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Effect of bias voltage on microstructure and properties of magnetron sputtering TaN coating

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Abstract. A series of TaN films were coated on Al₂O₃ at various bias voltages by magnetron sputtering technique. Effect of bias voltage on the microstructure, roughness, deposition rate, binding force of coating-substrate, electrical properties of the TaN coating were investigated by X-ray diffraction (XRD), atomic force microscope (AFM), profile meter, scratch tester and four points probe respectively. The results suggest that the TaN coating were formed by face-center δ -TaN crystals and the preferential orientation of the coatings varied with the bias voltage; the deposition rate and the binding force of coating-substrate reached maximum value of 5.71nm/min and 4.5N when the bias voltage was 80 V, the roughness and the square resistance reached the minimum value of 0.509nm and 15.58 $\Omega/\square$.

1. Introduction

TaN, as one of the irreplaceable functional coating materials, has been applied in integrated circuit extensively due to its advantages of wide range adjustable resistance, high strain factor, better stability under high temperature, etc. In recent years, TaN coatings get much more attention from the researchers, especially for application of thin film resistance, barrier material, circuit etching material in microelectronics field. Magnet sputtering is a kind of physics vapor deposition technology that can produce pure coatings with better complex performance, which has been promoted rapidly in industry. In the literatures, researchers pay much attention about the effect of nitrogen gas content on the structure and properties of the coatings, but few studies are there about how the bias voltage of direct current influences that. Here the author deposited TaN coatings on Al₂O₃ substrate by direct current magnet sputtering method and investigated the effect of negative bias voltage on the structure and properties of the coatings, to optimize the process and obtain TaN coatings with excellent complex performance.

2. Method

Enamel Al₂O₃ ceramic sheet was used as matrix and rinsed with acetone, absolute ethanol under ultrasound treatment for about 30 minutes, then wash the matrix with deionized water for 5 times and dry in the vacuum oven. The experiments were carried out via TSU-650 multifunctional magnet sputtering system produced by Technol technology Co. Ltd. The TaN coatings were deposited on the surface of Al₂O₃ substrate with 99.9% pure Ta target (75mm in diameter and 4.2 mm in thickness) mounted on the top of the chamber and the face of Ta target paralleled with the substrate. The water-cooled substrate holder was placed at the bottom, about 100 mm away from the center of the target



center of the target surface. The device was pumped down to a base pressure of less than 4×10^{-4} Pa before deposition, with a 2000L/s turbo-molecular pump backed up with a mechanical pump. Argon (flow rate of 48.5 sccm) gas was used as the discharge gas and nitrogen gas (flow rate of 1.5 sccm) was used as the reaction gas, the operating pressure was maintained at about 0.4 Pa. the target was pre-sputtered for 5 minutes prior to each run. The parameters of preparation of the samples are listed in Table 1

Table 1 Parameters for preparation of TaN coatings

No.	Total flow/SCCM	Partial Pressure of N ₂ /%	T/°C	Bias Voltage/V	t/Min
1	60	2.5	150	60	35
2	60	2.5	150	80	35
3	60	2.5	150	100	35
4	60	2.5	150	120	35
5	60	2.5	150	140	35

X-ray diffraction measurements were carried out on the samples on using the D/MAX-2000 PC X-ray diffractor with Cu K α ($\lambda=0.154051\text{nm}$) radiation. the surface morphology of the samples were measured using a Bruker Dimension Icon atomic force microscope(AFM) in the AC mode. Four points probe was used to test the square resistance of the coating. Ws-2005 scratch tester and Bruker Dektak XT were used to measure the binding force of coating-substrate and the thickness of the coating, respectively.

3. Result and discussion

3.1 Effect of bias voltage on the structure of TaN coating

Figure 1 shows the XRD patterns of the samples, and all the samples have shown face-center δ -TaN structure. However, their preferred orientation varies with the negative bias. When the bias voltage was 60 V, the coating has a preferred orientation in the face-centered cubic (111) plane and a significant peak in the face-centered cubic (200) plane. With the gradual increase of the bias voltage, the peak intensity of the face-centered cubic (200) surface gradually disappears, while the peak intensity of the face-centered cubic (111) surface gradually increases. The main reason is that different growth crystals have different requirements for the phase change driving force, so that the bias voltage and the rapid growth surface of the crystal have established a certain relationship, which in turn affects the preferred orientation of the coating.

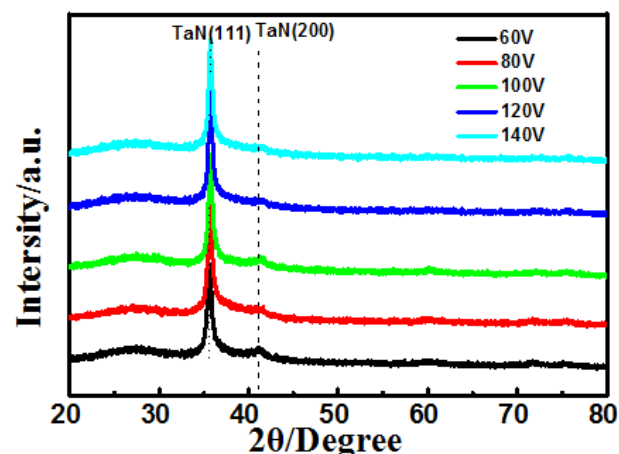


Figure 1 XRD of TaN coating prepared under different bias voltage.

3.2 Effect of bias voltage on the roughness of TaN coating

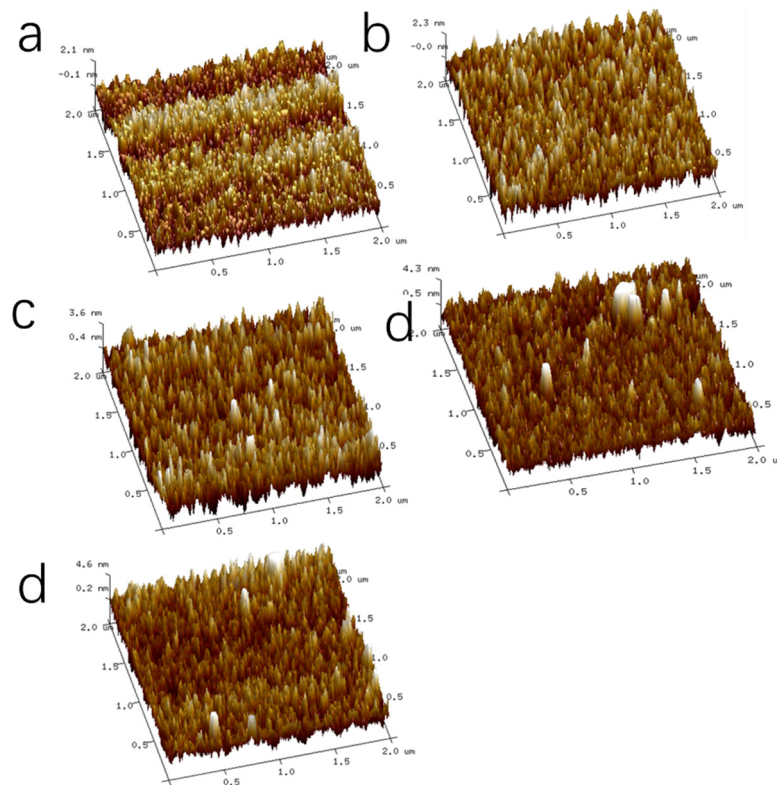


Figure 2 3D AFM images of TaN coating prepared under (a) 60V (b)80V (c) 100V (d) 120V (e) 140V bias voltage

The bias voltage has a significant effect on the particle size and the density of particle packing. During physical vapor deposition, ions from the gas phase exert a bombardment effect on the surface of the film, which affects the growth kinetics, surface morphology and crystal structure of the coatings.^[7] Figure 2 shows AFM photographs of TaN coatings at different bias voltage. When the bias voltage was 60V, 80V, 100V, 120V and 140V, the corresponding roughness was 0.53nm, 0.509nm, 0.736 nm, 0.76 nm, and 0.941 nm respectively. It can be seen that the roughness of the film first decreases and then increases with the increase of bias. When the bias is 80V, the roughness is 0.509nm at the minimum. The above phenomenon can be analysed from the mechanism of film deposition. The film forming process is mainly the competition between film growth and etching. When the bias is low, the ion energy is low, and the etching and sputtering effects on the film are relatively small. Therefore, the surface of the coating is relatively loose and rough. When the bias reached 80V, the bombardment effect of the deposited ions was enhanced, the activity of the deposited ions was increased, and the deposited particles were continuously transported and diffused laterally, thus increasing the density of the film and significantly reducing the surface roughness. However, excessive negative bias will increase etching and reverse sputtering on the film, increase the holes on the film surface, thin film thickness, and increase the roughness of the film, which also verifies the results of earlier scholars^[8,9].

3.3 The influence of bias on the deposition rate of TaN coating

Figure 3 shows the relationship between bias and film deposition rate. FIG. 3 shows that the deposition rate of TaN thin films first increases and then decreases with the increase of bias^[10]. When the bias voltage increases from 60V to 80V, the positive ions and neutral groups in the plasma move to the base surface, accelerating the adsorption and reaction on the base surface. Therefore, the growth rate of the thin film increases. When the bias exceeds 80V, active nitrogen and argon ions from plasma play a leading role in etching and sputtering of the film. Therefore, the deposition rate of the film gradually decreases with the increase of bias.

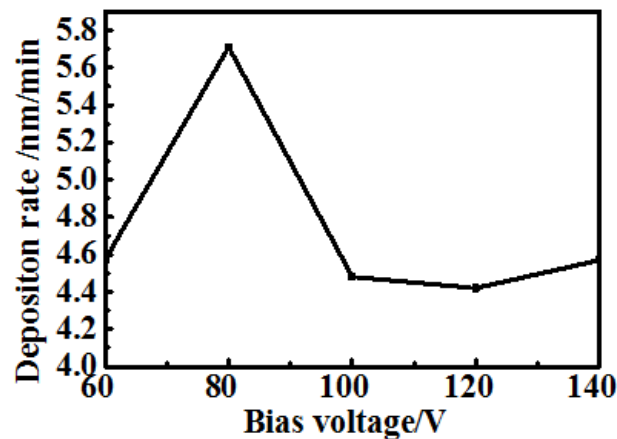
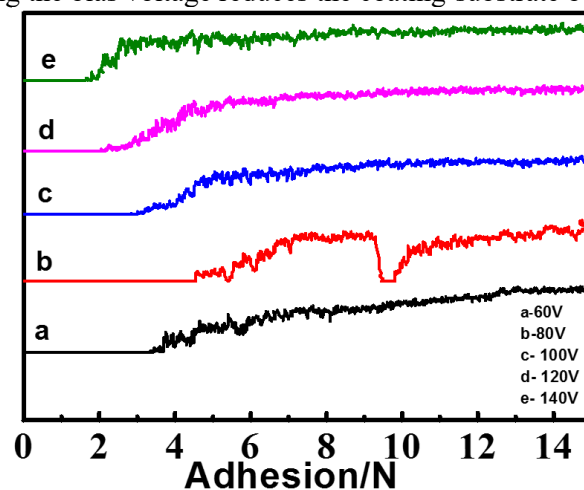


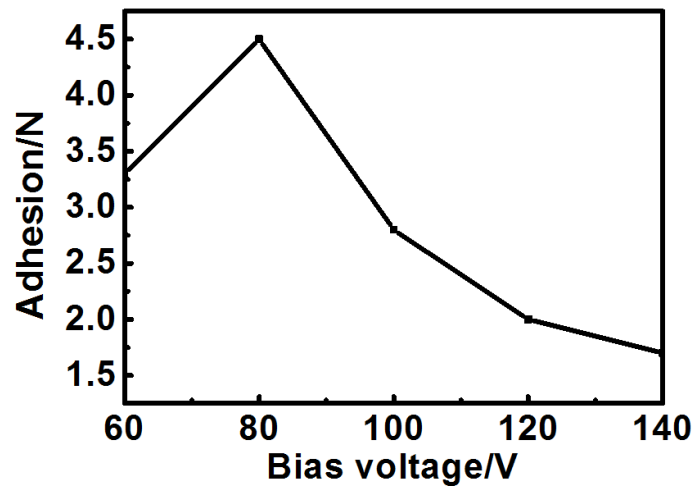
Figure 3 Relationship between bias voltage and coating deposition rate

3.4 Influence of bias on TaN coating-substrate adhesion

The interfacial bonding strength between coatings and substrate is one of the most important properties of coatings. Fig. 4 shows the adhesion between TaN film and Al_2O_3 substrate under different bias voltage. As can be seen from FIG. 4, when bias voltage was added, the coating-substrate binding force of the prepared film was stronger than that of the coating without bias voltage. When the bias voltage was 80V, the coating-substrate binding force was the highest. The main reason is that when the bias was applied to the substrate, the number and energy of ions deposited particles were increased, so that the number and energy of bombardment on the sharp parts of the substrate surface were increased, thus further enhanced the interface bonding of the coating, promoted the formation and broadening of pseudo-diffusion transition zone, and thus increased the adhesion between the coating and the substrate. However, further increasing the bias voltage reduces the coating-substrate bonding strength.



a) Relationship between membrane base adhesion and acoustic emission under different bias voltage



b) the relationship between bias voltage and coating-substrate adhesion

Fig. 4 Adhesion between TaN coating and Al_2O_3 substrate at different bias voltage

3.5 Influence of bias voltage on electrical properties of TaN coating

It can be seen from Fig. 5 that as the bias voltage increases, the square resistance of the film first decreases and then increases, but as a whole, the range of variation was $15\Omega/\square \sim 25\Omega/\square$ which is largely related to the coating thickness under the condition of a bias voltage of 80V. Studies have shown that the higher the coating thickness, the lower the resistivity, which can be explained by the fact that the parallel connection of the low resistivity inner layer and the high resistivity surface layer causes the sheet resistance to decrease with increasing thickness.^[11]

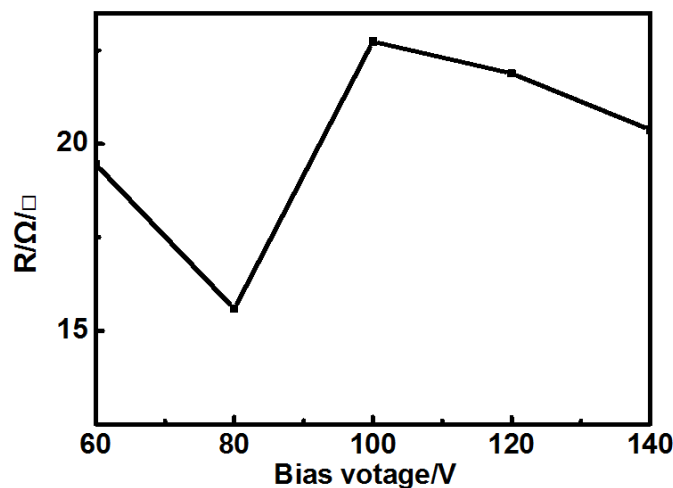


Figure 5 Relationship between bias voltage and square resistance

4. Conclusions

1) The TaN coating is formed mainly by the face-centered δ -TaN, and the preferred orientation varies with the bias voltage; the surface roughness of the coatings decreases first and then increases with the increase of the bias voltage. The minimum roughness is 0.509 nm when the pressure was 80V.

2) The deposition rate of the coatings and the binding force of the coating-substrate both increased at the initial and then decreased with the increase of the bias voltage, reaching the maximum when the bias voltage was 80V, which were 5.71 nm/min and 4.5 N, respectively;

3) The electrical property of the coating was less affected by the bias voltage, ranging from $15 \Omega/\square$ to $25 \Omega/\square$. When the bias voltage was 80 V, the minimum square resistance is $15.58 \Omega/\square$, as the thickness of the coating was largest.

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