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Deposition Rate and Energy Enhancements of TiN Thin-Film in a Magnetized Sheet Plasma Source

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***Abstract*—**Titanium nitride (TiN) has been synthesized using the sheet plasma negative ion source (SPNIS). The parameters used for its effective synthesis has been determined from previous experiments and studies. In this study, further enhancement of the deposition rate of TiN synthesis and advancement of the SPNIS operation is presented. This is primarily achieved by the addition of Sm-Co permanent magnets and a modification of the configuration in the TiN deposition process. The magnetic enhancement is aimed at optimizing the sputtering rate and the sputtering yield of the process. The Sm-Co permanent magnets are placed below the Ti target for better sputtering by argon. The Ti target is biased from –250V to –350V and is sputtered by Ar plasma produced at discharge current of 2.5–4A and discharge potential of 60–90V. Steel substrates of dimensions 20x20x0.5mm^3 were prepared with N_2:Ar volumetric ratios of 1:3, 1:5 and 1:10. Ocular inspection of samples exhibit bright gold color associated with TiN. XRD characterization confirmed the effective TiN synthesis as all samples exhibit the (200) and (311) peaks of TiN and the non-stoichiometric Ti_2N (220) facet. Cross-sectional SEM results showed increase in the TiN deposition rate of up to 0.35μm/min. This doubles what was previously obtained [1]. Scanning electron micrograph results give a comparative morphological picture of the samples. Vickers hardness results gave the largest hardness value of 21.094GPa.

***Keywords*—**Chemical vapor deposition, Magnetized sheet plasma, Thin-film synthesis, Titanium nitride.

I. Introduction

TITANIUM nitride (TiN) has been considered high-technology material in the fields of microelectronics, aerospace and biomaterials for a long time [2]. This is due to the many exceptional properties it has. This hard wear-resistant coating enhances the performance and increases the lifetime of tools and devices. This is mainly associated to its thermo-physical and tribological behavior as this material can meet high temperature strength and possesses sufficient inertness [3].

There are many techniques employed in synthesizing thin film titanium compounds. However, the coatings obtained from vacuum-based methods show superior adhesion and better-quality formation to substrates [4]. The highly non-equilibrium condition of synthesis of compounds developed in these methods results to the formation of nano- and submicro-crystalline structures and, as a rule, have enhanced physical-mechanical properties compared with polycrystalline materials [5].

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A novel route in the deposition of TiN to substrates is the sheet plasma negative ion source (SPNIS) where substrate heating is no longer required. In addition, the produced thin-film has an excellent adhesion to substrate with short processing time and much simpler operation. The synthesis of TiN using SPNIS has been reported with a deposition rate of 0.166μm/min [1].

This study presents a modified approach in the synthesis of TiN using the SPNIS. This is a follow through of the previous studies done on TiN synthesis and an attempt to advance its operation. A modified configuration in the SPNIS is introduced with the noticeable addition of samarium-cobalt (Sm-Co) permanent magnets. The addition of these magnets promises the enhancement in the deposition rate of TiN to substrates.

The deposition rate is vital in coating materials since it shows the capability of the machine and the enhancement it can make. Key factors that directly affect the deposition rate are the sputtering rate and sputtering yield. These two quantities primarily take into account the energy distribution of the ions [6]. Hence, a further approach that can be done to maximize the deposition rate in the TiN synthesis is by increasing the aforementioned parameter.

The deposition of thin-film TiN is conducted on stainless steel substrates of type 316 having an initial hardness value of 10 GPa. To observe the enhancements, characterization of the samples is done using the x-ray diffractometry (XRD) to show effective TiN deposition; the scanning electron microscopy (SEM) for the calculation of deposited film thickness and surface morphology; and Vickers Hardness Test to see the increased hardness of the samples.

II. Experiments

The experiment uses the sheet plasma negative ion source (SPNIS) to produce the TiN deposited substrate. Fig. 1 shows the schematic diagram of the apparatus. The apparatus has two main parts – the production chamber and the deposition chamber. The production chamber houses the gas inlet for the argon (Ar) and the electrical feed through for the tungsten (W) filament cathode. The deposition chamber houses the titanium disk target, which is connected to the anode, the gas inlet for the N_2 gas, the substrate, and the pump side. It is in this main vacuum chamber where the sputtering and the deposition of TiN occur. Stainless steel type 316 with dimensions 20x20x0.5mm^3 is used as the substrate.

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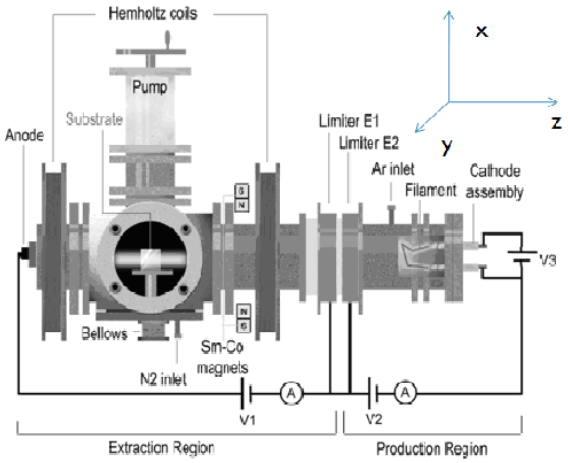


Fig. 1. Schematic diagram of the sheet plasma negative ion source.

In the modified configuration, the Ti target is placed below the substrate with a distance of 1 cm as shown in Fig. 2. The 2.2-kGauss Sm-Co permanent bar magnets with dimensions 40mmx20mmx10mm are placed below the Ti target. The magnet has a uniform field with the top surface as the north pole and the bottom surface as the south pole (inset of figure 2). The Ti target is biased from –250V to –350V while the steel substrate is not biased.

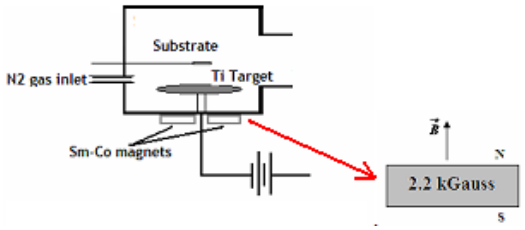


Fig. 2. Modified configuration of the TiN synthesis.

Before the deposition process, the substrate sample is washed with de-ionized water and then undergoes ultrasonic cleaning. Then, it is placed carefully in the extraction chamber. To attain a low base pressure of ~10^{-6} Torr, the source is evacuated using the oil diffusion pump with a back up rotary pump. A 30-minute discharge cleaning is then carried out using argon plasma. When the desired base pressure is achieved, Ar is introduced in the production chamber. Then, a high negative bias is applied to the target. N_2 gas is introduced to the deposition chamber to complete the synthesis of TiN. Except for the N_2 and Ar flow rates and their ratio, experimental parameters are the same for all samples. These parameters are:

- Ar plasma is produced at 2.5–4A, 60–90V;
- total gas filling pressure is at 40 mTorr;
- Ti target is biased from -250 to -350V;
- substrate is not biased;
- duration of the deposition procedure is set at 30 minutes.

After the experiments, the samples are characterized using XRD for the crystal structure and the confirmation of TiN synthesis, SEM for the surface morphology and film thickness, and Vickers hardness test to see the enhancement of the samples in terms of the hardness value.

III. Results and Discussions

Initially, the argon ion has energy between 60-90 eV due to the sputtering voltage. The high negative bias of the Ti target is chosen to help accelerate the positive Ar ions to the target by increasing the energy of the ions to 310–440 eV. The ion energy is further increased by about 1440 eV with the addition of the Sm-Co permanent magnets rated at 2.2 kGauss. Also, the magnets are positioned just below the target so that electrons are confined nearer the target. The combined effect of these accelerating Ar ions and the trapped electrons is the further increase of Ar ionization. The total energy of these ions in the set-up ranges from 1750-1880 eV. Hence, there is an increase in energy by 1keV. The energy increase brings a very significant increase in the sputtering yield as can be seen graphically using Fig. 3.

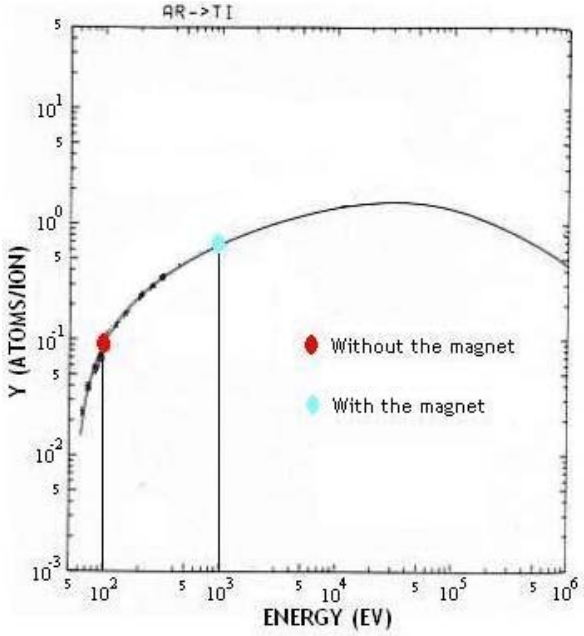


Fig. 3. Ti Yield as a function of Ar ion energy [7]

The enhancement in the substrate can be seen first-hand by ocular inspection of the TiN deposited samples. In Fig. 4 below, a comparison of the TiN deposited substrate in the previous experiment and the recent experiment is illustrated. Clearly, the result from this study shows a brighter TiN deposit. This demonstrates that there is enhancement in the deposition process.

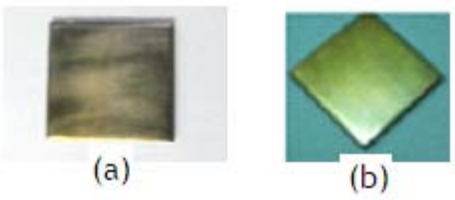


Fig. 4. Pictures comparing (a) previous result and (b) the recent result

Effective TiN synthesis in this modified configuration is confirmed through the XRD spectroscopy scans of the samples. All samples exhibit the stoichiometric TiN (200) and TiN (311) and the nonstoichiometric Ti_2N (220) as shown in Fig. 5. This implies high uniformity in the deposit with zero texture. This makes the deposition of TiN desirable for applications to high-speed devices such as cutting, drilling, and milling tools.

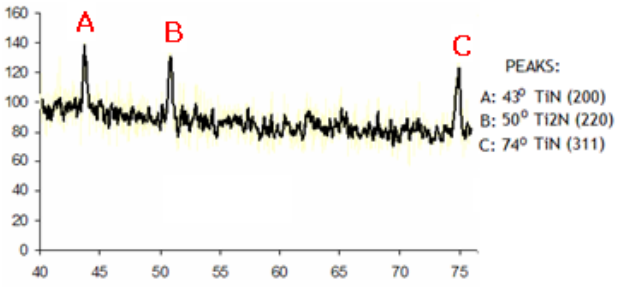


Fig. 5. XRD Scan

Further enhancement is shown in the SEM micrograph results where the deposition thickness can be obtained and the deposition rate calculated. Cross-sectional SEM of a sample shown in Fig. 6 shows a smoother substrate surface. Moreover, it provides the thickness of the deposited thin films. The highest thickness value outcome is 10.36 µm. The deposition rate can be calculated by dividing the cross-sectional thickness by the deposition time. Hence the maximum deposition rate is 0.345 µm/minute. This value is twice the previous result reported on SPNIS which is 0.166 µm/minute.

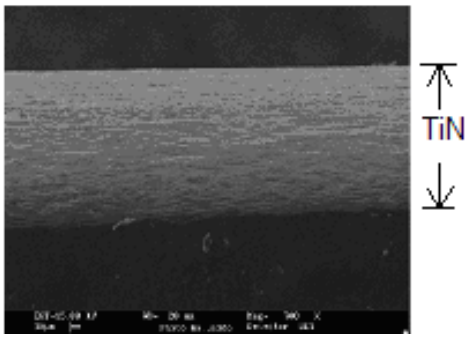


Fig. 6. Cross-sectional SEM of a sample

Finally, the hardness values of the samples have significantly improved as shown in the Vickers Hardness test results. The uncoated steel type 316 has an initial hardness of 10 GPa. After successful TiN deposition, the hardness value increased up to 21.094 GPa. Hence, TiN is advantageous in coating and surface treatment industries.

ACKNOWLEDGMENT

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