as well as CrN and TiN monolayers, were fabricated on well-polished high-speed-steel by arc ion plating, respectively. The microstructure and mechanical properties of the multilayers have been measured and analyzed using traditional methods. According to the SEM morphology, the number of the macro-particles on CrN is larger than TiN surface, showing that the CrN film has coarser surfaces. The periodic structure of the multilayers can be clearly seen by cross-sectional SEM. The smooth, regular and sharp interfaces indicate that the sublayer combines both with each other and with the substrate tightly. It is found that the mechanical properties of these thick multilayers, which have thickness of several to more than 10 μm, have been improved when compared with CrN or TiN single layer. It is considered that the interfaces and the alternating soft/hard substructure of CrN/TiN bilayer contribute to the enhanced mechanical properties.

1. Introduction

Transition metal nitrides, such as TiN, CrN, TiAlN, etc., are usually deposited onto the surface of cutting tools and moulds due to their high hardness and excellent wear-resistance [1,2]. However, the mechanical properties of monothetic metal nidride layer cannot satisfy modern cutting requirement. It is found that this problem may be solved by using metal/ceramic multilayered coatings that possess a high hardness as well as a high toughness when compared to the hogomeneous coatings [3–5]. Toughening of lamellae coatings can be a result of several mechanisms, e.g., energy dissipation by ductile fracture of the metal phase, delaminating of interfaces and sliding or shearing at interfaces [6–9]. But in some cases this has been at the expense of reduced hardness and wear-resistance. Application of multilayered coatings, consisting of alternative ceramic bilayers, may overcome this limitation [10–12]. The multilayer, consisting of CrN/TiN bilayers, is an ideal candidate, since deposition of both PVD TiN and CrN is well-established technique. While TiN possesses high hardness, both TiN and especially CrN have shown high fracture toughness as compared to other commercial PVD coatings [13,14]. It is considered that the effects of interface on the mechanical properties are significant while the thickness of the monolayers is of nanometer levels, therefore, many studies have been carried out to investigate the multilayers consisting of several or even tens of bilayers, which usually have thickness modulation period less than 100 nm [15–17]. These research results show that the presence of a large number of interfaces between individual layers of a multilayered structure results in a drastic increase in hardness and strength. What happens if the multilayers are composed of thick monolayers which have thickness of micrometer scale? Will the effects of interfaces still exist? Since CrN has lower internal stress, higher toughness and better wear-resistance than TiN film [18], it is prospective to improve mechanical properties even for the thick TiN/CrN multilayers. To our best knowledge, there have been few reports on the interfacial effects of thick multilayers. This work is dedicated to the microstructure and mechanical properties of thick multilayered CrN/TiN, which is consisting of micrometer-scale monolayers, with respect to those of CrN or TiN single layer. It is found that the interfaces in the thick multilayers do have effects on the mechanical properties.

2. Experimental details

The substrate, made of high-speed steel W18Cr4V, was strictly cleaned, dried and put into the deposition chamber after being ground and polished manually. The background vacuum was pumped to 3×10^{-3} Pa, and then the substrate was further cleaned by Ar plasma glow discharge for 3 min at 5 Pa. The metallic Cr or/and Ti, with purity of no less than 99.99%, were used as the arc targets, respectively. Only nitrogen, which acts as the reactive gas,

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Table 1
Experimental details.

No.	Arc current (A)/arc voltage of Cr target (V)	Arc current (A)/arc voltage of Ti target (V)	Nitrogen flow (sccm)	Negative bias (V)	Repetition number of CrN/CrN bilayer	Deposition time for each individual layer (min)
1	70/25		250	300		20
2		60/20	250	300		20
3	70/25	60/20	250	300	1	10
4	70/25	60/20	250	300	2	10
5	70/25	60/20	250	300	3	10
6	70/25	60/20	250	300	1	30
7	70/25	60/20	250	300	2	30
8	70/25	60/20	250	300	3	30

was input into the chamber during deposition. All nitride films were deposited onto the unheated substrate, which was positioned 30 cm in front of the targets and was applied with a negative bias of 300 V. The substrate ion current density, estimated by the current and the total area of the substrate, was about 40 A/m^2. Detailed deposition parameters are listed in Table 1, where samples 1 and 2 were CrN and TiN single layer, while samples 3–8 are multilayers composed of CrN/TiN bilayers, respectively. CrN layer was deposited prior to TiN for the multilayer samples.

The thickness of the films was measured by the XP-2 profiler and the deposition rate was obtained by dividing the deposition time. The SHIMA-DZU SSX-550 scanning electron microscopy (SEM) was adopted to observe surface topography and cross-section morphology of the films, respectively. Phase and microstructure of the samples were characterized by X-ray diffraction (XRD). The mechanical properties, hardness and elastic modulus, were characterized by the XP nano-indenter, while adhesion strength of the films to the substrate was measured by the MFT-30 scratch tester. The maximum load used to measure the hardness for the CrN and TiN monolithic films was 200 mN, and that for the multilayers was 630 mN.

3. Results and discussion

3.1. Deposition rate

The thickness of the films was determined by the steep step in the profile lines. The deposition rate for the samples was obtained through dividing the thickness of the coating(s) by the deposition time. In case of multilayers composed of Cr/N/TiN bilayers, total thickness and total deposition time were applied, and thus giving the average value. Table 2 shows the results. Deposition rate of the CrN single layer was 100 nm/min and that of TiN 60 nm/min, respectively. In principle, the average deposition rate should be 80 nm/min since the bilayer consisting of one TiN single layer and one CrN single layer. According to Table 2, one may see that the average deposition rate of the multilayers is a little bit less than the expected value. It indicates that the average deposition rate decreases as the number of bilayers increases. This might be explained by the “toxication” of the target by reacting with nitrogen as deposition process goes on, forming a thin layer of nitride on the target surface, which lowers the evaporation rate and thus decreases the deposition rate [19–21].

Table 2
Thickness and deposition rate of the samples.

Sample No.	1	2	3	4	5	6	7	8
Thickness (nm)	2000	1200	1586	3212	4560	4794	9360	13500
Average deposition rate (nm/min)	100	60	79.3	80.3	76	79.9	78	75

3.2. SEM morphology

The top surface and the cross-sectional morphology of the coatings were observed by SEM, as illustrated by Figs. 1 and 2, respectively. Fig. 1(a)–(c) shows the top surface morphology of samples 1, 2 and 8, respectively. On the top surface of the coatings there exist many white macro-particles (also called as droplets), which were formed by ejection of the melted spots of the metallic target(s) onto the substrate due to the high temperature arc [22,23]. As indicated in Fig. 1, there are more macro-particles on the CrN surface (a) than the TiN surface (b). According to the deposition sequence of the multilayers, as described in the experimental details, the top layer of the multilayers is TiN, which has less macro-particles (Fig. 1(c)). Munz et al. [24] and Nordin et al. [25] reported that the number of droplets of TiN was less than that of CrN, since the later has a higher melting point. It seems that our results are contrary to theirs. We believe that the formation of nitride on the target surface plays an important role, since in our case, only nitrogen (and no argon) was applied during the deposition. It is readily for nitrogen to react chemically with the metallic target and form a thin layer of nitride on the surface, reducing the droplets as well as the deposition rate, as mentioned above. Another probable reason is that the arc power for Cr was (1750 W) higher than Ti target (1200 W), according to the arc currents and voltages given by Table 1.

The cross-sectional morphology of samples 7 and 8 is shown in Fig. 2. It is seen that both the interface between the multilayers and the substrate and those within the multilayers are clear and distinct. Each layer is tightly adhesive to another, and no crack or hole is observed. One may also see from these pictures that the upper layers that were deposited late have a smaller thickness compared with those formerly deposited despite the deposition time for each layer was the same, indicating that the deposition rate becomes slow as time goes on, which is accordant well with the results shown in Table 2.

3.3. XRD results

The crystalline structures of all samples are measured by X-ray diffraction and the results are given in Fig. 3, where the sample number is marked beside each spectrum. The films, including single layer of CrN and TiN as well as multilayers, have a face-centered cubic (FCC) crystalline structure, as indicated by the sharp diffraction peaks. A very small shift of the peaks can be found as compared with those in the standard PDF cards, indicating that both nitrides are not stoichiometric, which is common in the reactive depositions. It is seen that the CrN single film is (220) preferred, while TiN single film has the strongest diffraction intensity from its (111) plane. In the XRD patterns of the multilayers, peaks indicating the phases of both TiN and CrN are observed. One should pay attention to the peak Fe (110),