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Preparation and Characterization of Al-Mg-B thin films by magnetron sputtering

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Abstract. Hard and superlight thin films laminated with boron carbide have been proposed as candidates for strategic use such as armor materials in military and space applications. We prepared Al-Mg-B films by sputter deposition on Si (100) substrates with one $AlMgB_{14}$ target. The films were characterized by X-ray diffraction, atomic force microscope, GD-OES spectroscopy. The results show that films of AlMgB with different compositions have been deposited by changing the target power and deposition temperature.The influences of substrate temperature and sputtering power on the quality of the films are discussed.

Introduction

$AlMgB_{14}$ is of great interest due to its extreme hardness [1,2], low density [3,4], high thermal stability [5], and desirable thermoelectric properties [6,7]. The hardness of $AlMgB_{14}$ materials (with Ti and Si additions) reported by researchers at the Ames Laboratory ranges from 35 to 46 GPa [2], which is comparable to the hardness of cubic-BN. $AlMgB_{14}$ is a very promising candidate as wear-resistant, self-lubricating, and protective coatings for cutting tools and protective coatings for microdevices and microelectromechanical components [8,9].

Up to now, $AlMgB_{14}$ in bulk or powder form have been prepared by several methods [1-7].There are rare reports on its preparation in the thin film form till now. Y. Tian et al[9-12] recently reported the deposition of Al-Mg-B thin films with pulsed laser deposition (PLD) using an alloy composite target on Si (100), SiO_2 and carbon-coated copper grids. Ce Yan et al [13] prepared Al-Mg-B films by sputter deposition using multiple unbalanced planar magnetrons equipped with two boron and one AlMg targets. Zhanling Wu et al[14] prepared Al-Mg-B films by magnetron sputtering on Si (100) substrates with a three target. But the stoichiometric hard boride $AlMgB_{14}$ orthorhombic structure has not been prepared,in this paper, we started to prepare Al-Mg-B thin films by magnetron sputtering with one $AlMgB_{14}$ targets. Study the effects of the deposition parameters in order to obtain Al-Mg-B thin films with optimum properties. Depositing high-quality Al-Mg-B thin films by magnetron sputtering would lay a good foundation for practical applications of Al-Mg-B thin films.

Experimental methods

Al-Mg-B thin films were deposited on p-type Si (100) substrates with one $AlMgB_{14}$ target magnetron sputtering system in an argon (Ar) atmosphere. This apparatus (FJL560Ⅱ) is designed to synthesize new materials by sputtering a target. A sintered $AlMgB_{14}$ (purity: 99.95%) disk with the diameter of 60 mm and the thick of 3mm were used as the targets. A substrate holder was located 80 mm away from the target. Prior to the deposition, the substrates were cleaned in ultrasonic baths of methanol and distillated water and were etched in a 5% hydrofluoric solution to strip off the native oxide. Afterwards, they were dried by nitrogen gas and placed into the vacuum chamber immediately. Furthermore they were sputter-cleaned in argon discharge with the negative bias voltage of 650 V for 10 min. The targets were also pre-sputtered for 10 min. The detailed deposition parameters are listed in Table 1 and Table 2.A sets of samples were prepared: sample a-d deposited under sputtering power 30W at room temperature,400℃,500℃,600℃.Sample e-g deposited under the boron sputtering power of 50W and 100 W at room temperature.

The XRD patterns of Al-Mg-B films were collected by D8 ADVANCE BRUER AXS X-ray diffractometer using Cu Kα X-rays and a small incident angle geometry. GD-OES electron micro-probe analysis was used for composition analysis of Al-Mg-B thin films. Atomic force microscopy (AFM)(CSPM5000) was performed to obtain the surface morphology of these films.

Table 1
Deposition parameters of Al-Mg-B thin films by magnetron sputtering.

Base pressure	5.0 X10^{-4}Pa
Working pressure	5.0 X10^{-1}Pa
Ar flow rate	20 sccm
Deposition time	2 h
RF power of AlMgB target	30W
Substrate temperature	25℃, 400℃,500℃,600℃

Table 2
Deposition parameters of Al-Mg-B thin films by magnetron sputtering.

Base pressure	5.0 X10^{-4}Pa
Working pressure	5.0 X10^{-1}Pa
Ar flow rate	20 sccm
Deposition time	1h
RF power of AlMgB target	30W,50W,100W
Substrate temperature	25℃

Results and discussion

1. X-ray diffraction

Target is measured by X-ray diffraction shown in Fig.1.The presence of $AlMgB_{14}$ can be seen as a broad peak in the region of 41.5^o-43^o(2θ)[15]. The major peaks are corresponding to $AlMgB_{14}$ structure.The major crystalline phases in the target are $AlMgB_{14}$, the rest impurity phases were $MgAl_2O_4$.

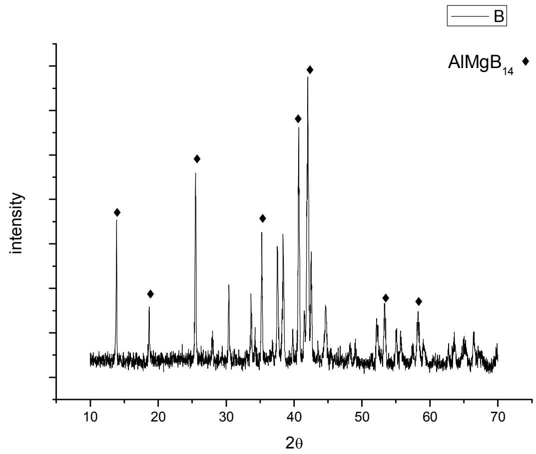

Fig.1XRD spectra of $AlMgB_{14}$ target at 1500 under 60Mpa

Seven samples of Al-Mg-B thin films were measured by X-ray diffraction, and they showed almost different X-ray diffraction patterns, even if they were prepared at the same sputtering power of 30W and different substrate temperatures. Fig.2 is the XRD pattern of sample a-d for comparison. There is no obvious other diffraction peak corresponding to the ternary boride structure for sample d (600℃). This indicates that the films are X-ray amorphous. For sample c(500℃) there are two weak diffraction

peak at the θ–2θ scan of 15° and 33.7°,it is no obvious other diffraction peak corresponding to the ternary boride structure. The film gives featureless XRD patterns characteristic to amorphous structures.Sample b(400℃) have similar patterns that show diffractions peak at around 33.7°and 55-57°, implies the formation of some nanocrystals inside the film. The peak at 33.7° can be correlated with the diffraction from the (211) crystallographic planes of the AlMgB14 orthorhombic crystal structure. In the $AlMgB_{14}$ structure, the (211) peak appears along with the strongest peaks, i.e., the (213) peak at 42.03°, (011) peak at 13.89°, and (132) peak at 40.69°. These peaks are usually present in nearly stoichiometric AlMgB14 structure[13],but they are absent in the film. Thus in the presented diffraction patternof the sample b(400℃), the weak peak at 33.7° may possibly originate in the texturing of the nanocrystals in the thin film.Sample a(25℃) is no obvious other diffraction peak corresponding to the ternary boride structure. This indicates that the films are X-ray amorphous.

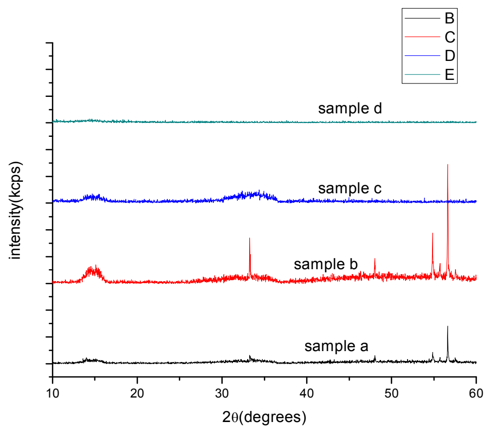

Fig.2 XRD spectra of Al-Mg-B thin films deposited at different temperature under 30W sputtering power

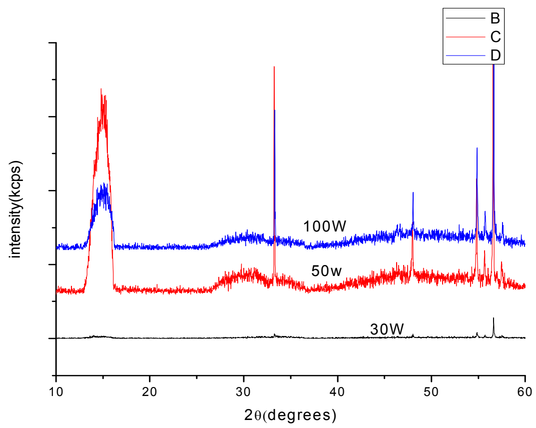

Fig.3 XRD spectra of Al-Mg-B thin films deposited at different sputtering power under 25℃

Fig.3shows XRD patterns by sputter deposition films under different sputtering power (samples E, Fand G). The XRD analysis of the 30W film is similar to the sample a. The diffraction peak of the 50W film have similar patterns with the 100W film that show strong diffractions at around 33.7°and 55-57°, implies the formation of some nanocrystals inside the film. The peak at 33.7°can be correlated with the diffraction from the (211) crystallographic planes of the $AlMgB_{14}$ orthorhombic crystal structure. Fig.3 show strong diffractions at around 13.89°,but the peak is wider indicating amorphous structures. For the 50W and 100W samples, an additional strong peak at around 57° is observed, the peak shape, the rule, the peak width is narrow,indicating nanocrystalline structures. This peak, which does not correspond to the $AlMgB_{14}$ structure, suggests new phase formation at this sputtering power. Fig. 3 shows crystallization degree of 50W and100W thin films is higher than 30W thin films .

2 Surface morphologies(AFM)

Fig.4 shows the AFM photographs of the surface morphologies of the Al-Mg-B thin films. Four samples all displayed the surface profile dominated by domed features. The surface of samples is dimple rugged. From Fig.4, we can observe that the surface smoothness of the films varies with changing the substrate temperature. The film prepared at 600℃ has smoother surface than that at room temperature. The dependence of surface roughness on the substrate temperature can be explained by the migration and the diffusion of the reactants on the substrate surface. At lower substrate temperature, atoms absorbed on the substrate surface have lower mobility, so the films showed a rougher surface. With increasing the substrate temperature, the enhancement of the migration ability of the atoms results in the increase of the surface smoothness[14].From the above results, we found that the surface smoothness of Al-Mg-B thin films is strongly dependent on the substrate temperature.

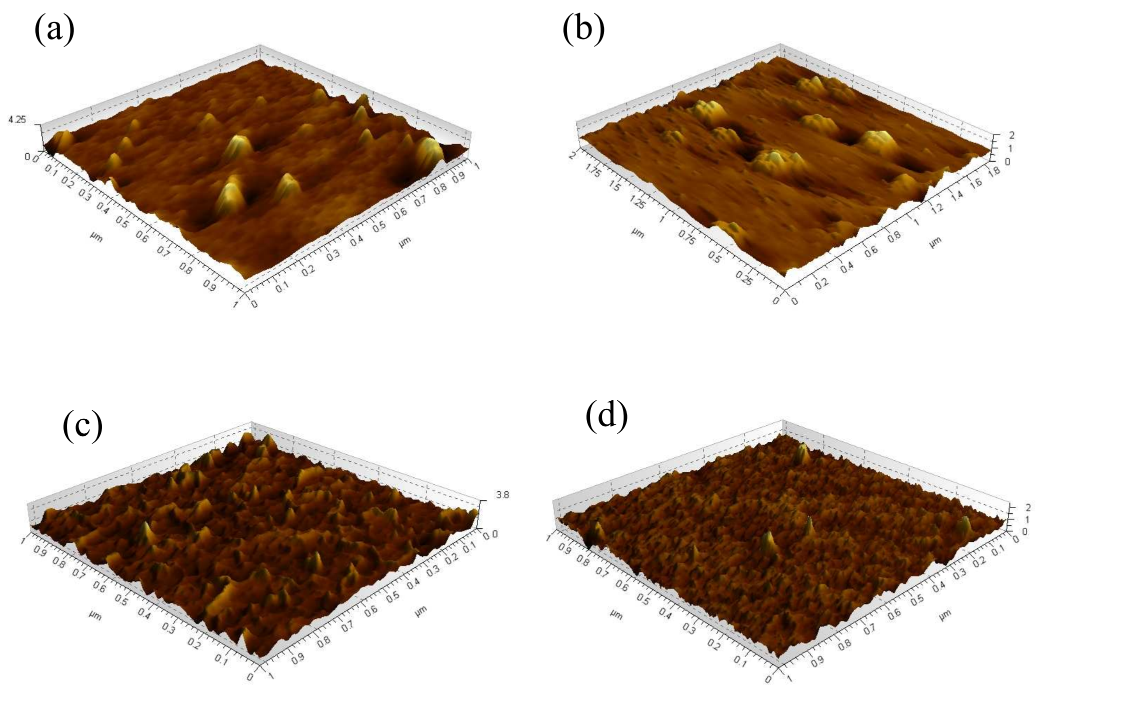

Fig. 4. AFM surface morphologies of Al-Mg-B thin films deposited at room temperature ,400 ℃,500℃,600℃ under the sputtering power 30W : (a)room temperature, (b)400℃, (c)500℃, (d)600℃ ,respectively.

3 GD-OES electron micro-probe analysis

Fig.5 shows element distribution curves of Al-Mg-B thin films deposited at room temperature under 100W sputtering power. Fig.a shows element distribution curves of element weight percentage.Fig.b shows element distribution curves of element atomic percentage. we can observe that metal-rich Al-Mg-B films are shown in Fig. 5, which does not correspond to the $AlMgB_{14}$ structure.

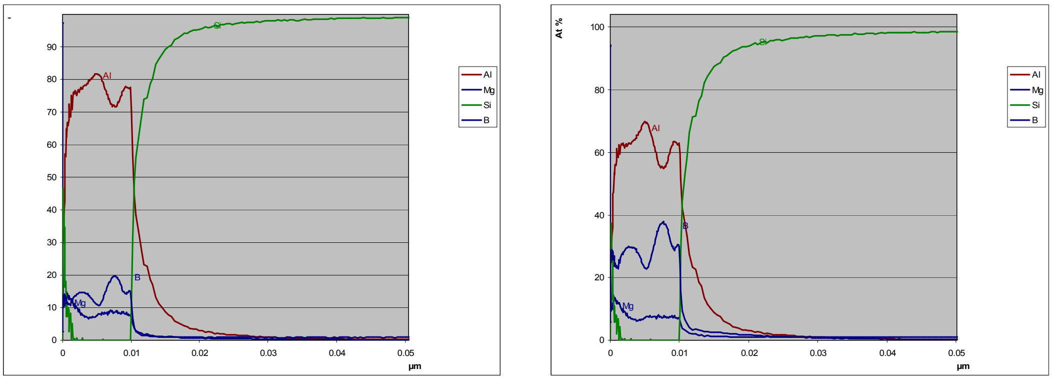

Fig .5 Element distribution curves of Al-Mg-B thin films deposited at room temperature under 100W sputtering power.(a) element distribution curves of W%;(b) element distribution curves of At%

Summary

In summary, Al-Mg-B thin films were successfully prepared by magnetron sputtering using a AlMgB target. Films of AlMgB with different compositions have been deposited by changing the target power and deposition temperature. The analyses results indicate that the surface of the films becomes smoother with increasing the substrate temperature. Metal-rich AlMgB composite films have been prepared at room temperature and100W,respectively.While the stoichiometric hard boride $AlMgB_{14}$ orthorhombic structure has not been prepared.The optimized preparation parameters must be addressed in any subsequent study.

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