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Effects of superimposed pulse bias on TiN coating in cathodic arc deposition

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Abstract

Orthogonal designs are used to investigate the main factors when doing experiments in which pulse bias is superimposed on d.c. bias during cathodic arc deposition of TiN. Pulse peak, duty cycle, frequency, direct voltage, arc current and pressure all are investigated when coating TiN on HSS substrates. Roughness, surface micrograph, microhardness and thickness are tested. By analysis of variance, it is shown that pressure and frequency are the main factors. R_a and droplet density of the film with (d.c. + pulse) bias decrease. A simple explanation for the result is suggested. © 2000 Elsevier Science B.V. All rights reserved.

Keywords: Pulse bias; Orthogonal design; Cathodic arc

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1. Introduction

High ionizing rate and high bombardment energy are unique technical advantages of cathodic arc deposition. With these advantages, cathodic arc deposition has been developed rapidly in the last 20 years, but the macroparticles (MP) produced in evaporation are a serious problem. In order to resolve the problem, many methods have been used, such as steered arc [1,2], filtered arc [3,4], rotating cathode [5], sufficiently poisoned cathode [6] and pulse bias [7,8]. Pulse bias, with its high bombardment energy, may influence the characteristic of the film on many aspects. It is found that a superimposed pulse bias results in better adhesion, better film uniformity and a change in microstructure [8].

Compared with direct bias, the number of changeable parameters is increased when pulse bias is used. It is necessary to investigate how the effects of the various factors influence the characteristic of the coated films and to improve coating processing. Some references have discussed the results of superimposed pulse bias, but these discussions focused on the case of one variable while other parameters were fixed [7,9]. In fact there are many parameters which can influence the characteristics of the film. It is important to investigate the main factors while every parameter is independently adjusted. In this paper orthogonal designs are used. By analysis of variance, we try to find out the main factors which influence the characteristics of the film when pulse bias is used.

2. Experiment

The factors investigated were pulse peak voltage, frequency, duty cycle, arc current, pressure and the amplitude of direct bias. Every factor had three levels, therefore it was a problem of six factors and three levels. Provided there were no interactions among factors, we selected L_{18} (2×3^7) orthogonal table (Table 1) [10], where pulse peak voltage, duty cycle, d.c. bias voltage, frequency, arc current and pressure were arranged from column 2 to column 7, and were named factor A, B, C, D, E, F, respectively. Column 1 and column 8 were blank. All 18 samples were called in order from No. 1 to No. 18 according to the orthogonal table. In contrast we did an experiment with only direct bias. The sample was named No. 20 whose deposition parameters were also listed on Table 1.

All experiments were carried out in a MIP-4-650

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Table 1
L_{18} (2×3^7) orthogonal table

Expt. No.	1 e	2 A Pulse peak V_P (V)	3 B Duty cycle r	4 C Direct bias V_D (V)	5 D Frequency f (kHz)	6 E Arc current I (A)	7 F Pressure P (Pa)	8
1	1	300	0.08	0	15	50	0.4	1
2	1	300	0.33	75	30	65	0.7	2
3	1	300	0.58	150	45	80	1.0	3
4	1	550	0.08	0	30	65	1.0	3
5	1	550	0.33	75	45	80	0.4	1
6	1	550	0.58	150	15	50	0.7	2
7	1	800	0.08	75	15	80	0.7	3
8	1	800	0.33	150	30	50	1.0	1
9	1	800	0.58	0	45	65	0.4	2
10	2	300	0.08	150	45	65	0.7	1
11	2	300	0.33	0	15	80	1.0	2
12	2	300	0.58	75	30	50	0.4	3
13	2	550	0.08	75	45	50	1.0	2
14	2	550	0.33	150	15	65	0.4	3
15	2	550	0.58	0	30	80	0.7	1
16	2	800	0.08	150	30	80	0.4	2
17	2	800	0.33	0	45	50	0.7	3
18	2	800	0.58	75	15	65	1.0	1
20[a]		–	–	150	–	50	1.0	

[a] No. 20 is the experiment with only direct bias.

Multi-arc ion plating equipment. Base vacuum pressure was 3.0×10^{-3} Pa. Four evaporators were used, rotation was 3 rev./min and Ti ion bombardment was carried out with 800 d.c. volts in 120 s. The period of deposition was 60 min. All substrates were made of HSS with diameter 20 mm and 10-mm thick. Their surfaces were polished to $R_a < 0.02$ μm.

Our main concern was roughness, thickness, microhardness and micrograph of the film. Since the coating period was the same, the average deposition rate can be expressed by film thickness, which was obtained by measuring the cross-section of the film. The micrograph and cross-section of the film were investigated by scanning electronic microscopy (Hitachi S-450). A profilometer (Talyor-Hobson, model Talysurf 5-120) was used to measure surface roughness. The microhardness was measured by a microhardness tester (Futuretech, model FV-7) with a 10-g load.

3. Results

Test results are listed on Table 2. As every factor has three level, the degree of freedom (DF) of factors A, B, C, D, E, F are equal to 2 and then DF of the total error is equal to 5. As for surface roughness, the mean squares of A and B is smaller than that of the error sum of squares, then the DF of total error is 9. The results of analysis of variance for surface roughness, thickness and microhardness are listed on Table 3. According to the F-test, if $F > F_a$, the factor is significant, where a is the level of significance. For example, when $a = 0.01$, it means there is $1 - a = 0.99 = 99\%$ probability to say the factor being significant. From the *F distribution* [10], we get the following $F_a(f_1, f_2)$,

Table 2
Test results

Expt. No.	Roughness (R_a)	Thickness (μm)	Microhardness HV
1	0.4229	4.23	3197.30
2	0.2754	5.29	3196.23
3	0.1304	6.36	2746.40
4	0.2344	5.56	2871.67
5	0.2965	5.27	3186.13
6	0.2963	2.80	2256.83
7	0.3568	5.00	3313.60
8	0.1666	7.21	2720.97
9	0.5292	5.94	2680.53
10	0.3064	3.74	2869.90
11	0.2977	4.62	3508.80
12	0.2705	7.02	3130.23
13	0.2762	4.55	2776.43
14	0.4004	4.60	3297.80
15	0.3088	5.57	2730.70
16	0.3098	7.09	2984.30
17	0.2549	4.09	2733.70
18	0.3374	6.00	3031.97
20[a]	0.3612	4.90	3146.50

[a] No. 20 is the result of direct bias.

Table 3
Results of analysis of variance

Source of variation	F value for R_a	F value for thickness	F value for hardness
A (V_P)		11.54[a]	5.22
B (r)		3.12	9.11[a]
C (V_D)	2.94	2.31	6.27[a]
D (f)	3.67	27.79[b]	5.36
E (I)	2.97	3.97	5.80
F (P)	8.49[b]	18.70[b]	3.89

[a] Represents high significance.
[b] Means significance.

where f_1 and f_2 represent DF of factors and DF of error, respectively,

$$F_{0.01}(2,9) = 8.02,\ F_{0.05}(2,9) = 4.26,\ \ F_{0.1}(2,9) = 3.01$$

and

$$F_{0.01}(2,5) = 13.3,\ \ F_{0.05}(2,5) = 5.79,\ \ F_{0.1}(2,5) = 3.78$$

From Table 3, we found that:

1. for surface roughness, pressure is a highly significant parameter;
2. for thickness, in fact for deposition rate, pressure and frequency are high significance parameters. Direct bias voltage is significant; and
3. for microhardness, there is no high significant parameter. The significant are duty cycle and direct bias.

4. Discussion

It is well known that when pressure rises, it will make the cathode poisoned and increase the collision rate among travelling particles [11], therefore pressure is a highly significant parameter with regard to surface roughness and deposition rate.

We know that high bias voltage means strong ion bombardment and it will result in a change in microstructure and better film uniformity, but according to the results of analysis of variance, pulse peak voltage is not a significant parameter on surface roughness. Firstly, we think that the change in microstructure dependent on the collision between the growing film and arriving particles. But the effect of collision is controlled by momentum rather than by energy. Since

$$eV_s = 1/2\ \ mv^2$$

$$p = mv = \sqrt{\frac{2eV_s}{m}} \propto \sqrt{V_s}$$

where p, v, m, e represent momentum, velocity, mass and electrical charge of arriving particles, respectively, V_s means pulse peak. Momentum is therefore proportional to the square root of V_s. So a variation of V_s from 300 to 800 V will not cause a great change of the particle's momentum. Secondly, the MP from the evaporator is negatively charged. After a long distance travelling, it may lose its negative charge and become neutral. The bias voltage is unable to prevent these neutral MP arriving at the substrate. Thirdly the effect of collision is decided not only by positive particles but also by neutral particles which are not accelerated by V_s. Therefore we conclude that surface roughness is not largely dependent on pulse peak V_s.

Another result of Table 3 is that frequency is a significant factor for deposition rate. Generally, a higher bias voltage is in favor of sputtering rather than deposition. When bias voltage (d.c. + pulse) is put on between substrates (negative, cathode) and chamber (anode, ground), most of it drops in a narrow space near the substrate. The distance d, which represents the distance from substrate to the point of ground potential, is approximately 1 mm and is related to pressure and bias voltage [12]. Variable frequency may cause the changes of d. The mechanism of this narrow range's oscillation is complicated. Under pulsed bias, it may have an influence on the deposition rate.

From Table 3, we found that R_a of No. 3, No. 8, No. 20 are 0.1304, 0.1666, 0.3612, respectively. Although R_a of No. 8 is a litter bit larger than that of No. 3, it is much smaller than that of No. 20. More importantly, comparing parameters of No. 8 and No. 20, we found that only pulse bias is superimposed on No. 8, while other parameters are the same. The micrograph of No. 8 (Fig. 1) is more smooth than that of No. 20 (Fig. 2) and the droplet density is 1.7×10^5 and 7.4×10^5 mm^{-2}, respectively. Thus we can say that direct bias with superimposed pulse voltage improves roughness of the film greater than only with direct bias.

There have been many papers about microhardness [13,14]. Conclusions have often been different because of different experimental conditions. Hultman pointed out that ion bombardment creates additional point defects and enhances atomic mobilities which annealed out defects [15]. We think a suitable bias voltage is moderate, that is for d.c. bias, $V_D = 150$ V, for pulse bias, $r = 0.33$.

5. Conclusion

When pressure, arc current, direct bias voltage and three factors of pulse power supply, such as peak

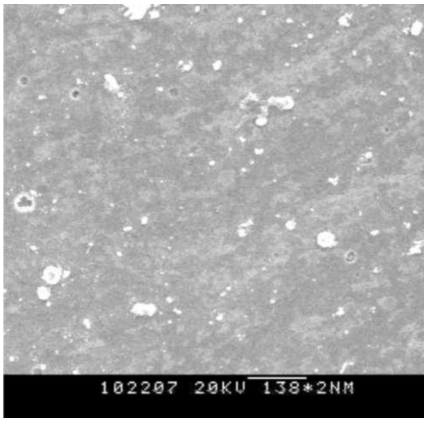

Fig. 1. Micrograph of No. 8 deposited at $V_P = 800$ V, $r = 0.33$, $V_D = 150$ V, $f = 30$ kHz, $I = 50$ A, $P = 1.0$ Pa.

voltage, frequency and duty cycle are all adjusted independently, we can say that

1. surface roughness and deposition rate are not very depended on pulse peak V_s but on pressure;
2. direct bias with superimposed pulse voltage improves roughness of the film greater than only with direct bias; and
3. for microhardness, there is no highly significant parameter. The significant are duty cycle and d.c. bias voltage.

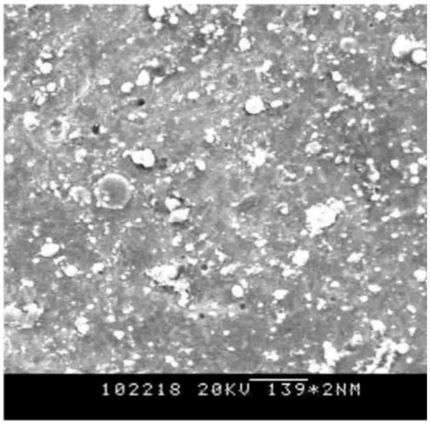

Fig. 2. Micrograph of No. 20 deposited at $V_D = 150$ V, $I = 50$ A, $P = 1.0$ Pa.

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