


## Microstructure and nanomechanical properties of TaN coating prepared by RF magnetron sputtering

G. Saravanakumar <sup>1</sup> , P. Gomathi <sup>2</sup>, V. Bharathi <sup>1</sup>, L. Ravikumar <sup>1</sup> , G. Balakrishnan <sup>3</sup> 

<sup>1</sup> Department of Mechanical Engineering, B.M.S. College of Engineering, Bengaluru, Karnataka, India

<sup>2</sup> Department of Mathematics, B.M.S. College of Engineering, Bengaluru, Karnataka, India

<sup>3</sup> Saveetha Institute of Medical and Technical Sciences, Saveetha Dental College and Hospital, Tamilnadu, India

 balaphysics76@gmail.com

### ABSTRACT

In this work, TaN is coated on the high-speed steel substrates at 500 °C using radio frequency magnetron sputtering and its microstructural and nanomechanical properties are examined. The structural, surface morphology and mechanical properties are analyzed by X-ray diffraction, atomic force microscopy and nanoindentation, respectively. X-ray diffraction studies indicated the presence of hexagonal Ta<sub>2</sub>N and cubic TaN phases at 5 sccm nitrogen flow at room temperature. It shows that the film prepared at 2 sccm and 500 °C revealed the strong intensity peak of FCC TaN phase, while the film prepared at 10 sccm and 500 °C showed that the broad peak demonstrating the nanocrystalline nature of the film. Atomic force microscopy analysis indicated the formation of crystallites of uniform size and homogeneous distribution. The surface roughness is ~ 2–6 nm in all the deposition conditions. The hardness of the TaN films has increased from  $9.46 \pm 1.15$  to  $30.05 \pm 3.79$  GPa with decreasing N<sub>2</sub> flow rate from 10 to 2 sccm. The microstructure depends on the preparation technique, processing parameters and nitrogen content in the films. TaN coating on high speed steel increased the hardness resulting the increase of wear resistance of the tool, leads to increase of the tool life.

### KEYWORDS

tantalum nitride films • high speed steel • X-ray diffraction • atomic force microscopy • nanoindentation

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## Introduction

Coating is an important process for the engineering components such as thermal barrier coatings for turbine engines, antireflection coating for many optoelectronic devices, wear resistant coatings for both cutting tools and the kitchen utensils. Hard coatings are developed to protect the materials from wear and corrosion and hence increased the life time of the components. The development of the hard coatings is forced by the constant requirements from the cutting tool industries. Few micron thick TiC coatings were deposited on cutting tools by chemical vapor deposition (CVD) to solve the problems in the cutting tool industry in 1969. It is observed that the metal carbide and nitride coatings can protect the materials from chemical and abrasive wear. Wear of the coating takes place as the cutting speed is increased. Most preferably Al<sub>2</sub>O<sub>3</sub> coating is utilized to avoid oxidation. Bilayer coating of TiC and Al<sub>2</sub>O<sub>3</sub> took the centre of attraction in industrial coating in 1975. These coatings protected the cutting boundaries



from the harsh environmental conditions. However, the CVD deposition technique is performed at high deposition temperature (900 °C), which causes poor rupture strength and toughness [1–6].

Physical vapour deposition (PVD) technique is an alternative technique to the CVD. The PVD coated TiN on the high speed steel (HSS) drill bits were the first industrial coatings in 1982. This technique produced crack free coating at lower deposition temperature of ~ 500 °C. TiN coating established by PVD method, led to the growth of TiCN coatings with increased hardness, but the problem encountered in the form of oxidation resistance. Hence, more attention has been focused on TiAlN system. Similarly, there was an increase of nanostructured coatings with hardness of more than 40 GPa. In the present scientific world, there are more than 100 different coatings available, which suits the specific requirement and demands of the modern industries with enhanced life time of components. Moreover, the cutting tool industries constantly searching for higher cutting speeds, which needs higher hot hardness and better oxidation resistance. Hardness, oxidation resistance and toughness are the three main factors, in the hard coating industries [7–9]. HSS-M2 type is the popular high speed steel replaces T1 high speed in most applications due to its better properties. Because of its low resistivity, structural integrity with Si, and wear-resistance capabilities, the cubic metastable phase TaN is favoured as a diffusion barrier for Cu interconnections in microelectronics. HSS materials are used in many applications such as cutting tools, gear cutters, milling cutters, tool bits, and drills due to its superior wear resistance and excellent hardness [10–12].

For the TaN films with low nitrogen, the hardness of the material is microstructure dependent. The hardness is increased with the decrease of crystallite size. TaN coatings are known for their hardness, wear and oxidation resistant, thermal stability and inertness. The recent research on tantalum nitride thin films has shown that they could be useful as extremely hard coatings [13–15]. Because of their superior wear resistance, TaN are applied to steel as protective coatings. Many research work on TaN is completed on their applications in thinfilm resistors and diffusion barriers. Few works is carried out on their applications in hard and wear resistant coatings. The scope of the present work is to increase the properties such as hardness and wear resisting behaviour of HSS with TaN coating. Increases hardness and wear resisting characteristics will increase the life time of the cutting tools (HSS). Different surface modification technologies are available to provide high wear-resistance and corrosion-resistance, thermal resistance and increasing hardness of the surface of the material. Among the many deposition techniques, the magnetron sputtering technology is widely used because of its advantages such as high deposition rate, purity of the films, stoichiometry, good adhesion of film, low pressure operation, uniform deposition on substrates and wide range of materials can be deposited. The work is carried out to increase the hardness and wear resisting behaviour of the HSS using nanostructured TaN coating and hence, increase the life time of the cutting tools (HSS) [13,16,17].

## Materials and Methods

### High speed steel

HSS are ferrous based alloys having the elements such as W, Mo, Cr, V, and Co. HSS materials are primarily used for cutting tools, since they have the capacity to retain a high hardness at high cutting speed. The elements present in the HSS are tested and the values are (wt. %) C = 0.866, Mn = 0.652, Si = 0.944, P = 0.023, S = 0.023, Al = 0.039, Cr = 2.98, Ni = 0.393, Mo = 3.51, V = 1.32, Ti = 0.006, W = 4.67, Cu = 0.138, Co = 0.777, Fe = 84.436.

TaN coatings are deposited on HSS substrates by radio frequency (RF) reactive magnetron sputtering. Pure Ar and N<sub>2</sub> are used as sputtering with reactive gases and mirror finished. HSS are used as substrates with a hardness of 727 HV. The HSS substrates are 20 × 20 × 5 mm<sup>3</sup> in size, cleaned thoroughly and fixed on a substrate holder cum heater. The base vacuum of the chamber is about 3 × 10<sup>-6</sup> mbar. The tantalum interlayer is deposited for 10 min using direct current (DC) power supply (360 V, 0.30 Amp) to improve the adhesion strength. Then, TaN thin films are deposited using the RF power of 100 W. The other deposition parameters are given in Table 1. The stylus profilometer is used to find the thickness of the films. The crystallinity of the films is analysed by X-ray diffractometer (PANalytical Xpert Pro) using CuKα1 (λ = 0.15406 nm) radiation. Surface topography and surface roughness are investigated using atomic force microscopy (Digital Instruments Nanoscope IIIa AFM) in non-contact mode. The hardness of the coatings is investigated using a nanoindenter (Hysitron Model-TI 950 Tribo Indenter) equipped with a Berkovich diamond indenter. The maximum indentation depth is ~ 70 nm.

**Table 1.** Deposition parameters

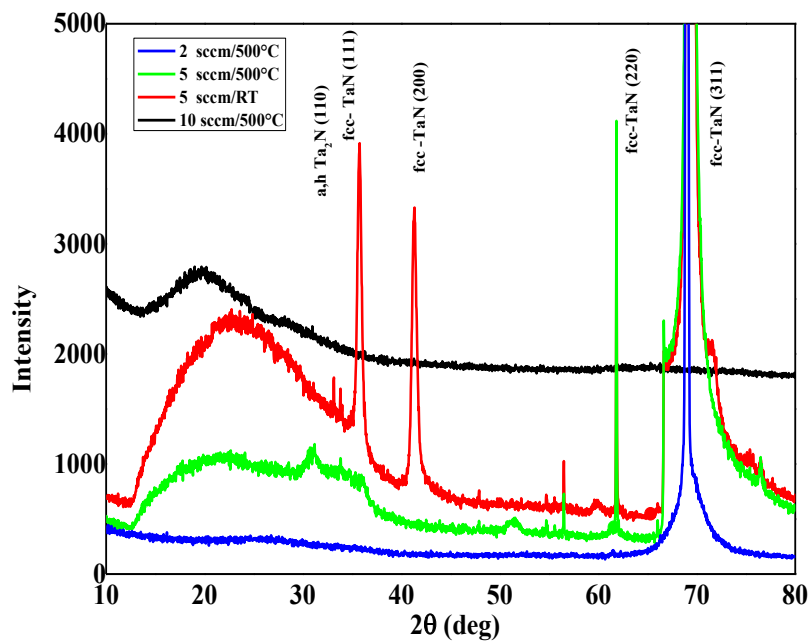
RF sputtering power, W	100
Argon flow rate, sccm	30
Nitrogen flow rate, sccm	2, 5 and 10
Substrates	HSS and Si (100)
Substrate temperature, °C	500
Interlayer	Tantalum
Deposition time, h	1
Target	Tantalum
Base vacuum, mbar	3.0 × 10 <sup>-6</sup>
Deposition pressure, mbar	3.0 × 10 <sup>-2</sup>
Substrate to target distance, mm	80

## Results and Discussion

### Microstructural studies

X-ray diffraction (XRD) pattern of the TaN films deposited on HSS substrates is shown in Fig. 1. The thickness of the coating is ~ 2 μm. Tantalum nitride has different stable and metastable phases. The films are deposited under four different conditions. The film prepared at 10 sccm N<sub>2</sub> flow rate and 500 °C shows broad peak indicating the nanocrystalline nature of the film. The film prepared at 5 sccm N<sub>2</sub> flow rate and room temperature revealed peaks at angles 33.4, 35.6, 41.2, 47.8, 56.5, 62.0 and 68.7° corresponding to the hexagonal Ta<sub>2</sub>N (110) (JCPDS File No: 39-1485) and FCC TaN (111),

(200), (220) and (311) phases (JCPDS # 49-1283). The film deposited at 5 sccm and 500 °C shows the peaks at angles 62.1 and 69.2° indicating the FCC structured TaN (220) and (311) phase [18–21]. The film deposited at 2 sccm and 500 °C shows the very strong peak at 69.1° reveals the FCC TaN phase with (311) orientation. These studies indicate that 2 and 5 sccm at 500 °C indicate the formation of FCC TaN phases. At room temperature and 5 sccm nitrogen flow, both Ta<sub>2</sub>N and TaN phases are observed. When adding nitrogen during the deposition, it strongly influences the phase composition, microstructure and properties. The increase in N<sub>2</sub> partial pressure increases the reaction probability between N<sub>2</sub> and Ta, and the TaN content in the film increases. The crystallinity of the films increased with the decrease of N<sub>2</sub> partial pressure. As the N<sub>2</sub> partial pressure, decreased from 10 to 2 sccm, the (311) reflection is stronger corresponding to the FCC TaN phase.

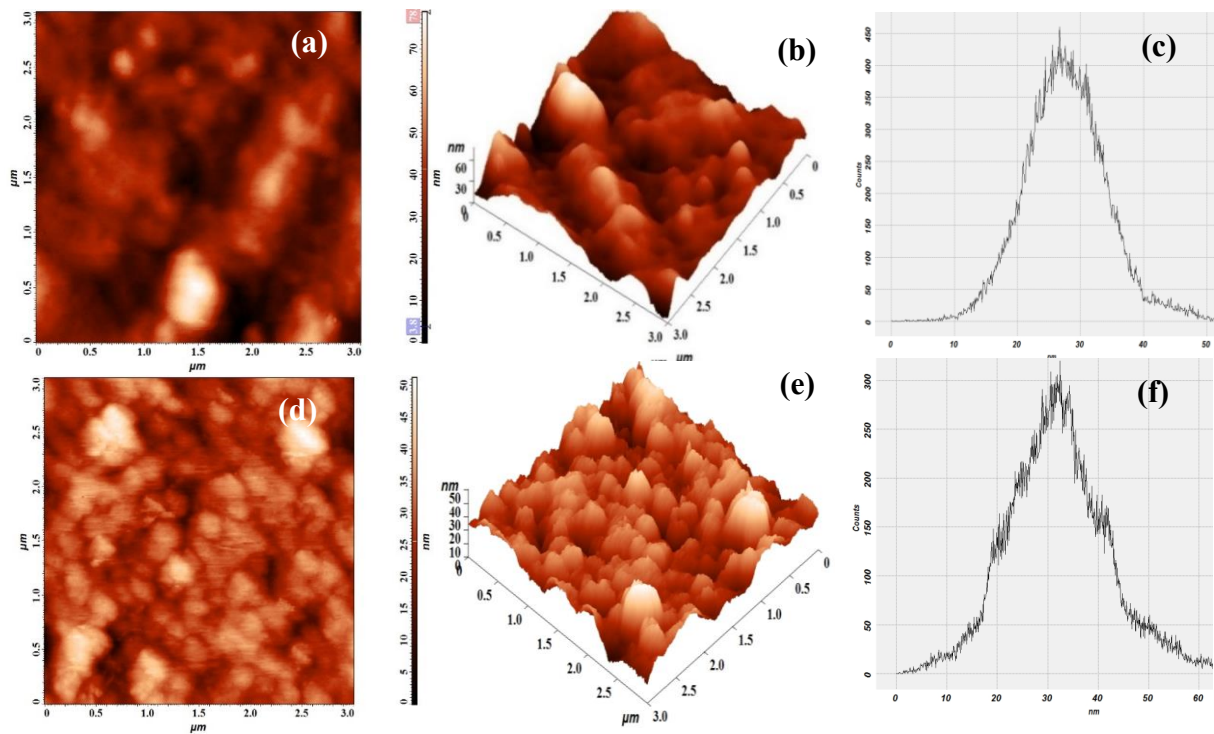


**Fig. 1.** XRD pattern of TaN films deposited on HSS-M2 substrates at 500 °C with different nitrogen flow rates

Michaela Grosser et al. [22] studied the phase composition, chemical and electrical properties of TaN<sub>x</sub> films with different nitrogen content. Aditya Aryasomayajula et al. [9] studied the TaN films deposited using pulsed DC magnetron sputtering method and the XRD studies showed the amorphous nature at low sputtering power. Bernoulli et al. [23] analyzed the effect of N<sub>2</sub> and Ar flow rate on structure and hardness of TaN and a transition from amorphous TaN to FCC TaN was observed. Elangovan et al. [14] prepared TaN films using pulsed DC magnetron sputtering and the XRD studies revealed the occurrence of  $\alpha$  and  $\beta$  Tantalum in the films prepared in pure Argon atmosphere. The FCC TaN structures occurred for 2 sccm N<sub>2</sub> flow rate and cubic TaN for 5–25 sccm of N<sub>2</sub> with argon at 773 K. Nie et al. [19] prepared TaN films by reactive RF magnetron sputtering under various Ar/N<sub>2</sub> pressure and observed the changes from BCC tantalum to a mixture of hexagonal Ta<sub>2</sub>N, and FCC TaN. Based on the microstructure, the property can be tuned for our applications. The phase formation is

either Ta<sub>2</sub>N or TaN phase in the present work depending on the nitrogen flow rate. XRD peaks for the films shows the TaN phase, Ta<sub>2</sub>N and TaN mixed phase and amorphous/nanocrystalline phase with the decrease in N<sub>2</sub> content. Therefore, it shows a structural dependence with respect to the nitrogen flow rate.

The surface topography and roughness of the films are investigated using AFM. The typical 3D images of the prepared films on silicon substrates with  $3 \times 3 \mu\text{m}^2$  size are shown in Fig. 2. Atomic force microscopy (AFM) analysis of TaN coatings on silicon substrates at ambient temperature and 500 °C reveals significant differences in surface morphology and microstructural characteristics. The TaN coatings deposited at room temperature have a surface that is clearly granular, with small, tightly packed grains that have clear edges. The surface topography shows a fairly nanoscale structures with grain sizes that can be seen between 25–30 nm. The height distribution histogram for the room temperature sample shows a narrow, symmetrical distribution, which means that the surface properties are the same. On the other hand, the TaN coating deposited at 500 °C has a surface shape that is very different. The higher deposition temperature has made it easier for grains to grow, resulting in larger, more discrete crystallites. The surface topography shows a better three-dimensional grain structure with distinct height differences than the room temperature sample. The 3D surface representation clearly shows the increased roughness of the surface and more defined grain boundaries. Individual grains exhibit enhanced faceting and greater height variations, indicating superior crystalline formation. The height distribution histogram for the 500 °C sample shows a wider distribution with a slight asymmetry. This means that the surface is more uneven and the height changes more throughout the scanned area. The sample, deposited at room temperature has a surface roughness of  $\sim 2\text{--}3 \text{ nm}$ , while the surface roughness was  $\sim 4\text{--}6 \text{ nm}$  for the sample deposited at 500 °C. The thermal energy

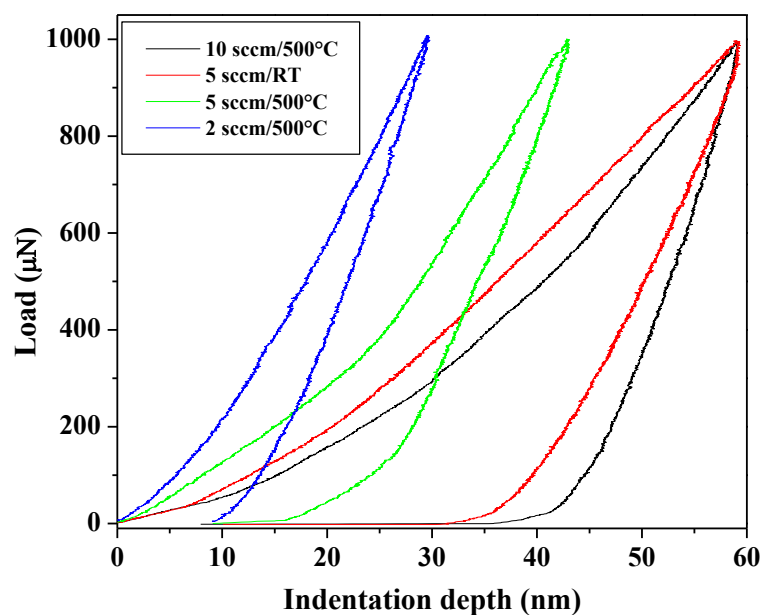


**Fig. 2.** (a,d) 2D, (b,e) 3D AFM images with (c,f) histogram of the TaN films deposited on Si at RT and 500 °C

is low at room temperature, the nucleation density is high, and grain growth is limited, which makes the microstructure less hard. At 500 °C, more thermal energy makes atoms move easily, which causes grains to merge and grow, resulting in larger grains with better crystallinity and higher hardness. The results are in concurrent with our XRD results [18,24–26].

### Mechanical properties

The indentation studies are performed to a depth of 70 nm and ten indentations (Fig. 3) are carried out on each sample and the mean value is calculated. The microstructures, such as crystallite size, columnar structure, voids, film purity are the major components in deciding the properties. It is noticed that the hardness of the films is increased with the decrease of crystallite size. In the present work, N<sub>2</sub> flow rate is varied from 2 to 10 sccm and the mechanical property is found to be varied systematically (Table 2). For the TaN deposited at 10 sccm flow rate revealed the hardness of  $9.46 \pm 1.15$  GPa, while at 5 sccm flow rate at 500 °C indicated the hardness of 17.21 GPa, whereas the film deposited at 2 sccm flow rate showed the hardness of  $30.05 \pm 3.79$  GPa. This clearly indicates that the hardness increased from  $9.46 \pm 1.15$  to  $30.05 \pm 3.79$  GPa with the decrease of N<sub>2</sub> flow rate.



**Fig. 3.** Nanoindentation curves of TaN thin films deposited at different N<sub>2</sub> flow rates

**Table 2.** Nitrogen flow rates versus hardness of the films

Sample	Nitrogen flow rate, sccm	Nitrogen content, %	Substrate temperature, deg	Hardness, GPa	Elastic modulus, GPa
A	10.00	25.00	500	$9.46 \pm 1.15$	$229.80 \pm 11.40$
B	5.00	14.00	RT	$10.44 \pm 1.20$	$198.90 \pm 14.45$
C	5.00	14.00	500	$17.21 \pm 1.80$	$332.80 \pm 19.31$
D	2.00	6.00	500	$30.05 \pm 3.79$	$346.40 \pm 24.00$

Nie et al. [19] deposited the TaN films at different N<sub>2</sub> partial pressure range 0.0–30.0 % and the highest hardness of 27.8 GPa is obtained for the film prepared at 10 % N<sub>2</sub> partial pressure. The TaN films with low nitrogen, the hardness shows a structural






dependence. The decrease of crystallite size causes the hardness increased from 16 to 24 GPa. The processing settings and techniques of preparation determine the characteristics of the material. Research on tantalum thin films has shown that they can be used as extremely durable coatings [27–29]. Westergard [10] found that the TaN samples possess the hardness of 2250 to 3300 HV range and found to increase with  $N_2$  flow rate. Yang et al. [6] reported the hardness and elastic modulus of 26 and 237.1 GPa, respectively for the TaN layer deposited using RF power of 150 W. Firouzabadi et al. [30] deposited the tantalum nitride films and found the formation of hexagonal  $Ta_2N$  and cubic TaN with increasing nitrogen flow rate. The present results are similar with the results obtained from other research groups [19,28–30]. The selection among these coatings (TiN, CrN, TiAlN, and TaN) is contingent upon the particular application. Although  $\gamma$ - $Ta_2N$  exhibits superior hardness, TiN ( $31 \pm 4$  GPa) provides commendable hardness with defined process settings. TiAlN (25–35 GPa) provides an optimal equilibrium with improved thermal stability, whereas CrN (18–25 GPa), despite its relative softness, may be favoured for applications having superior toughness with reduced residual stress. The enhanced hardness of specific TaN phases render them appealing for cutting tools and wear-resistant applications.

The TaN are of FCC structure and exhibit the strong covalent bonding with high bond density. The crystallized structure of FCC offers isotropic mechanical characteristics and dense atomic packing results in high cohesive energy, and the  $Ta_2N$  crystallizes in a hexagonal close-packed arrangement, which has the metallic character by the increased content of tantalum. The reduced level of nitrogen leads to reduced  $Ta_2N$  covalent bonds and increased anisotropic mechanical behaviour by layered hexagonal structure. TaN phase exhibited the greater hardness due to the nitrogen content and the structure whereas  $Ta_2N$  phase exhibited the lower hardness. In practice, TaN should be used when high hardness and wear resistance are needed, whereas  $Ta_2N$  phase should be used when the toughness is needed.

## Conclusions

Tantalum nitride coatings are deposited on HSS and silicon substrates using RF reactive magnetron sputtering technique. The thickness of the coating is  $\sim 2 \mu m$ . The microstructure and mechanical properties are studied at different  $N_2$  flow rate. The hardness of the uncoated HSS is 727 HV. The XRD studies revealed the polycrystalline hexagonal  $Ta_2N$  and cubic TaN phases. The crystallite sizes increased with the decrease of  $N_2$  partial pressure. The peak orientation changes as the nitrogen flow rate varied. The 3D AFM images show the formation of nanocrystallites with uniform distribution. The surface roughness is  $\sim 2$ –6 nm for the films. The nanoindentation studies indicated the hardness of  $9.46 \pm 1.15$  GPa for the films deposited at 10 sccm  $N_2$  flow rate, while it is  $30.05 \pm 3.79$  GPa at 2 sccm  $N_2$  flow rate. It is found that decrease in nitrogen content increases the hardness value of the coated sample. Also, the hardness is  $17.21 \pm 1.80$  GPa at a substrate temperature of 500 °C, while it is  $10.44 \pm 1.20$  GPa at room temperature. Hence, the TaN coating on high speed steel contributes to the increase of hardness. The increase of hardness value, resulting increase of wear resistance of the tool, and hence increasing the tool life time.

## CRediT authorship contribution statement

**G. Saravanakumar**  **Sc**: conceptualization, methodology, software; **P. Gomathi** **Sc**: experimental work, manuscript draft; **Vasanth Bharathi**  **Sc**<sup>®</sup>: characterization of the samples and interpretation; **L. Ravikumar**  **Sc**<sup>®</sup>: experimental work and supervision; **G. Balakrishnan**: writing, sample analysis and review.

## Conflict of interest

The authors declare that they have no conflict of interest.

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