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Microstructure and mechanical properties of Al-Mg-Ti-B coatings prepared by high power impulse magnetron sputtering (HIPIMS) at room temperature

Congcong Zhang [a], Ping Zhu [b], Feng Huang [b], Jun Ouyang [a,c,*], Yun Tian [a,**]

[a] Key Laboratory for Liquid–Solid Structural Evolution and Processing of Materials, Ministry of Education and School of Materials Science and Engineering, Shandong University, Jinan 250061, PR China
[b] Ningbo Institute of Materials Technology and Engineering, Chinese Academy of Sciences, Ningbo, Zhejiang 315201, PR China
[c] Suzhou Institute of Shandong University, Suzhou 215123, PR China

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ABSTRACT

In this work, Al-Mg-Ti-B coatings were synthesized at room temperature via high power impulse magnetron sputtering (HIPIMS). The as-deposited Al-Mg-Ti-B coatings exhibited a dense and X-ray amorphous microstructure. It was demonstrated that the chemical composition and mechanical properties of Al-Mg-Ti-B coatings were significantly altered by applying different negative bias voltages during the HIPIMS process. The XPS results showed that oxygen concentration in the coatings was reduced from ~22 at.% to ~8 at.% and the number of oxygen-boron bonds dropped with an increasing bias voltage, which contributed significantly to the improved mechanical properties. Nano-indentation results showed that the nano-hardness and elastic modulus of the HIPIMS Al-Mg-Ti-B coatings were as high as 37 GPa and 348 GPa, respectively.

1. Introduction

As a new class of ultra-hard, light-weight materials, boron-rich borides containing B_{12} icosahedra have attracted intensive research interests for their intriguing functional properties and multifaceted technological applications [1–3]. A typical boron-rich boride, $AlMgB_{14}$, was first reported by Matkovich and Economy in 1970 [4]. Since then there have been many studies on its crystalline structure [5] and various properties [6–11], which include a high electrical conductivity [9] and a good thermal stability [1]. The hardness of $AlMgB_{14}$ ranges between 32 GPa and 35 GPa [1], and the hardness of $AlMgB_{14}$ containing 30 wt% TiB_2 can reach ~46 GPa, which is comparable to that of the cubic BN (c-BN). Thus, $AlMgB_{14}$-based boron-rich borides have potential applications as protective and wear-resistant coatings for cutting tools and microelectromechanical devices [1,6,8,10].

In particular, $AlMgB_{14}$-TiB_2 coatings with a nanocomposite microstructure, $AlMgB_{14}$ and TiB_2 nanocrystals being imbedded in an amorphous matrix, have been systematically studied [11,12]. It has been shown that $AlMgB_{14}$-TiB_2 coatings have high hardnesses (>40 GPa) [12] and can substantially reduce the friction coefficient and wear in tribological processes [13–15]. So far, $AlMgB_{14}$-TiB_2 coatings have been fabricated by pulsed laser deposition (PLD) [10] and magnetron sputtering [12,13,15]. In recent years, a novel physical vapor deposition (PVD) technique-high power impulse magnetron sputtering (HIPIMS) emerged, which can generate a high density plasma containing highly ionized sputtered species by pulsing the target with a high peak power density (e.g. 1–3 kW/cm^2) [16–18]. It is evident that HIPIMS discharges can lead to dense coating structures, strong adhesion as well as smooth coating surfaces [16–20]. Moreover, the high density plasma in a HIPIMS process produces a high ion flux towards the growing coating [20]. The ion bombardment on the growing coating transfers momentum and energy to the adatoms to enhance their surface-diffusion. Therefore, HIPIMS can improve mechanical properties of a coating without external substrate heating [21], which is desirable for the fabrication of many hard coatings with a reduced thermal budget and cost. In order to achieve the desired benefits, an effective control of the strong ion bombardment to the substrate is needed in a HIPIMS process. A natural approach to do this is to apply a negative bias voltage to the substrate to control the flux and kinetic energy of the bombarding ions [20,22,23].

Zhang et al. [23] demonstrated that the chemical composition and microstructure of TiB_2 coatings deposited via HIPIMS were significantly altered by increasing the bias voltage from 0 V to −200 V. Nedfors et al.

* Correspondence to: Jun Ouyang, Key Laboratory for Liquid–Solid Structural Evolution and Processing of Materials, Ministry of Education and School of Materials Science and Engineering, Shandong University, Jinan 250061, PR China.
** Corresponding author.
E-mail addresses: ouyangjun@sdu.edu.cn (J. Ouyang), ytian@sdu.edu.cn (Y. Tian).

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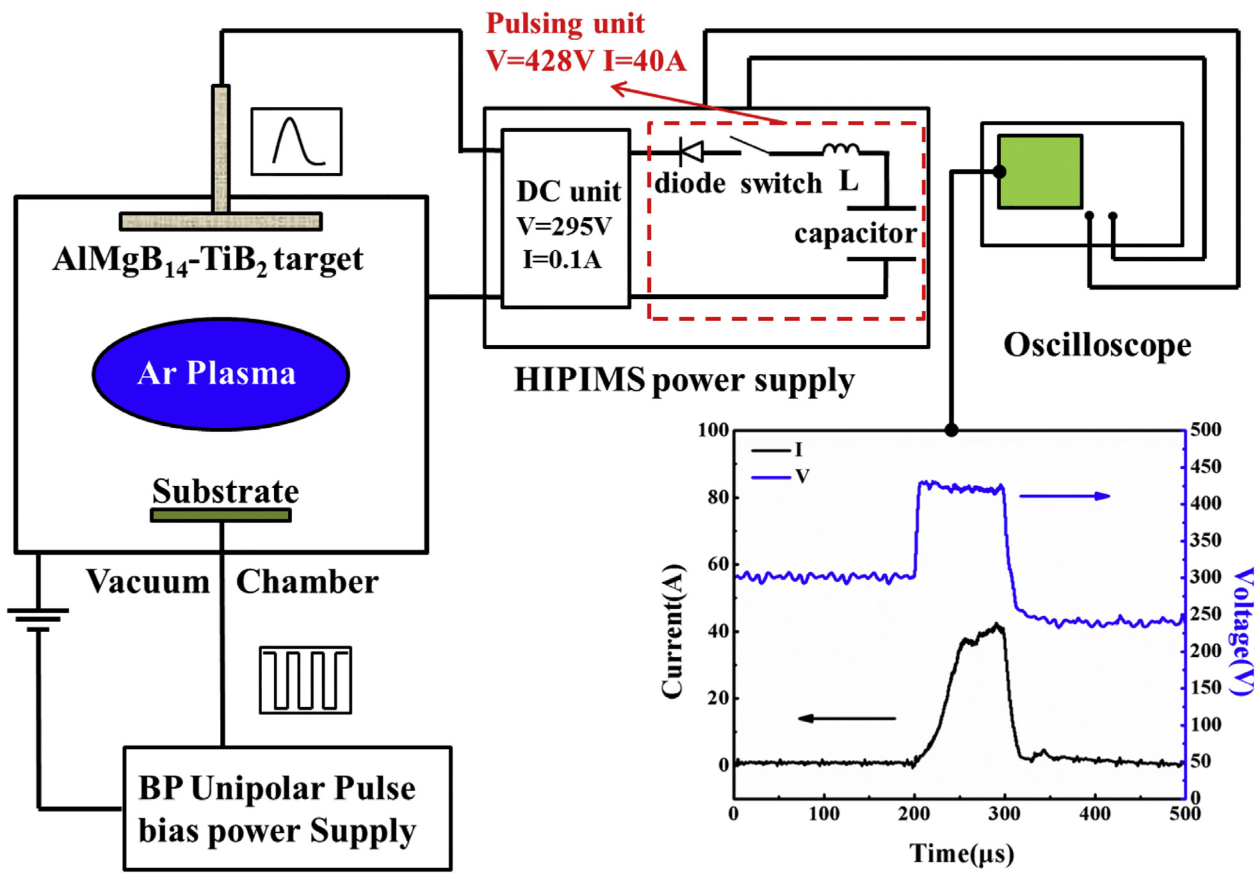

Fig. 1. Illustration of the DCMS-enhanced HIPIMS deposition system.

[22] also reported that, by introducing a bias voltage the (001) texture was promoted in a HIPIMS TiB_2 coating. However, there have been no reports on HIPIMS deposition of $AlMgB_{14}$-TiB_2 coatings. In this work, a $AlMgB_{14}$-TiB_2 target was used to synthesize coatings via a HIPIMS process at room temperature (no external substrate heating) with a bias voltage ranging between 0 V and −150 V. Using the characterization methods of X-ray photoelectron spectroscopy (XPS), X-ray diffraction (XRD), scanning electron microscopy (SEM) and nano-indentation, we investigated the effects of a bias voltage on the chemical composition, microstructure and mechanical properties of the $AlMgB_{14}$-TiB_2 coatings. Note that since both the stoichiometry and microstructure of a PVD coating can be significantly different from its target material, Al-Mg-Ti-B is used instead of $AlMgB_{14}$-TiB_2 in the following sections to represent the coatings being fabricated and studied.

2. Experimental details

The Al-Mg-Ti-B coatings were deposited on low resistance, single crystalline (100) Si substrates (ρ = 1–20 $\Omega\cdot$cm, 10 × 10 mm) by using a HIPIMS process enhanced by direct current magnetron sputtering (DCMS). In Fig. 1, the experimental setup of this DCMS-enhanced HIPIMS process is illustrated. The target was a hot-pressed stoichiometric $AlMgB_{14}$-50 vol% TiB_2 ceramic (Φ = 50 mm, t = 6 mm), and the silicon substrates were mounted on a stationary substrate holder with a target-substrate distance of 90 mm. Prior to the deposition process, the substrates were sequentially cleaned in ultrasonic baths of acetone and alcohol for 5 min each, before being loaded into the vacuum chamber. Then a 8×10^{-4} Pa base pressure was reached in the chamber, and the substrates were sputter-etched at a negative pulsed bias of −600 V (40 kHz and 50% duty cycle) for 10 min. During the depositions, the argon gas flow rate and pressure were kept at 45 sccm and 1.6 Pa, respectively, while the direct current was kept at 0.1 A. To investigate the influence of ion bombardment on the microstructure and mechanical properties of the coatings, bias voltages of 0 V (floating potential), −50 V, −100 V and −150 V were applied to the substrates during different deposition runs. The detailed deposition conditions, including the pulsing parameters of the HIPIMS power supply, were summarized in Table 1.

Grazing-incidence X-ray diffraction (XRD) was carried out to investigate the phase structures of the as-deposited Al-Mg-Ti-B coatings, using a Bruker AXS D8 Advance diffractometer operated with a Cu K_α radiation. The XRD patterns were collected at a grazing angle of 1°. The cross-sectional morphologies of the coatings were observed by using a HITACHI SU-70 Scanning Electron Microscope (SEM). Composition and chemical bonding states of the coatings were analyzed using a Thermo Fisher Scentific ESCALAB 250 X-ray Photoelectron Spectrometer (XPS). Prior to the XPS analysis, the samples were sputter-etched for 20 min to remove surface contamination. Lastly, to evaluate the

Table 1
The deposition parameters for the Al-Mg-Ti-B coatings.

Base pressure				$\leq 8 \times 10^{-4}$ Pa			
Substrate temperature				Room temperature			
Sputtering pressure				1.6 Pa			
Target-substrate distance				90 mm			
Ar gas flow rate				45 sccm			
Bias voltage				0 V, −50 V, −100 V, −150 V			
Deposition time				2 h			
Pulsing parameters	P_a (W)	P_p (kW)	V_a (V)	V_p (V)	I_a (A)	I_p (A)	I_d (A/cm^2)
150 Hz, 1.5% duty cycle	205	16.8	304.3	428	0.5	40	2.04

P_a and P_p are the average and peak target power, V_a and V_p are the average and peak target voltage; I_a and I_p are the average and peak target current, while I_d is the peak target current density.