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Influence of rapid annealing temperature on the mechanical properties of TiCN thin film as prepared by cathodic arc

Wuttichai Phae-ngam[1], Ganatee Gitgeatpong[1], Jedsada Prathumsit[1], Viboon Tapanwong[2], Hideki Nakajima[3], Tanapoj Chaikeeree[4], Pattarinee White[5*]

[1]Faculty of Science and Technology, Phranakhon Rajabhat University, Bangkhen, Bangkok, 10220 Thailand
[2]Thapanin Co., Ltd., Bangpleeyai, Bangplee, Samutprakarn, 10540 Thailand
[3]Synchrotron Light Research Institute, Muang, Nakhon Ratchasima, 30000 Thailand
[4]Department of Electrical Technology Education, Faculty of Industrial Education and Technology, King Mongkut's University of Technology Thonburi, 126 Pracha Uthit Rd., Bang Mod, Thung Khru, Bangkok, 10140 Thailand
[5] Division of Physics, Rajamangala University of Technology Krungthep, Bangkok, 10120 Thailand
[*]**Corresponding Author:** pattarinee.w@rmutk.ac.th

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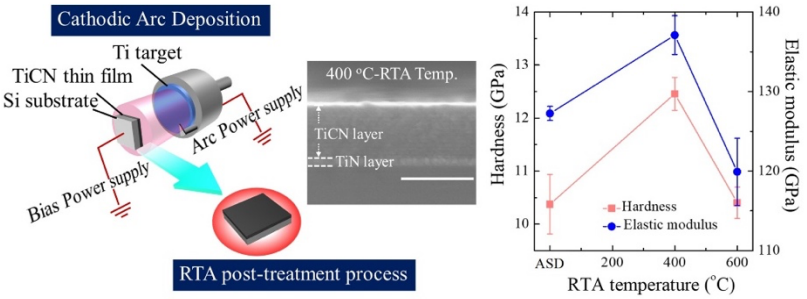
Abstract

In this study, titanium carbonitride (TiCN) thin films were deposited using cathodic arc deposition techniques. The as-deposited TiCN thin films were subsequently subjected to annealing treatment by rapid thermal annealing (RTA) technique at a temperature range from 400 to 600 °C. The effect of RTA temperature on the crystallinity, morphology, chemical composition, and mechanical properties of the TiCN thin films was investigated. The grazing incident X-ray diffraction (GIXRD) analysis confirmed the presence of a dominant face-centered cubic TiCN phase. Cross-sectional field-emission scanning electron microscopy (FE-SEM) images revealed a compact and homogeneous morphology, which became more pronounced with increasing RTA temperatures. The X-ray photoelectron spectroscopy (XPS) indicated the atomic concentration of the primary element (Ti, C, and N) remained relatively stable throughout the annealing process. Furthermore, the hardness of the TiCN thin films improved at 400 °C-RTA temperature.



Keyword: TiCN; Cathodic arc deposition; Thin films; RTA; Nanoindentation

1. Introduction

Ti-based compound thin films, such as TiN, TiC, and TiCN thin films, are extensively employed in various industry sectors, including chemical, bioengineering, tool, aerospace, and automotive industries [1-5]. This widespread use is attributed to their exceptional properties, including high hardness, toughness, anti-corrosion, thermal stability, and excellent wear resistance. Recently, the TiCN thin films have garnered significant attention in tribological applications because they offer a combination of the high hardness and low friction coefficient of the TiC phase with the high toughness,

excellent wear resistance, low friction coefficient and biocompatibility of the TiN phase [6-10]. These properties make TiCN a promising material for cutting tools, coating in high-wear environments, and medical device applications [11-12].

Several deposition techniques based on physical vapor deposition (PVD) methods, such as reactive magnetron sputtering [13-15] and arc-evaporation [16], pulsed laser deposition [17], and cathodic arc deposition [18-21] have been employed to fabricate the TiCN thin film. Among these, cathodic arc deposition is a versatile thin films deposition technique that utilizes the natural energy of the depositing ions, typically in the range from tens to hundreds of eV without bias [22]. This deposition method gains deposition rates, suitable for large area-coatings and excellent uniformity. Furthermore, it is widely used in decorative and hard coatings for the tool industry, as it provides high film homogeneity, density, good adhesion and the capability to deposit high melting point materials [23-25]. Notably, the deposition parameters in this method significantly influence the film's crystalline, morphology, and mechanical properties, enabling optimization for specific operating environments, particularly under high-temperature conditions [19, 26-30]. Consequently, understanding the film's structural evolution and the changes in its resulting mechanical properties have become important.

Generally, the TiCN prepared by cathodic arc deposition exhibit certain drawbacks that can limit their performance in industry applications. One of the major issues is microparticle contamination, which results from high-energy ion bombardment during deposition. These particles create surface roughness, leading to defects that affect coating uniformity and mechanical properties. Additionally, the high-energy deposition process induces significant residual stress, which can cause film cracking and poor adhesion [19, 31].

To address these challenges, post-annealing treatments were relatively straightforward and convenient approaches to modifying the physical, chemical, and mechanical properties of thin film materials [32-34]. Annealing at elevated temperatures could facilitate crystal structure relaxation, reduce defects, and enhance crystalline arrangement, ultimately improving properties such as electrical conductivity and mechanical properties. However, limited research has been conducted to comprehensively evaluate the effect of annealing temperature and mechanical properties of TiCN thin films.

In this study, the TiCN thin films were deposited using cathodic arc deposition. Following film deposition, the as-deposited TiCN thin films were subjected to post-annealing at temperatures ranging from 400 to 600 ^{o}C using the rapid thermal annealing (RTA) technique under low-vacuum conditions. Note that the RTA offer advantages over the conventional annealing process, due to its high heating rate and short processing time, which could minimize unwanted diffusion effect, suppress material decomposition and reduce undesirable phase transformations [35].

The influence of RTA temperature on film crystallinity, morphology, surface roughness, chemical composition, and mechanical properties was systematically investigated and analyzed.

2. Materials and Methods

The TiCN thin film deposition was performed with a commercial cathodic arc deposition system. High-purity titanium (99.95%) was used as target material. Further detail regarding the cathodic arc deposition system can be found in previously published work [21]. The square 1 cm^2 silicon (Si) wafer substrates were cleaned using ultrasonication in acetone, isopropanol, and DI water for 15 minutes each. The deposition chamber was evacuated to the base pressure of 2×10^{-2} Pa. Before the TiCN thin film deposition, a TiN ultra-thin film layer was prepared as an adhesion layer between the TiCN coating and the Si substrate. The TiN ultra-thin film layer

was deposited using argon (Ar) and nitrogen (N_2) gases for 1 minute, while all other deposition conditions were identical to those used for the TiCN thin film, as described below.

During the TiCN thin film coating, high-purity argon (Ar), nitrogen (N_2), and acetylene (C_2H_2) gases were introduced into the deposition chamber at flow rates of 480, 1250, and 2800 sccm, respectively, which were controlled by a mass flow controller. The deposition parameters were set as follows: a substrate bias of -100 V, substrate temperature of 130 °C, substrate rotation speed of 4 rpm, arc potential of 300 V, and deposition time of 3.5 minutes. After the deposition of TiCN thin film, the as-deposited TiCN thin films were annealed at temperatures ranging from 400 to 600 °C under a low-vacuum state with a pressure of about 6×10^{-3} mbar. This annealing process was performed using a 1-minute RTA system to investigate the film properties.

The film crystalline structure, morphology, and surface roughness before and after annealing treatment were analyzed by grazing incident X-ray diffraction (GIXRD; RigakuTtraz III), field-emission scanning electron microscopy (FE-SEM; Hitachi SU8030) and atomic force microscopy (AFM; SEIKO, SP400). The chemical composition was determined by X-ray Photoelectron Spectroscopy at the Synchrotron Light Research Institute (Public Organization, Thailand) using BL3.3Ua [36]. The mechanical properties of the as-deposited and annealed TiCN thin films were investigated by nanoindentation test (TI-900, Tribo Indenter, Hysitron) with a Berkovich 142.3°.

3. Results and Discussion

The GIXRD analysis was conducted to confirm the crystalline structure according to the RTA temperature of the TiCN thin films, as shown in Fig. 1. The results revealed that both the as-deposited and annealed TiCN thin films exhibited a peak at 2θ of 36.4, 42.2, and 61.3, corresponding to (111), (200), and (220) crystal plane of the FCC TiCN phase (ICDD card number 42-1448), respectively. This observation confirms that all prepared TiCN thin films were polycrystalline structures. Moreover, after annealing at 400 and 600 °C, a small peak around 2θ at 52° could be observed, corresponding to the Ti-rich TiCN film, as reported in the literature [1].

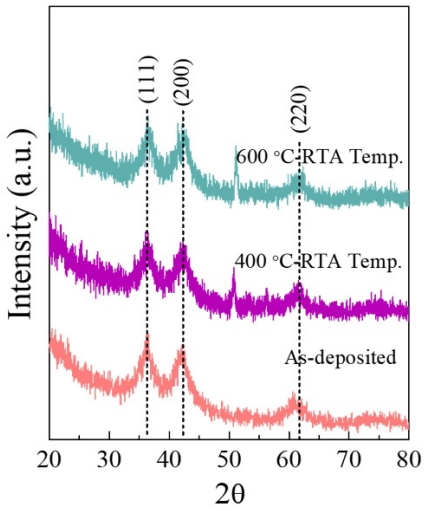


Fig. 1 GIXRD of as-deposited and annealed TiCN thin film at different RTA temperatures.

The effect of RTA temperature on the morphology of the TiCN thin film was observed using FE-SEM. Cross-sectional images of the as-deposited and annealed TiCN thin film were presented in Fig. 2(a)-(c). The results clearly indicate that a homogeneous TiCN thin film was successfully deposited on the TiN ultra-thin film adhesion layer, exhibiting excellent adhesion properties. While the thickness value of the as-deposited TiCN thin film was about 228 nm. It was observed that the thickness of the TiCN thin film decreased with increasing RTA temperature, as shown in Fig. 2(d). The reduction in film thickness could likely be attributed to the elimination of nano/micro-void defects and enhanced compactness of the film during the RTA treatment.

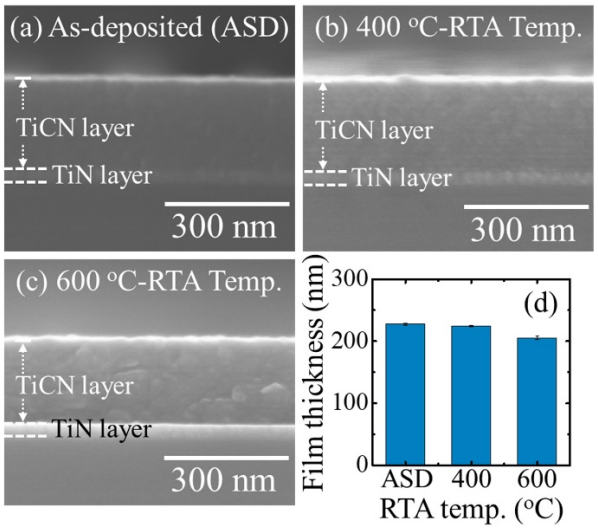


Fig. 2 (a-c) Cross-sectional FE-SEM images and film thickness of as-deposited and (d) annealed TiCN thin film at different RTA temperatures.

The surface morphology and roughness of the as-deposited and annealed TiCN thin film at different RTA temperatures were analyzed using AFM, as shown in Fig. 3. The result demonstrates that the surface roughness exhibits only minor variation with increasing the RTA temperature. Specifically, the surface roughness slightly decreases from 1.57 to 1.20 as the RTA temperature increases from as-deposited to 600 °C. This slight decrease in surface roughness can be attributed to grain boundary diffusion, coupled with strong atomic migration and the reduction of void defect, which is consistent with the observations from FE-SEM results.

The chemical composition was determined using a highly surface-sensitive XPS technique. Fig. 4(a) presents the XPS full survey scan of the as-deposited and annealed TiCN thin films. Peaks corresponding to the core orbitals Ti2p, N1s, C1s, and O1s core were observed at the binding energies of 458.2, 399.2, 284.6, and 531.2 eV, respectively, for all prepared TiCN films. It is important to note that the presence of oxygen could come from the surface oxidation occurring during the RTA process under a low-vacuum state. Also, high carbon concentration could be observed in all TiCN thin film samples due to the carbon contamination during the annealing and transfer of samples to the analysis chamber. Fig. 4(b) illustrates the atomic concentration of the element in all prepared TiCN thin films. The results indicate that the titanium level slightly increased and nitrogen composition decreased after RAT treatment.

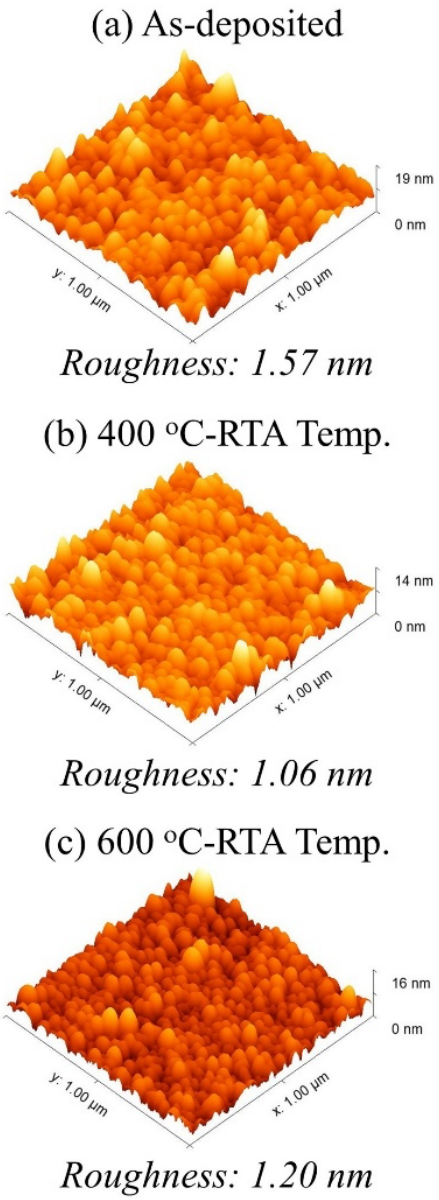


Fig. 3 AFM images and surface roughness of as-deposited and annealed TiCN thin film at different RTA temperatures.

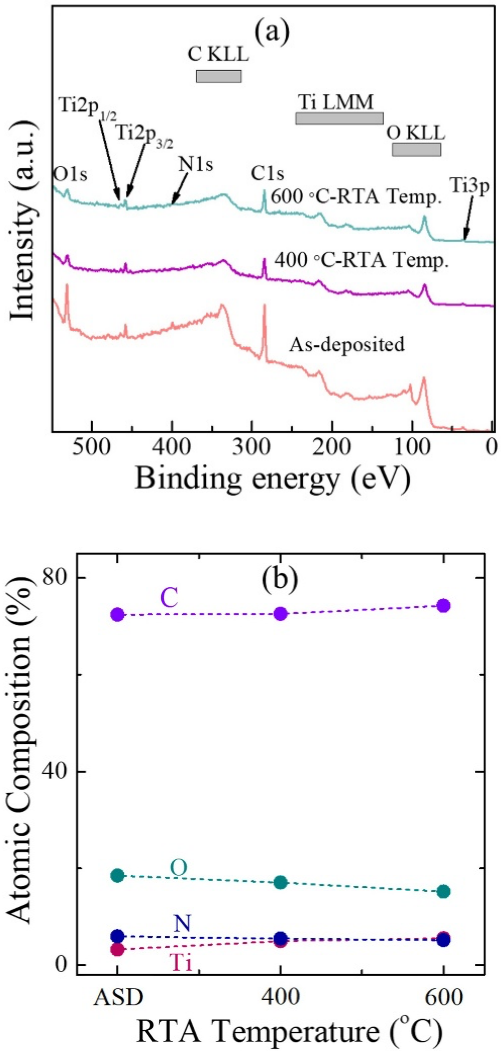


Fig. 4 (a) XPS spectra in survey scan and (b) atomic concentration of elements of as-deposited and annealed TiCN thin film at different RTA temperatures.

The hardness of the as-deposited and annealed TiCN thin films was measured by nanoindentation. To minimize the influence of the Si substrate on the hardness measurement, the applied load was carefully selected to ensure the indentation depth remained below 10% of the film thickness. This approach effectively isolated the mechanical property induced by RTA temperature. The loaded indent depth results of the as-deposited and annealed TiCN thin film on Si substrate are presented in Fig. 5(a). The result shows that the penetration depth of the TiCN thin film prepared at 400 °C was lowest, which could be attributed to its higher hardness compared to other samples under the same applied loads. The relationship between the measured hardness (H) and elastic modulus of the TiCN thin film at different RTA temperatures is illustrated in Fig. 5(b). The results showed that both the hardness and elastic modulus increased from 10.38 to 12.46 GPa and 127.24 to 137.09 GPa, respectively, as the RTA increased to 400 °C. This improvement might be attributed to the enhanced atomic diffusion, which reduces nano/micro-void defects by filling the gap and improving the compactness of the TiCN coating. However, as the RTA temperature increases to 600 °C, both hardness and elastic modulus decrease, which could be explained by the increase in grain size at higher RTA temperature [37]. Our TiCN thin film hardness is within the same range as that of TiCN film deposited on Si substrates by plasma-enhanced chemical vapor deposition (PECVD) [38] and magnetron sputtering [1], which typically ranges from 11.84 to 14.09 GPa. However, other studies have reported significantly higher values for thicker TiCN films prepared by cathodic arc deposition, with hardness ranging from 23.6 to 41.9 GPa [19, 39]. These films were achieved by optimizing reactive gas conditions and precisely suppressing oxygen incorporation.

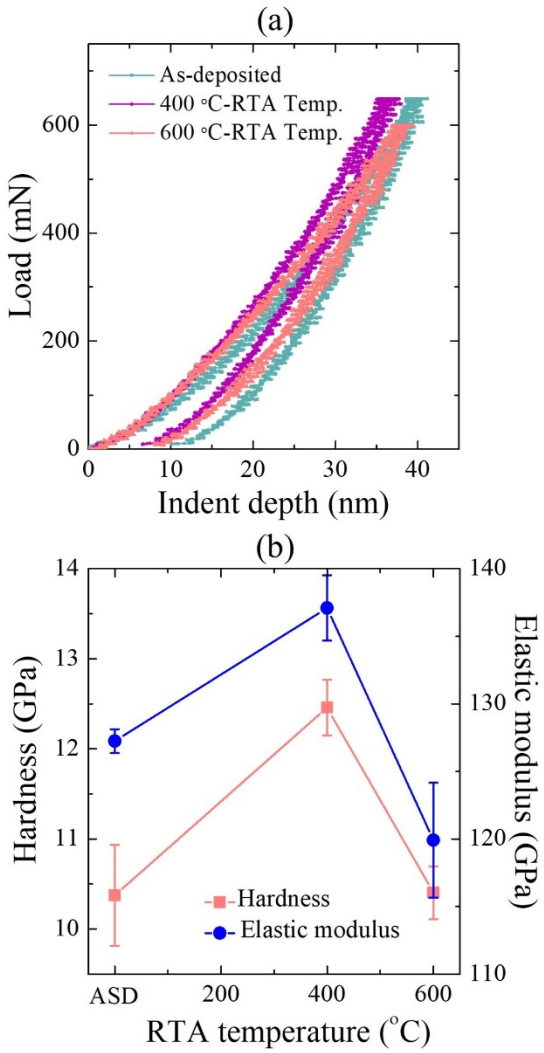


Fig. 5 (a) Load displacement and (b)hardness and elastic modulus of as-deposited and annealed TiCN thin film at different RTA temperatures.

4. Conclusion

In conclusion, the TiCN thin films were successfully deposited by the cathodic arc deposition technique and subsequently via RTA treatment at temperatures ranging from 400-600 °C. The GIXRD analysis confirmed the presence of a polycrystalline FCC structure of TiCN phase in all prepared TiCN thin film samples. The FE-SEM observations revealed that the annealed process enhanced the film's morphology, resulting in a highly compact structure. The atomic concentrations of the Ti, C, and N from XPS do not significantly change over the RTA temperatures. Notably, the hardness of TiCN thin films improved at 400 °C. These findings demonstrate that the RTA technique is an effective post-treatment method for optimized mechanical properties of the TiCN thin films, making it a promising approach for industrial-scale applications.

5. Acknowledgement

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