EFFECT OF SUBSTRATE BIAS ON THICKNESSES AND HARDNESS OF TiN FILM CREATED BY ARC TECHNOLOGY

ẢNH HƯỞNG CỦA THIÊN ÁP ĐẾ ĐẾN CHIỀU DÀY VÀ ĐỘ CỨNG CỦA LỚP MÀNG TiN ĐƯỢC TẠO BẰNG CÔNG NGHỆ HỒ QUANG

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ABSTRACT

This study is aimed to investigate the influence of substrate bias on thicknesses and hardness mechanical properties of arc deposition technology TiN coating which is prepared on the surface of SKD61 alloy and Silic. The thicknesses of coatings were detected by examining the cross-sectional optical microscope image base on Silic sample. Vicker's hardness testing was carried out at four random points for zirconium alloy. Arc deposition TiN coating on SKD61 alloy has a maximum hardness of 2850HV achieved at the -300V substrate bias and thicknesses about 1,96 micormet were achieved at the -100V substrate bias.

Keywords: *Substrate bias, arc technology, TiN coating, mechanical properties.*

TÓM TẮT

Nghiên cứu này nhằm mục đích khảo sát ảnh hưởng của hiệu điện thế bias đối với chiều dày và độ cứng của lớp phủ TiN được phủ lên mặt hợp kim SKD61 và Silic. Độ dày của các lớp phủ được phát hiện bằng cách kiểm tra hình ảnh của kính hiển vi quang học trên mặt cắt ngang. Thử nghiệm độ cứng của Vicker được thực hiện tại bốn điểm ngẫu nhiên đối với đầu đo hợp kim zirconium. Lớp phủ TiN hợp kim SKD61 có độ cứng lớn nhất 2620HV đạt được ở hiệu điện thế bias là -300V và độ dày khoảng 1,96 micormet đã đạt được ở hiệu điện thế bias là -100V.

Từ khóa: *Thiên áp đế, công nghệ hồ quang, lớp phủ TiN, tính chất cơ học.*

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1. INTRODUCTION

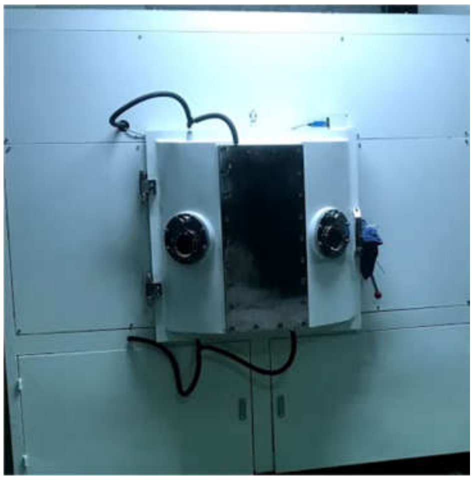
Deposition of TiN films has been widely introduced to various industrial coating fields, such as hard, protective, and decorative coatings on mechanical tools and ornaments, due to their high hardness, golden-like color, high wear, and excellent corrosion resistance [1, 2]. Especially, great attention has been paid to the interrelationship between the microstructure and the resulting mechanical, thermal, and physical properties. The results show that these properties are closely related to the method of deposition and to the process parameters. Control of the microstructural characteristics such as grain size, shape, textures, porosity, density, and packing factor is vital for ensuring the reliability of TiN films in structural and functional applications [3]. Many deposition parameters, often related to each other, can influence the final film's properties, such as the hardness. The same deposition parameters will also influence all fluxes toward the substrate during deposition. For instance, the metallic, ion, momentum, and energy fluxes will be functions of many deposition parameters. Finally, the intrinsic film properties, such as the microstructure, crystallographic texture, stoichiometry, and density, will be influenced. In principle, a direct relation between the deposition parameters and the final films can be determined. In conventional magnetron sputtering, the deposition parameters, such as the substrate temperature, the sure, the target power, the substrate bias, and the energy and flux of bombarding particles utilized for growing the films, are known to influence the resulting microstructure and properties of the films. For thicknesses in the range of tens of nanometers, a orientation is usually reported whereas the orientation takes place in films with thicknesses greater than hundreds of nanometers [4, 5]. At low deposition temperatures (T = 450^{0}C), the TiN films exhibit a columnar grain morphology with the preferential orientation of textured grains. However, higher temperature deposition results in non-competitive growth with a fully dense orientation in the initial monolayer of the film [6]. Among the various deposition parameters, it is known that the physical properties of the film can be changed by the application of a negative bias voltage to the substrate. Although a number of studies have been concerned with the microstructural evolution and texture evolution during the growth of polycrystalline TiN prepared by sputter deposition [4, 5], the effect of the substrate bias voltage on the crystallographic texture and the microstructure of TiN films grown by using reactive DC arc supply power has never been reported. The texture evolution mechanisms in TiN thin films are well been to be influenced by such as strain energy, surface free energy, surface diffusivity, and adatom mobility; the influence of each varies as a function of the processing parameters [6]. The potential use of nanocrystalline TiN films in tribological and microelectronic device applications may be successfully realized only if thorough insight is gained with regard to means to achieve the formation of desired microstructures through appropriate process controls

The present paper is devoted to TiN films deposited by means arc deposition technology various substrate bias voltages. The effect of the substrate bias voltage on the mechanical properties of TiN films is discussed. Decreased up to a critical value of nitrogen flow rate but then no more rapid decrease beyond this critical point. Several researchers have investigated the effect of reactive gas on the surface morphology and roughness of multi-component films such as TiN [7]. Neverthless, no systematic study has yet been conducted on the relation between substrate bias voltage and nitrogen flow rates to surface morphology and its effect on various properties of the deposited films. Therefore the basic purpose of this study is to investigate the effect of substrate bias voltage on the hardness and thickness of DC arc soure cathode TiN coatings.

2. EXPERIMENTAL SETUP

The reactive arc deposition technique was used Ti target and nitrogen gas. The TiN coatings were deposited on silic and SKD61 steel samples. The SKD61 substrate was mirror polished. Prior to introduction into the chamber, the Si and SKD61 substrates were ultrasonically cleaned in acetone, methanol and deionized water and were dried by blowing nitrogen gas. A turbo-molecular pump backed by a rotary pump was used to achieve a base pressure of $5x10^{-3}$Pa before introducing the gas mixtures. Once a high vacuum of at least $5x10^{-3}$Pa was reached, the sample holder was heated and maintained at 450^0C for the deposition. The targets were then sputter cleaned with argon for 10 minutes while the substrates were shielded by shutters over the magnetrons. After the pre-deposition, reactive gas of high purity nitrogen was injected into the deposition chamber to form titanium nitride. The arc deposition was carried out in a gas mixture of Ar and nitrogen (with pure 99.99%) [8]. The flow rate of nitrogen and Ar was controlled by a mass flow controller. The nitrogen flow rate was varied between 500 to 1000sccm and the Ar flow was kept constant at 400sccm (standard cubic centimeters per minute). The negative substrate bias was varied from 50V to 300V. The target power of Ti (Direct Current) targets was 60A. The working pressure of Ar and nitrogen was set at 0.5mPa. During deposition the substrate was continuously rotating to give uniform coatings. The deposition times at the different nitrogen flows were varied to ensure a constant coating thickness of 2000nm.

Figure 1 shows the components of the arc coating equipment HCM-700. SKD61 for nuclear fuel elements was used as substrate material in this study. The substrates were obtained by wire cutting with dimension of Φ20 x 5mm and the heat effect zone was polished and removed to avoid the change of the microstructure of zirconium alloy substrate.



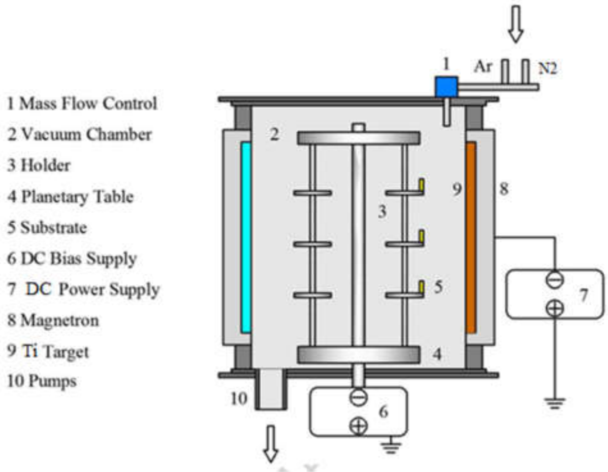


Figure 1. Schematic of the arc deposition

3. RESULTS AND DISCUSSION

The thicknesses of the deposited coatings were measured using weight-gain measurements and were verified using cross-sectional scanning electron microscopy on a steel sample Silic. Figure 2 shows the cross-sectional images of TiN-coated Silic prepared by different substrate bias. For all tested specimens, the TiN coatings appear golden yellow and are attached to Si alloy substrate with a well-defined interface.

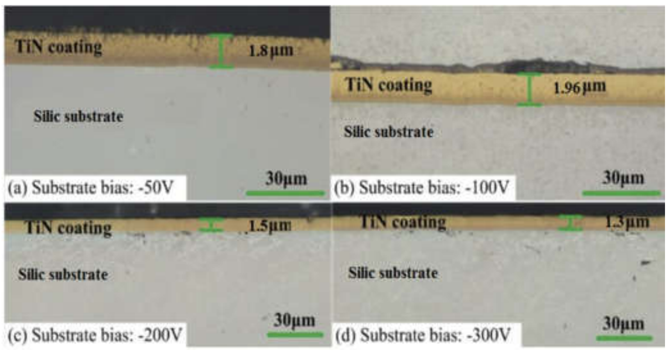


Figure. 2. Cross-sectional images of TiN-coated Si prepared by different substrate bias

After measuring, we have the following coating thickness Table 1.

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Table 1. Bias voltage and averad hardness

Bias voltage	Thickness of coating (µm)
-50	1.8
-100	1.96
-200	1.5
-300	1.3

The thickness of specimens prepared with substrate bias of -50V, -100V, -200V and -300V are shown in Figure 3. It is clear that the thickness or deposition rate of the specimens prepared with substrate bias of -50V, -100V is obviously greater than the specimens prepared with substrate bias of -200V, -300V. It means that more ionizing particles can be deposited on the surface of substrate with lower negative substrate bias in unit time.

Among the various deposition parameters, the physical properties of the film can be changed by the application of a negative bias voltage to the substrate during deposition. The application of TiN films for tribological applications may be successfully realized only if thorough insight is gained with regard to means to control the dependence of the crystallographic texture on various substrate bias voltages.

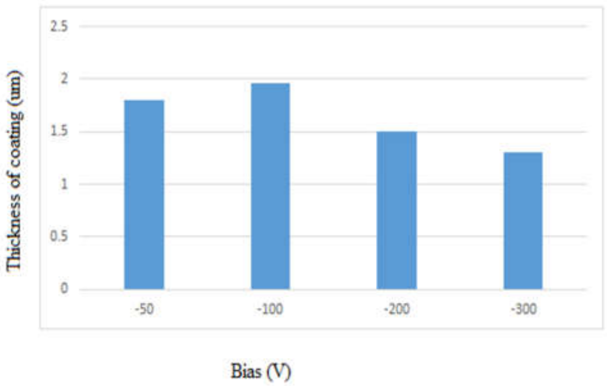

Figure 3. Negative bias of substrate (V)

The film hardness was measured with a nanoscale tester on a steel sample SKD61.

Figure 5 shows the hardness of the TiN coatings as functions of the bias voltage. A increase in the hardness was observed from 1760HV to 28500HV as is also shown in Table 2. Because when the voltage bias increases causes the force acting on the atoms to increase.The force acting on the atoms enabling the atoms to fit tightly together. Therefor the hardness increases.

Table 2. Bias voltage and averad hardness

Bias voltage	Average hardness (Vicker)
-50	1760
-100	2230
-200	2480
-300	2850

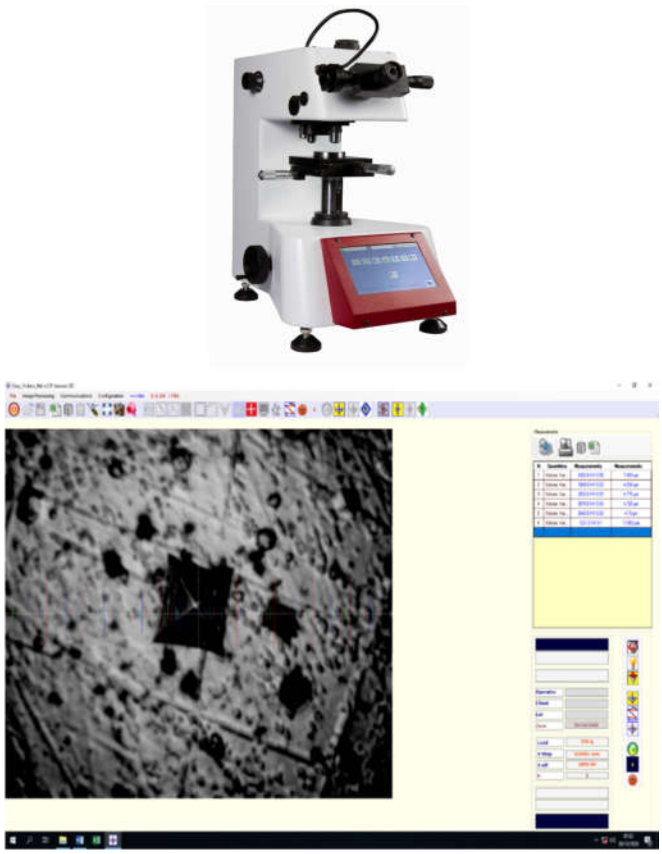

Figure 4. Hardness measuring ISOSCAN HV2 OD machine

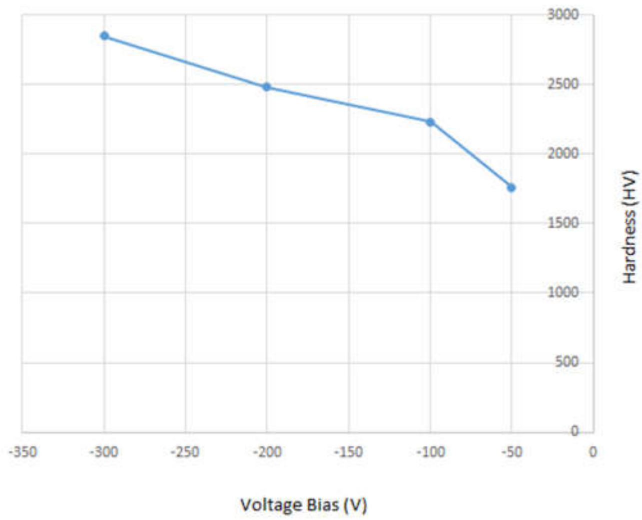

Figure 5. Hardness functions of the bias voltage for the TiN coatings on substrate SKD61 sample

4. CONCLUSIONS

TiN coatings synthesized by cathodic arc evaporation at different substrate bias voltage were investigated. The mechanical properties of the coatings were also studied. Test results allow to present the following conclusions:

- Deposition rate of the coatings decreases with substrate bias voltage increase. Because then the atoms with high kinetic energy collide with the membrane increases. This process can reduce the formation of membranes

- The hardness of the TiN coating increases as the substrate voltage difference increases. But the thickness of the coating will be thin. This is explained because when the voltage of the substrate voltage increases, the kinetic energy of the particles when rising up and hitting the ground can cause splashes of pre-deposited particles.

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