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AlMgB14-Based Films Prepared by Magnetron Sputtering at Various Substrate Temperatures

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The films were deposited by magnetron sputtering the $AlMgB_{14}$ target at different substrate temperatures (T_S) in the range of 100-500 °C. The films were annealed at 1000 °C in vacuum. The deposited films were characterized by XRD, AFM, FTIR spectroscopy, nano- and micro-indentation and scratch testing. The films exhibit hardness that is much lower than the one of the bulk $AlMgB_{14}$ materials, which is due to the amorphous film structure in which the strong B-B bonds are absent and the weaker B-O bonds dominate.

Keywords: $AlMgB_{14}$, Magnetron Sputtering, Infrared Spectroscopy, Hardness, Tribological Properties.

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1. INTRODUCTION

The hard bulk materials based on $AlMgB_{14}$ (BAM) are intensively studied during last decade owing to their extreme hardness (35-46 GPa), low friction coefficient and chemical inertness [1,2]. However, the investigation of the films based on BAM is in an infant stage. In particular, these films were prepared mostly by pulsed laser deposition, magnetron sputtering of several targets that contain the components of $AlMgB_{14}$ [3].

In this work we investigate the films deposited by magnetron sputtering the $AlMgB_{14}$ target at different substrate temperatures (T_S) in the range of 100-500 °C. The films were characterized with XRD, FTIR spectroscopy, nano- and micro-indentation and scratch testing.

2. EXPERIMENTAL

2.1 Film Preparation

The films were deposited by magnetron sputtering the $AlMgB_{14}$ target prepared by hot pressing at different substrate temperatures T_S = 100, 200, 350, 450 and 500 °C. The substrates were polished Si (100) wafers. The substrate bias was -50 V. The argon flow rate, working pressure and DC discharge power were 51 sccm, 0.17 Pa and 7.1 W/cm^2, respectively. The basic pressure approximates 10^{-3} Pa. The film deposited at 450 °C was annealed in vacuum (~10^{-3} Pa) at 1000 °C during two hours.

2.2 Film Characterization

The films were characterized with XRD, FTIR spectroscopy, nano- and micro-indentation and scratch testing. X-ray diffraction (XRD) investigations of the films were carried out by using a diffractometer “DRON-3M”. The films surface was studied by an atomic-force microscope (AFM) “NanoScope IIIa Dimension 3000TM”. The chemical bonding was studied by Fourier traform infrared spectroscopy (FTIR) with the help of a spectrometer “FSM 1202” LLC “Infraspek”. Nanoindentation was carried out with the help of a device G200 equipped with the Berkovich indenter. Nanohardness (H) and elastic modulus (E) were determined using the Oliver and Pharr procedure [3]. Knoop hardness (HK) was determined by a device “MICROMET 2103 Microhardness Tester” (BUEHLER, USA) at loading of 10 mN. The thickness of the films was estimated by an optical profilometer "Micron - alpha" (Ukraine). The film thicknesses approximate 0.6-0.8 μm, and slightly decrease with increasing T_S. The scratch tests were performed with the help of a scratch tester “Micron-gamma” (Ukraine) by using the Vickers diamond pyramid that moves with the velocity of 9 μm/c under increasing loading from 0 to 0.3 mN.

3. RESULTS AND DISCUSSONS

In Fig. 1 we show the XRD spectra of the deposited and annealed films. One can see that the as-deposited films do not show any reflexes related to crystallites, and, correspondingly, they are X-ray amorphous. The reflexes around $2\Theta \approx 12^0$ and 25^0 in the XRD spectrum of the annealed film point to the presence of the crystallites of the rhombohedral or tetragonal boron [PDF: 011-0618, 012-0377, 031-0206, and 031-0207]. So, the annealing at 1000 °C leads to the formation of the boron crystalline islets in the amorphous matrix. The crystallites related to boron oxides were not revealed.

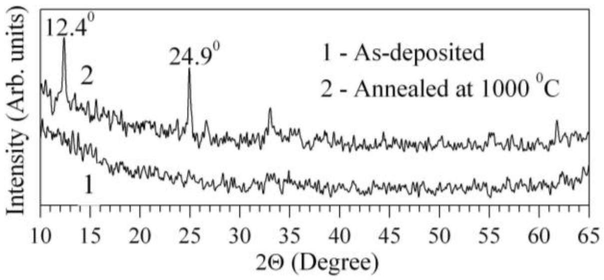

Fig. 1 – XRD patterns of the as-deposited and annealed at 1000 ^{0}C films.

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The investigation of the AFM images of the film surfaces (not shown here) points out that, for the films deposited at 100 °C and 500 °C , the RMSs determined at the area of 5 μm × 5 μm were 1.6 nm and 1.0 nm, respectively. These data indicate that an increase of substrate temperature promotes a reduction of the roughness of the film surface.

The FTIR spectra of the films deposited at different T_S are presented in Fig. 2. The numerals denote the wavenumbers of the absorption bands related to the B-O-B bending and B-O stretching vibrations in the tetragonal BO_4 and trigonal BO_3 units [4]. We see that an increase in T_S leads to reducing the intensity of B-O vibrations.

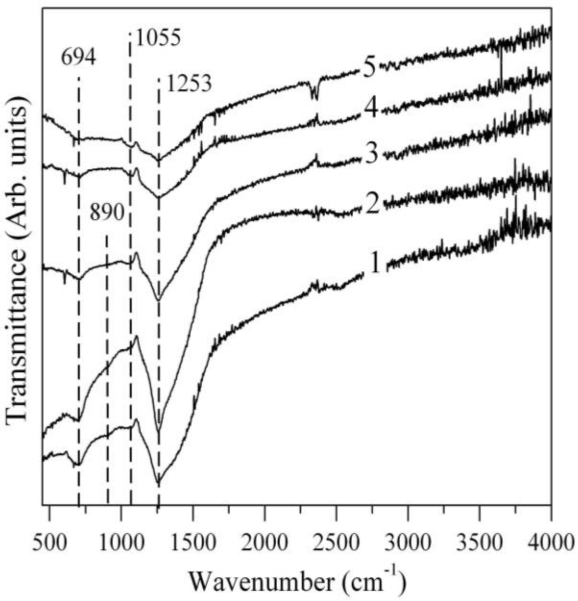

Fig. 2 – FTIR spectra of the films deposited at T_S =100 (1), 200 (2), 350 (3), 450 (4) and 500 °C

The nanohardness and elastic modulus increase with T_S and at annealing (cf Fig. 2). The Koop hardness increases from 8.3 GPa to 17.9 GPa, the friction coefficient decreases from 0.12 to 0.08 and the adhesion of the films to silicon substrates strengthens when T_S increases from 100°C to 500°C (not shown here). It follows that mechanical properties improve at the annealing, and when substrate temperature increases due to reducing the B-O bonds (cf. Fig. 1). The films exhibit the hardness that is much lower than the one of the bulk BAM materials [1], which is due to the amorphous film structure in which the strong B-B bonds are absent and the weaker B-O bonds dominate.

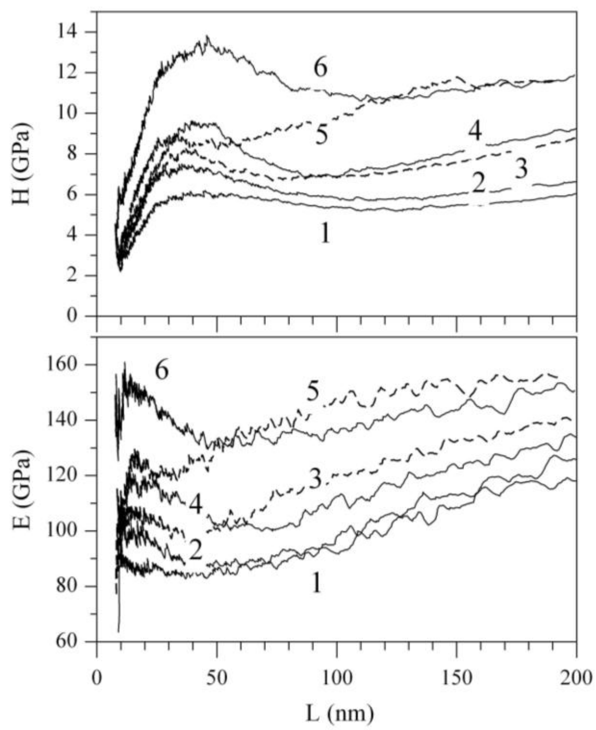

Fig. 3 – Nanohardness (H) and elastic modulus (E) as functions of indenter penetration (L) for the films deposited at various T_S (cf. Fig. 2). The curve 6 corresponds to the sample 4 annealed at 1000 °C.

4. CONCLUSIONS

Al-Mg-B films were deposited on silicon wafers by DC magnetron sputtering at different substrate temperatures in the range of 100-500 °C. The as-deposited films were X-ray amorphous. The formation of boron crystallites occurs during annealing at 1000 °C. An increase in substrate temperature leads to reducing film surface roughness, friction coefficient and to increasing hardness, elastic modulus and film adhesion. The films exhibit the hardness (6-14 GPa) that is much lower than the one of the bulk BAM materials, which is due to the amorphous film structure in which the strong B-B bonds are absent and the weaker B-O bonds dominate. The hardness of the annealed film is higher compared to the hardness of the as-deposited films, which can be explained by the presence of boron crystallites in the annealed film. It was shown that the improvement of the mechanical properties of the films with increasing substrate temperature and after annealing is due to reducing the B-O bonds.

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