



Structure and properties of $(\text{AlCrMnMoNiZrB}_{0.1})\text{N}_x$ coatings prepared by reactive DC sputtering

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ABSTRACT

The microstructure and properties of $\text{AlCrMnMoNiZrB}_{0.1}$ nitride films prepared by reactive direct current sputtering at various N_2 -to-Ar flow ratios (R_N) were investigated. The films had an amorphous structure at low R_N and a face-centered cubic structure at a high R_N . As the R_N increased, the decrease in clusters and defects resulted in a dense columnar structure and low surface roughness. The peak hardness and modulus of the nitride films were 10.3 and 180 GPa, respectively. The enhanced hardness is ascribed to the increased metal–nitrogen bonding, solid solution strengthening of several metallic nitrides, and lattice strain. The nitride films deposited at $R_N = 0.2, 0.5,$ and 0.8 had friction coefficients of 0.16, 0.12 and 0.15, respectively. Wear-out failure occurred within 400 s when $R_N = 0$ and 1.0. Adhesive wear was the dominant wear mechanism.

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1. Introduction

Transition metal nitrides are commonly used as protective surface coatings for industrial applications [1,2]. Simple binary and ternary nitride systems such as TiN, CrN, and TiAlN have been widely developed [3,4]. Multi-element systems with metallic and metalloid components have also been investigated [5,6]. However, majority of studies on coatings are still limited to quaternary systems.

Recently, Yeh and Cantor proposed a novel alloy system called high-entropy alloys (HEAs), composed of at least five principal elements, with concentrations between 5 at.% and 35 at.% [7–10]. Their simple structures and versatile properties result from the high mixing entropy, lattice distortion, sluggish diffusion, and cocktail effect. These materials have potential as coating systems for many applications, including the manufacture of tools, molds, dies, and mechanical parts [7–13]. Therefore, the preparation and characterization of metallic, nitride, and oxide films of HEAs are of great interest in exploring new and promising coating systems [14–18]. Metallic films and majority of the nitride films have amorphous and face-centered cubic (FCC) solid solution structures, respectively. The mechanical properties of nitride films are also enhanced by solid solution strengthening and lattice distortion, which results from the incorporation of nitrogen. To extend the

application fields of HEAs and understand the structure and the properties of their nitride films, a novel film system derived from the $\text{AlCrMnMoNiZrB}_{0.1}$ alloy was deposited by direct current (DC) magnetron sputtering under various N_2/Ar flow ratios (R_N). The chemical composition, microstructure, mechanical properties, and tribological properties of these nitride films were investigated.

2. Experimental procedure

2.1. Film preparation

The films were deposited on Si (1 0 0) wafers from an alloy target of $\text{AlCrMnMoNiZrB}_{0.1}$ by reactive DC magnetron sputtering in a mixed atmosphere of Ar and N_2 . The silicon substrates were cleaned ultrasonically in acetone before loading into the vacuum chamber. Prior to deposition, the substrates were heated to 500 K, and the target was pre-sputtered for 10 min to remove the surface impurities. The sputtering power and the working pressure were 80 W and 2.0 Pa, respectively. The distance between the substrate and the target was 75 mm. The deposition time was 2 h. The nitrogen flow ratio N_2/Ar , denoted as R_N , was varied from 0 to 1.0 to prepare films of various nitrogen contents from the $\text{AlCrMnMoNiZrB}_{0.1}$ target. Table 1 provides the detailed experimental conditions for the deposition.

2.2. Film characterization

The crystal structures of the deposited films were characterized by X-ray diffraction (XRD, X'Pert PROX) with a Cu K α radiation

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Table 1
Experimental parameters of the AlCrMnMoNiZrB_{0.1} nitride films.

Parameter	Value
Substrate material	Si (1 0 0)
Working distance	75 mm
Total flow rate	60 sccm
Nitrogen flow ratio (N ₂ /Ar)	0, 0.2, 0.5, 0.8, 1.0
Working pressure	2.0 Pa
Base pressure	8.0 × 10 ⁻³ Pa
Target power	80 W
Substrate bias	0 V
Substrate temperature	500 K
Substrate rotation speed	40 rpm
Deposition time	120 min

and a glancing incident angle of 4°. The chemical compositions of the films were analyzed using an electron probe microanalyzer (EPMA, JXA-8100). Atomic force microscope (AFM, SPM-9500-J3) was used to evaluate the surface morphology and roughness of the films. The plane-view and cross-sectional microstructures were observed under a scanning electron microscope (SEM, JSM-6700F). Film hardness and modulus were measured using a G200-type nanoindenter with a maximum load of 5 mN. The penetration depth of the indenter was controlled to less than one-tenth of the film thickness to avoid substrate effects.

The friction coefficient of the (AlCrMnMoNiZrB_{0.1})N_x films was evaluated using a UMT-2 Multi-Specimen Test System (CETR, USA) in the ball-on-plate contact configuration, wherein the lower plate containing the silicon wafer coated with the HEA nitride films slid against an Si₃N₄ ball (4 mm diameter, 18 GPa hardness) counterpart for a distance of 5.0 mm. The Si₃N₄ ball was cleaned with acetone in a supersonic bath before each test. All the tests were performed using a normal load of 200 mN, a constant sliding speed of 0.01 m/s at 25 °C, and a relative humidity of 10%. The friction coefficient–time plots were automatically recorded. The morphologies of the wear tracks and the corresponding wear scars were observed by SEM.

3. Results and discussion

3.1. Chemical composition and deposition rate

Fig. 1 shows the chemical compositions and relative content of the AlCrMnMoNiZrB_{0.1} nitride films analyzed by EPMA. The nitrogen content of the nitride films sharply increased with increasing

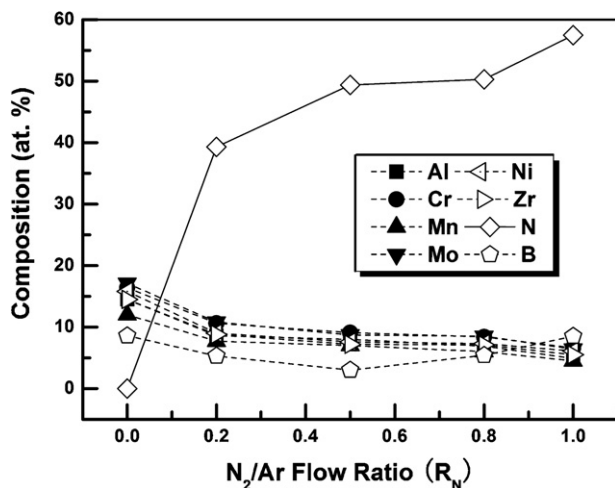


Fig. 1. Chemical composition and relative content of the AlCrMnMoNiZrB_{0.1} nitride films analyzed by EPMA as a function of R_N .

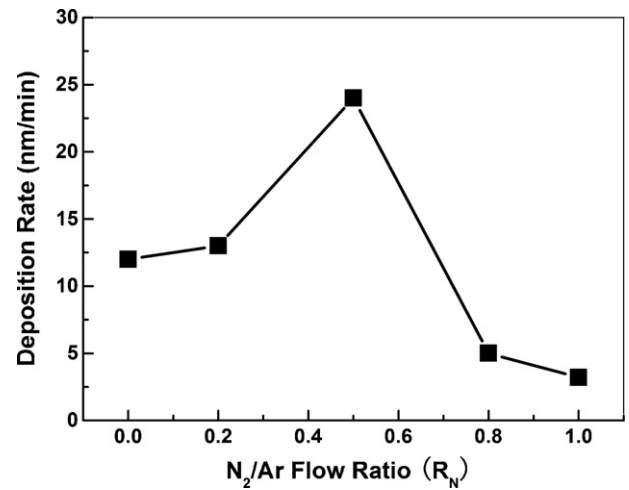


Fig. 2. Deposition rate of the AlCrMnMoNiZrB_{0.1} nitride films as a function of R_N .

R_N and reached ~50 at.% at $R_N = 0.5$. Thus, the HEA nitride films tended to form a solid solution of different nitrides with the stoichiometry MeN (Me: metal). As the R_N further increased, the nitrogen contents increased to >50 at.%. This is due to the incorporation of N atoms in the interstices of the coatings [19]. In addition, the boron content in the target should be only 1.64 at.%, but the EPMA results suggest that the alloy film have a B content of ~9 at.%. The unusual phenomenon may be ascribed to systematic error. This is because light element B and its low content result in great measurement error.

Fig. 2 is the plot of the film deposition rate as a function of R_N . The deposition rate increased slightly as R_N increased from 0 to 0.2. High Ar partial pressure ($R_N = 0$) causes more collisions between the Ar and alloy particles, which results in energy loss of the particles. These particles cannot reach the substrate or break through the gas-absorbed layer, thus the decrease in the film deposition rate. With the addition of nitrogen (when $R_N = 0.2$ and 0.5), the decreased argon partial pressure raised the number of secondary electrons and thus led to the increased deposition rate. The deposition rate decreased sharply from a peak of 24 nm/min at $R_N = 0.5$ to 4 nm/min at $R_N = 0.8$, and then decreased slowly to 3 nm/min at $R_N = 1.0$. This phenomenon is a typical result of target poisoning. Moreover, the nitrogen ions are ineffective for sputtering compared with argon [20–23]. Therefore, the deposition rate decreased as R_N increased.

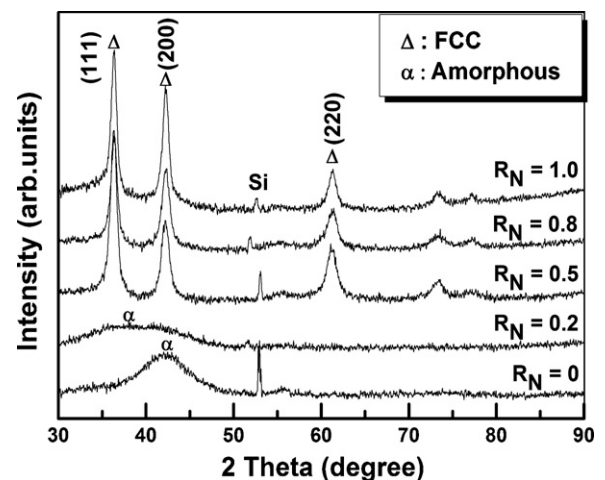


Fig. 3. XRD patterns of the AlCrMnMoNiZrB_{0.1} nitride films deposited on Si (1 0 0) substrates at different R_N values.

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