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The influence of deposition parameters on the structure and properties of aluminum nitride coatings deposited by high power impulse magnetron sputtering

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ABSTRACT

In this work, aluminum nitride (AlN) thin films were fabricated using the high power impulse magnetron sputtering (HIPIMS) process through the Taguchi method to determine the optimum deposition condition. A L9 array, signal-to-noise (S/N) ratio, and analysis of variance (ANOVA) were applied to study the deposition parameters (pulse frequency, duty cycle, temperature, and substrate bias) with consideration of the microstructure, hardness and transmittance properties of AlN coatings. The wurtzite hexagonal AlN structure was achieved for each coating. Typical fine and dense columnar structure was observed for AlN coating. Based on the higher the better concept using S/N ratio and the contribution using ANOVA, the substrate bias was the most influential parameter for the hardness of AlN coating. On the other hand, the duty cycle was important proportionally for the transmittance of AlN coatings. Confirmation tests with optimal deposition parameters were performed to verify the effectiveness of the Taguchi optimization analysis in this work. The maximum hardness of 25 GPa and transmittance of 83.4% were achieved, respectively, for the AlN coatings deposited with optimal deposition parameters.

1. Introduction

High power impulse magnetron sputtering (HIPIMS), or the so-called high power pulsed magnetron sputtering (HPPMS) is a newly developed coating technology since 1991 [1] and is characterized by its ultra-high peak current and peak power density to achieve unique thin film properties, such as high hardness, good adhesion and tribological performance [2,3]. In general, the HIPIMS technique can provide very high target peak current, high peak power density of several kW/cm^2 and high ionization rate due to its rather low duty cycle (<10%), short pulse on-time ranging from several to thousands of μs at the frequency range of 10 Hz to 10 kHz [4–6]. Recently, the application of the HIPIMS system is widely studied in almost all kinds of coatings including the fabrication of antibacterial Ag film [7], diamond like carbon [8], TiO2 [9], AlN [10,11], AlCrN [12] and CrN/AlN [13] nanolaminated thin films. The advantages of smooth surfaces, dense microstructures and better mechanical properties have been brought by the HIPIMS technique through the proper adjustments of pulse on/off time [12]. The configuration of aluminum nitride thin film has been extensively investigated due to its useful thermal, piezoelectric, optical and mechanical properties [14,15]. Many kinds of fabrication techniques, such as the chemical vapor deposition (CVD), pulsed laser deposition (PLD), and reactive magnetron sputtering [16,17] had been developed. However, the study of AlN coating grown by HIPIMS technique is rare and only limited information were reported [10,11,13].

In order to improve the hardness and transmittance of AlN coating by HIPIMS, the traditional method for optimizing process parameter values requires a number of combinations of experiments by changing one variable at a time, which is time, cost, and labor intensive. The Taguchi method is a systematic and efficient approach for the experimental design in determining the optimal process parameters to improve the desired quality properties of product [18–21]. Taguchi analysis applies a design of orthogonal arrays that provides a maximum number of main factors to be estimated in an orthogonal fashion based on a minimum number of experimental trials. The responses of the experimental trials are further transferred into a signal-to-noise (S/N) ratio to determine the optimal set of process parameters. In addition to the S/N ratio, a statistical analysis of variance (ANOVA) can be employed to indicate the contribution of process parameters on the preferable quality of AlN films. The applications of Taguchi principles and concepts have made extensive contributions to industry by bringing focused regard to performance, quality, and cost [18,21].

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Table 1
Deposition parameters of AlN films, factors and levels.

Symbol	Process parameter	Level 1	Level 2	Level 3
A	Frequency (Hz)	750	1000	1250
B	Duty cycle (%)	2.5	3	3.5
C	Deposition temperature (°C)	25	100	200
D	Substrate bias (V)	−50	−30	0

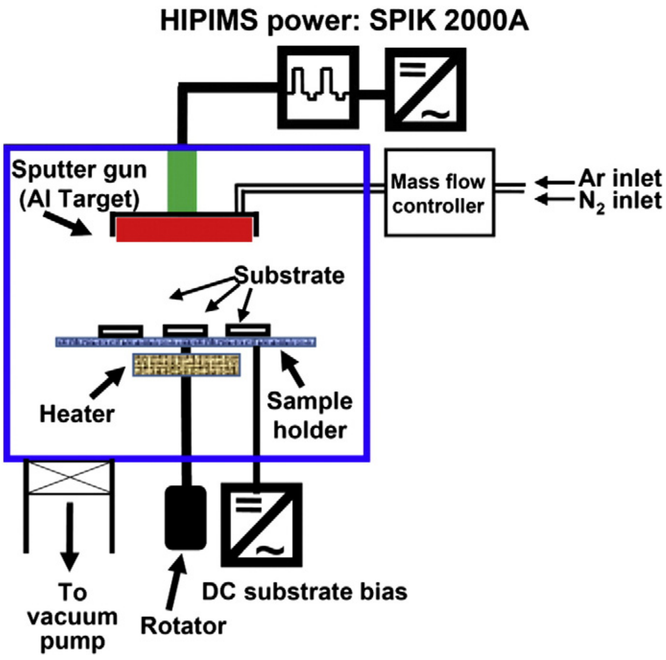

Fig. 1. A schematic illustration of the applied HIMIPMS device and the sputter unit.

Table 2
Deposition conditions for AlN films on the Taguchi orthogonal array table L9. Letters and numbers reflect the parameters and levels from Table 1.

Sample #	Control factors[a]			
	A	B	C	D
S1	1	1	1	1
S2	1	2	2	2
S3	1	3	3	3
S4	2	1	2	3
S5	2	2	3	1
S6	2	3	1	2
S7	3	1	3	2
S8	3	2	1	3
S9	3	3	2	1

[a] Letters and numbers reflect the parameters and levels from Table 1.

In this study, AlN coatings were fabricated by HIPIMS technique. Taguchi L9 orthogonal array was designed and used to analyze the individual and combined effects of HIPIMS process parameters, including the repetition frequency of target power, the duty cycle, the deposition temperature, and the substrate bias voltage. In addition, orthogonal arrays of Taguchi, the S/N ratio, and the ANOVA are employed to find out the optimal levels and to analyze the effect of the HIPIMS process parameters on the hardness and transmittance property of AlN coatings. Furthermore, confirmation test with the optimal levels of HIPIMS parameters for the hardness and transmittance was carried out, individually, in order to illustrate the effectiveness of Taguchi's optimization approach.

2. Experimental details

A high power impulse magnetron sputtering (HIPIMS) system was employed to deposit AlN thin films on Si wafer and hardened AISI420 disk substrates in this study. 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 500 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 cycle $(t_{on} / (t_{off} + t_{on}) \times 100\%)$ ranging from 750 to 1250 Hz and 2.5 to 3.5%, 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 2.5:1 was used to maintain the working pressure of 0.67 Pa. The deposition time was 2 h for each coating. The design of the Taguchi orthogonal array L9 is listed in Table 1. The deposition temperature was ranging from 25 °C to 200 °C. The direct current (DC) bias voltage ranged from −50 V to 0 V. A schematic illustration of the applied HIMIPMS device and the sputter unit is shown in Fig. 1. 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, respectively, during sputtering. The ion eroded Al target surface after each sputtering process was examined and the ion eroded area, of around 22.5 to 23.5 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. It seems that the eroded area is not strongly related to the peak power density. Before deposition, each sample was ion etched with bias discharge of −350 V DC for 15 min under argon atmosphere.

The Taguchi experimental design was applied to analyze the influence of the deposition parameters on the structure and properties of AlN coatings deposited by HPIMS. Table 1 shows that four control factors were studied: frequency (A), duty cycle % (B), substrate temperature (C), and substrate bias (D). Three levels were considered for each factor. According to the Taguchi design concept L9 orthogonal array is developed in this study and the structure of the array is present in

Table 3
Sample designation and typical deposition conditions for AlN thin films.

Sample #	S1	S2	S3	S4	S5	S6	S7	S8	S9
A, (Frequency (Hz))	750	750	750	1000	1000	1000	1250	1250	1250
t_{on} time (μs)	34	40	47	25	30	35	20	24	28
t_{off} time (μs)	1299	1293	1286	975	970	965	780	776	772
B, (Duty cycle (%))	2.5	3	3.5	2.5	3	3.5	2.5	3	3.5
Avg. target current (A)	0.91	0.98	1.02	0.91	0.96	1.00	0.88	0.96	1.01
Avg. target voltage (V)	553	522	500	568	536	510	596	546	510
Peak current (A)	80	68	60	76	68	60	76	68	56
Peak power density (kW/cm^2)	1.95	1.57	1.32	1.9	1.6	1.35	1.99	1.64	1.26
Sputter gas (sccm)	Ar:25								
Reactive gas (sccm)	N_2:10								
C, (Deposition temp. (°C))	25	100	200	100	200	25	200	25	100
D, (Substrate bias (V))	−50	−30	0	0	−50	−30	−30	0	−50