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Influence of high power impulse magnetron sputtering pulse parameters on the properties of aluminum nitride coatings

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ABSTRACT

The aluminum nitride thin film has recently gained importance due to its unique properties, which include good thermal and chemical stabilities as well as dielectric property. In this work, aluminum nitride thin films were fabricated using the high power impulse magnetron sputtering (HIPIMS) technique. Effects of duty cycle and pulse frequency of HIPIMS power on the microstructure and mechanical property evolution of AlN coatings were explored. The ionization ratio of Al^+ species in HIPIMS plasma was characterized to be around 55%. The peak power density increased to 1.60 kW cm^{-2} as duty cycle decreased to 3.5%. The deposition rate increased from 1.74 to 4.50 nm/min as repetition frequency reached to 833 Hz at a fixed duty cycle of 3.5%. Strong preferred orientation of hcp-AlN (002) was discovered for AlN films with lower oxygen contamination. In addition to the hcp-AlN phase, the α-Al_2O_3 phase was found when the oxygen concentration was higher than 3.0 at.%. The hardness enhancement effect was brought by the HIPIMS technique. The hardness increased with decreasing duty cycle and increasing peak power density. Greater hardness of around 26.5 to 28.0 GPa was achieved for the crystalline over-stoichiometric AlN thin film when the coating was fabricated using a 3.5% duty cycle. Meanwhile, the adhesion property was improved with increasing duty cycle and frequency. The AlN coating with both high hardness of 27 GPa and adequate adhesion property was fabricated at a repetition frequency of 1250 Hz and duty cycle of 3.5%.

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1. Introduction

Aluminum nitride thin films have been extensively investigated and applied in industries due to its unique thermal, piezoelectric, insulating and mechanical properties as well as its large optical band gap [1–5]. Many kinds of fabrication techniques, such as chemical vapor deposition (CVD) [6], pulsed laser deposition (PLD) [7], reactive magnetron sputtering [8–12], and reactive vacuum arc deposition [13] were employed to fabricate high quality AlN thin films.

The high power impulse magnetron sputtering (HIPIMS) technique was first reported by Kouznetsov et al. [14] presenting a high peak power density of 2800 W cm^{-2} and 70% ionization during Cu film synthesis in 1999. Since then, major effort and resources have been devoted to the research work on the HIPIMS technology, including analysis of the physics, plasma diagnosis, and deposition parameter [15–19]. In general, the HIPIMS technique can provide very high target peak current, high peak power density, of 0.5–10 kW cm^{-2}, and high ionization rate due to its rather low duty cycle (0.5–5%), and short pulse on-time ranging from several to thousands of μs at the frequency range of 50 to 5000 Hz [15]. Smooth surfaces, dense microstructures, and better mechanical properties are all advantages that the HIPIMS technique can bring through the proper adjustments of pulse on/off time configuration.

The study of AlN coating grown by HIPIMS technique, however, is limited [20–22]. Guillaumot et al. [20] studied the effect of nitrogen partial pressure on the fabrication of AlN coating. The hardness of stoichiometric hcp-AlN coating of around 18 and 8 GPa by HIPIMS and pulsed DC sputtering were reported, respectively. Jouan et al. investigated the ion energy distribution of HIPIMS plasma during the AlN deposition [21]. Better crystallized AlN coatings were obtained due to the large fraction of Al^+ ionic species coming from the target. Aissa et al. [22] explored the influence of working pressure on the characteristics of AlN films by conventional DC magnetron sputtering process versus HIPIMS technique. They discovered that the AlN films grown by HIPIMS had lower deposition rates and suffered from high residual stress.

Since the study of AlN coating grown by HIPIMS technique, especially the effects of duty cycles and frequencies on the mechanical properties of AlN film is not yet discovered clearly, the duty cycle, repetition frequency of target power, and substrate bias voltage were adjusted to fabricate six AlN coatings in this work. The optical emission signal and target current–voltage waveforms during HIPIMS process were

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recorded and discussed. The relationships between the duty cycle, repetition frequency, and film properties, including chemical composition, crystalline structure, microstructure, and mechanical properties were further explored in this work.

2. Experimental details

A high power impulse magnetron sputtering (HIPIMS) system was employed to deposit AlN thin films on Si wafers and hardened AISI420 disk substrates in this study. A detailed description of the HIPIMS system has been reported elsewhere [23]. A 3″ diameter pure aluminum (Al, 99.99 at.%) target was connected to a SPIK2000A pulse power supply (SPIK 2000A, Shen Chang Electric Co., Taiwan) operated in the unipolar negative mode at a constant power of 600 W. The pulse on-time, t_{on}, and pulse off-time, t_{off}, were adjusted to achieve various repetition frequencies ($1 / (t_{off} + t_{on})$) and duty cycles ($t_{on} / (t_{off} + t_{on}) \times 100\%$) ranging from 833 to 1250 Hz and 3.5 to 5%, respectively. The substrate-to-target vertical distance was kept at 12 cm. The chamber was pumped to a base pressure of 6.7×10^{-4} Pa. A gas mixture of Ar and N_2 at the ratio of 1:2 was used to maintain the working pressure of 0.67 Pa and to achieve a high crystallinity and c-axis oriented structure [13]. The deposition temperature was kept at 200 °C. A direct current (DC) power supply was used to apply a −30 V bias voltage to the substrate holder for samples N1 to N5, whereas a +150 V DC bias was applied for N6 to determine the ionization rate of Al target. A digital oscilloscope (DS 5205CA, RIGOL, USA) combined with a high voltage differential probe (SI-9010, Sapphire Instruments Co. Ltd., Taiwan) and a current probe amplifier (80I-110S, FLUKE, France) were used to measure the voltage and current waveforms of the target during sputtering. The ion eroded Al target surface after each sputtering process was examined and the ion eroded area, of around 26.0 to 27.0 cm^2, was measured using a digital caliper. The peak power determined from the digital oscilloscope was further divided by the ion eroded area to determine the peak power density of each deposition process. In general, the ion eroded area of Al target after DC sputtering was smaller than that of HIPIMS sputtered one. An optical spectrometry system (PLASUS EMICON, Germany) was connected to the sputtering system to monitor the plasma emission spectrum during deposition process. Before deposition, each sample was plasma etched with a DC bias discharge of −300 V for 15 min under an argon atmosphere. The sample designation and detailed sputtering parameters for each coating are listed in Table 1. The deposition time for each coating was in the range of 30 to 120 min and was also listed in Table 1. The crystalline structures of thin films were explored by an X-ray diffractometer (GA-XRD, PANalytical, X'pert, Holland) using the θ − 2θ powder mode and the thin film mode with an incidence angle of 1°. Cu Kα radiation generated at 40 kV and 30 mA from a Cu target was used. Elemental distribution of selected coating was evaluated with an Auger electron spectroscope (AES, PHI700, ULVAC-PHI, Japan) by the depth profiling technique. A beam energy of 5 keV was used to obtain the Auger spectra.

The cross-sectional morphologies of thin films were examined with a field emission scanning electron microscope (FE-SEM, JSM-6701, JEOL, Japan) operating at an acceleration voltage of 10 kV. Selected samples were further analyzed by transmission electron microscopy (TEM, JEOL, JSM-2100, Japan). The chemical composition of thin films was analyzed with a field emission electron probe microanalyzer (FE-EPMA, JXA-8500F, JEOL, Japan) operating at 12 kV. The thin films were coated on AISI420 disk substrates for the hardness and scratch tests. The surface roughness values of coatings deposited on Si wafers were measured by atomic force microscopy (AFM, DI-3100, Bruker, USA). The nanohardness and elastic modulus of AlN films were investigated by means of a nanoindenter (TI-900, TriboIndenter, Hysitron, USA), equipped with a Berkovich 142.3° diamond probe tip, under a displacement control mode. The displacement control mode was used to achieve the maximum indentation depth of around 70 nm to minimize the substrate effect. The loading rate was 1000 μN/s. Eight indentation tests were performed for each coating. The hardness and elastic modulus of each indent were determined on the basis of the Oliver and Pharr method [24]. The elastic modulus, E, was expressed as follows:

$$\frac{1}{Er} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i} \qquad (1)$$

where E_r and ν are the reduced elastic modulus and Poisson's ratio, respectively, for the thin film under test, and E_i (1140 GPa) and ν_i (0.07) are the corresponding parameters of the diamond indenter. The Poisson's ratio of 0.234 was used for AlN coating [25]. The fused quartz standard sample was used to calibrate the area function of the nanoindenter.

Table 1
Sample designation, typical deposition conditions, chemical composition and mechanical property characteristics for AlN thin films.

Sample designation		DC	N1	N2	N3	N4	N5	N6
Frequency (Hz)		DC	1000			1250	833	1000
t_{on} time (μs)			50	40	35	28	42	35
t_{off} time (μs)			950	960	965	772	1158	965
Duty cycle (%)			5	4	3.5	3.5		
Avg. target current (A)		2.18	1.51	1.45	1.34	1.32	1.4	1.36
Avg. target voltage (V)		278	402	419	452	458	433	444
Peak current (A)		1.33	80	92	94	94	96	100
Input gas (sccm)		Ar: 10, N_2: 20						
Substrate DC bias (V)		−30						+150
Peak power density (kW cm^{-2})		0.02	1.21	1.45	1.60	1.62	1.57	1.67
Deposition time (min)		30	60	75	120			
Film thickness (nm)		220.2	239.6	263.6	460.2	374.5	538.9	209.0
Deposition rate (nm/min)		7.34	3.99	3.51	3.84	3.12	4.50	1.74
Chemical composition (at.%)	Al	29.4	31.4	32.0	38.2	36.1	39.7	31.0
	N	59.0	63.5	58.3	58.8	58.1	58.4	60.0
	O	11.6	5.1	9.8	3.0	5.8	1.9	8.9
Al/(Al + N) atomic ratio		0.33	0.33	0.35	0.39	0.38	0.40	0.34
Crystalline phase		h-AlN, c-AlN, α-Al_2O_3	h-AlN, α-Al_2O_3	h-AlN, α-Al_2O_3	h-AlN, α-Al_2O_3	h-AlN, c-AlN, α-Al_2O_3	h-AlN	h-AlN, α-Al_2O_3
Surface roughness, Ra (nm)		1.06	3.16	5.23	17.87	7.11	10.30	7.36
Residual stress (GPa)		+1.12	−3.57	−1.95	−1.74	−2.04	−1.78	+0.15
Hardness (GPa)		12.7	20.4	21.5	28.0	27.0	26.5	9.4
Elastic modulus (GPa)		264	270	250	304	273	302	218
H/E ratio		0.05	0.08	0.09	0.09	0.10	0.09	0.04
Critical load (N)		39.7	22.5	19.0	13.4	24.1	13.2	–

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