

# Mechanical and tribological behaviour of thin TaN coating produced on AISI 1018 substrate by DC magnetron sputtering

R.Hariharan, R.Raja, Somu. Vasu

**Abstract:** The properties Tantalum nitrate (TaN) multilayer covering by DC Magnetron Sputtering at different testimony temperature (200oC and 400oC) were proposed in the examination. The TaN multilayer coating was stored sequentially with the layers at base weight  $3.7 \times 10^{-6}$  m.bar, working weight  $11.5 \times 10^{-3}$  m.bar, Ar: N stream rate 15:28 SCCM 100w by DC magnetron sputtering. The impact of microstructure of layer thickness was explored. Higher hardness was found, 200 oC covering around 180,188 and 144 GPa separately. The counter consumption conduct was additionally broke down by salt shower and dampness with fog. TaN coating at 200 °C helps prevent 100 W of TaN retention film from abrading, making the structure small and making the hardness higher than other information stored. Coating attributes, including surface morphology, hardness and tribological conductivity

**Index Terms:** TaN multilayer, Anti corrosion, Input power, Wearness .

## I.INTRODUCTION

Thin film deposition of tantalum, TaN, and Ta/TaN bi-layer on Ti and SS316-LVM substrates by RF sputtering by Jara, B. Fraisse, V. Flaud, N. Fréty, G. Gonzalez. it absolutely was found that the character of the substrate encompasses a sturdy influence on the tantalum section shaped. Formation of an  $\alpha$ -Ta section was obtained on SS316LVM, however, the  $\beta$ -Ta section was shaped on Ti and on TaN substrates. Optical characteristics of sputtered atomic number 73 oxynitride Ta(N, O) films by Chao-An author, Tsung Shune Chin. By victimization, the SEM and SPM analyses, the Ta(N, O) film not solely show a columnar structure however additionally encompass a swish surface with AN RMS roughness zero.22 nm for a movie as-deposited at three hundred °C. Fabrication and tribological behavior of sputtering TaN coatings by Kun-Yuan Liu, Jyh Wei Lee, Fan-Bean Chinese. swish wear scar edge and restricted delamination of the multilayer a-TaN/c-TaN coating area unit determined, indicating a harder mechanical behavior and stronger adhesion of the multilayer coating as compared to single TaN layers. Mechanical and corrosive characteristics of tantalum / TaN multilayer coatings by Guojia Ma, Guoqiang Lin, Shuili

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**R.Hariharan**, Research scholar, Assistant professor, Department of mechanical engineering, Bharath Bharath Institute of Higher Education and Research, Chennai 73, India.

**R.Raja**, professor, Department of mechanical engineering, Bharath Bharath Institute of Higher Education and Research, Chennai 73, India.

**Somu. Vasu**, Department of mechanical engineering, Bharath Bharath Institute of Higher Education and Research, Chennai 73, India.

**Email:** mech.hariharan17@gmail.com

Gong, Xing Liu, Gang Sun, Hongchen Chinese. The Ta/TaN multilayer was applied in a very fuel pump accent and therefore the test takes a look at showed the service lifetime of the half accrued five times. it's been proved that particle implantation competes for a vital role in enhancing adhesion and choice of acceptable modulation periods and layers improved the mechanical and corrosive properties.

A comprehensive study on the surface mechanical engineering of tantalum thin film victimization molecular dynamics simulation: The result of TaN layer, power, and temperature by M.Nikravesha, G.H. Akbaria, A.Poladib. The results showed that the surface roughness is often remarkably improved by employing a TaN seed layer. Higher sputtering powers at lower temperatures offer the simplest condition to provide drum sander tantalum films.

Microstructure evolution and protecting properties of TaN multilayer coatings by Y.H. Yang, F.B. Wu. The amorphous/crystalline structure for the 20-layer TaN multilayer coating showed AN elevated essential load for adhesion fracture (Lc2) around 31.6 N because of the big volume ratios for the amorphous structure at initial deposition stage. Superior corrosion resistance for the 20-layer TaN multilayer coating was found because of its amorphous structure.

Microstructure, hardness, and wear resistance of sputtering TaN coating by dominant RF input power by Y.H. Yang, D.J.Chen, F.B. Wu. The structure of the multilayer TaN films comprised a combination of the single-layer TaN films. a mean hardness between those of crystalline and plain TaN layers was expected. However, tribological behavior analysis disclosed narrower wear scar and restricted film failure on the damage track for multilayer systems. Consequently, the multilayer TaN systems showed larger sturdiness than did the single-layer TaN films.

## II.EXPERIMENTAL PROCEDURES

The AISI 1018 is employed as substrates during this study for varied analyses. The AISI 1018 substrates were at first cleaned in ultrasonic cleaner. Cleaned substrates are grinded with emery sheets with various grades 1000, 1200, 1500, 2000, followed by diamond



polishing paste to obtain mirror polish of the substrate Suitable surface conditions before sputter deposition. TaN coatings were de posited by direct current (DC) magnetron reactive sputtering technique. A Ta Targets with diameters and thickness of 2 inches and 3 mm, respectively, were used as sources of evaporation. Between DC sputtering, Ar and Nitrogen gases were passed in the ratio 15:2.8sscm. Coating for the substrates were done under different parameters like Base pressure  $3.7 \times 10^{-6}$  bar and Working pressure  $11.5 \times 10^{-3}$  m.bar. Temperatures for coating the substrates are 2000c and 4000c. Time taken for covering one substrate is 1 hr at 100w power. Voltage and amperes utilized for covering are 503v and 203A individually. The coated samples were under gone with various tests like XRD, SEM, AFM, MICRO HARDNESS, & CORROSION. SEM images for our samples were taken at various magnifications like 500x, 1000x, 2000x, 5000x, etc and scale varies from  $5\mu\text{m}$  to  $20\mu\text{m}$ .

The parameters of the corrosion test with salt spray is concentration of the sodium chloride is 5.2% NACL, chamber temperature is 34.0-35.1<sup>0</sup>c, Saline pH 6.7, pneumatic pressure 15 pounds per square inch, solution recovery volume per hour 1.3 ml, sample washing method before and after loading. The parameters of the corrosion with humidity are humidity is 98% as measured by hygrometer during the test, Temperature of the test is 33-35<sup>0</sup>c, Pressure of air for atomizing is 2 to 3 bar continuously by pressure regulator, composition of the salt solution for 1 liter of solution is 5% of sodium chloride, 1% of magnesium chloride, De- ionized water 94%, pH of the solution is maintained at 7.5 by addition of buffer solution, Measurement of pH is measured once in 8 hours., type of loading of specimens is tied with plastic wire and hung in the hangers.

### III.RESULTS AND DISCUSSION

#### X-Ray Diffraction:

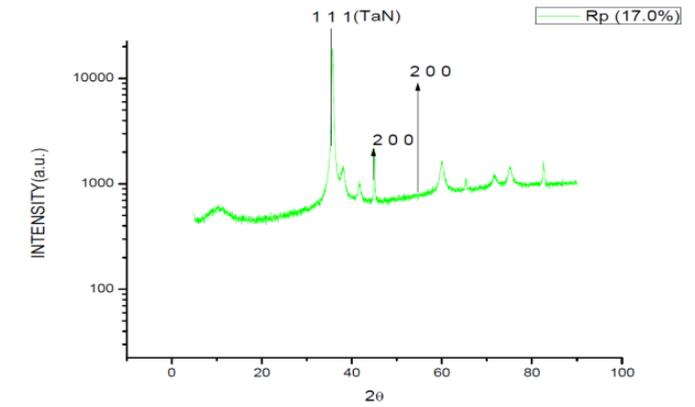
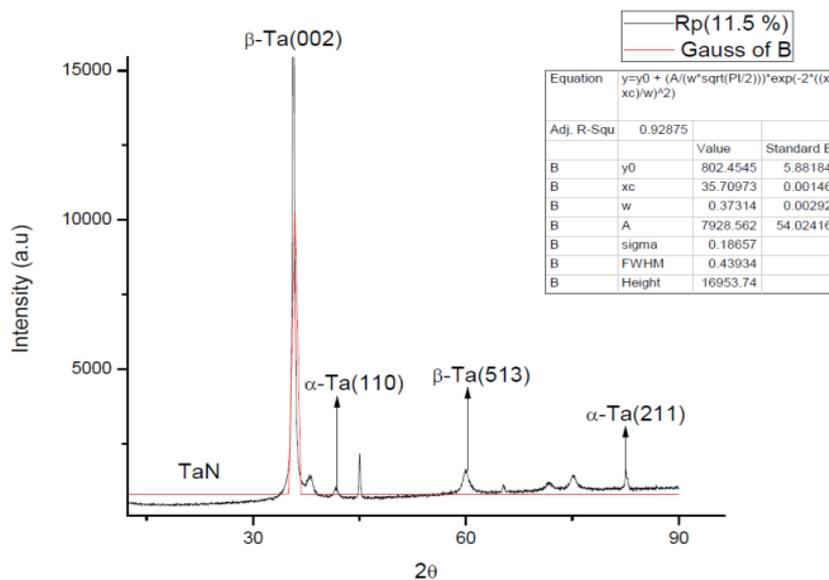


Fig:-1: Xrd patterns of TaN coated at 200<sup>0</sup>c

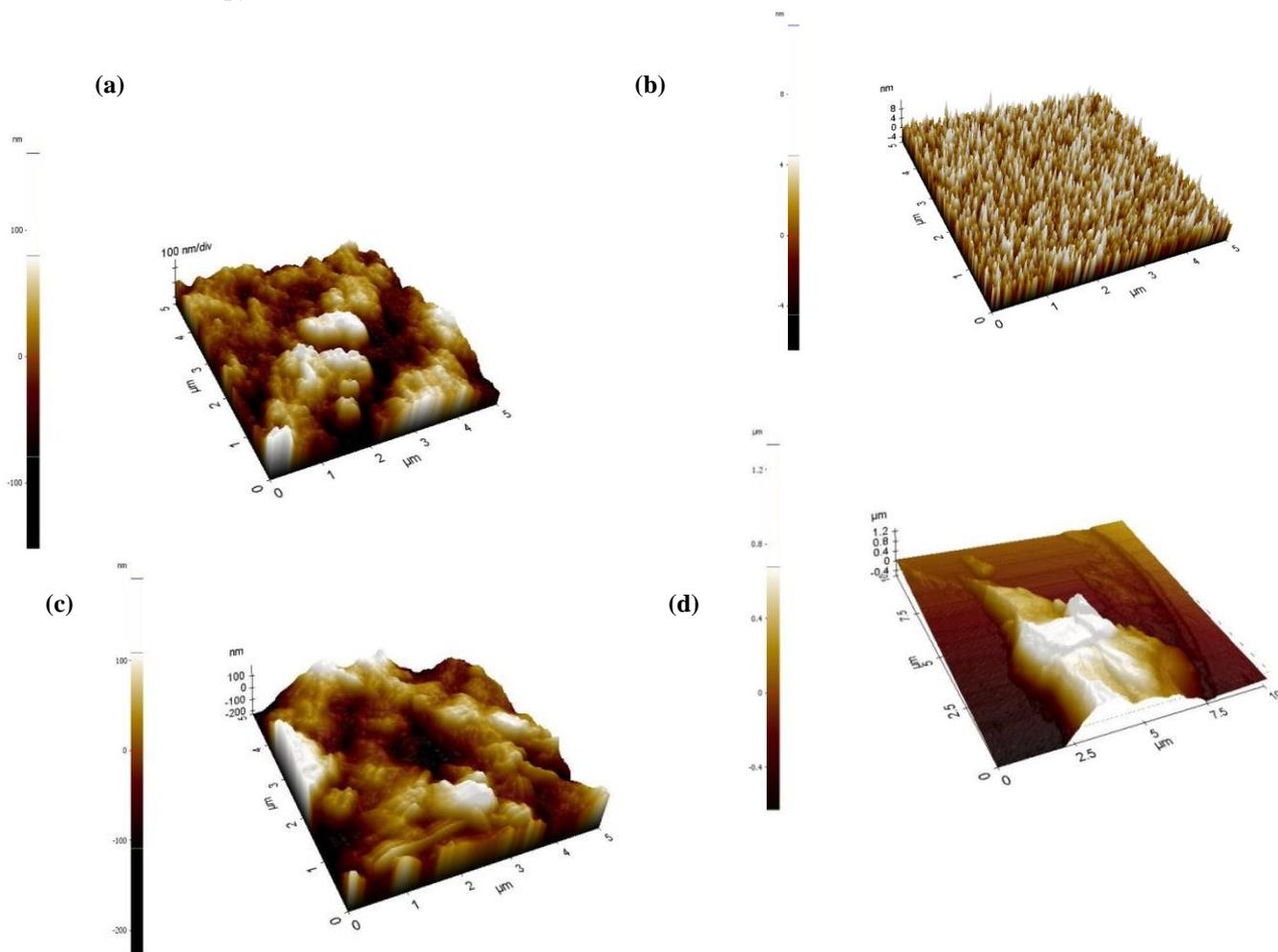
Fig.1: shows X-ray diffraction diagrams of low-alloyed steel coated with TaN at 200<sup>0</sup>c, the optical phenomenon of peaks from the substrate, their area, unit area, main peaks of cubic TaN, the main peak at 36.72<sup>0</sup> this is from 0<sup>0</sup> to 36.72<sup>0</sup> at this point the highest peak is shown and then normal small curves were appeared at a range of (200) and (111) this whole diffraction For sample coated at 200<sup>0</sup>c, from the fig.1:, it is observed that from the angle 2theta 0 to 36.72<sup>0</sup> there is no change in FWHM (full width at half maximum). At an angle of 2theta 30 degree observed an highest peak. However, the Ta-N system provides a complex series of related structures with small differences in the N content during spraying, as well as under transparent pressure, temperature, and / or time. Studies of X-ray diffraction (XRD), conducted by the researchers, show that tantalum nitride can be divided into eight phases with increasing N content: cubic TaN the crystal system formed by TaN at 200<sup>0</sup>c is cubic structure with unit cell is  $a=4.3580\text{\AA}$  and with the calc. density is  $15.644\text{ g/cm}^3$  XRD equipped with Cu K $\alpha$ 1 X-ray source (XPRT-PRO-MRD) at a Data range of 4.790<sup>0</sup> - 89.805<sup>0</sup>, with a Wavelength of 1.540598  $\text{\AA}$  The phase and structure of the Ta (N) coated at 400<sup>0</sup>c were determined through XRD technique, as shown in Fig.2 For sample coated at 400<sup>0</sup>c, from the fig.2 it is observed that from the angle 2theta 0 to 42.66

there is no change in fwhm (full width at half maximum). At an angle of 2theta 44.32 observed a change. The intensity is observed to be high at 2theta of 40.93. as 2theta increases there is an gradual decrease in intensity. In this the highest peak is noted at 44.32 and then the remaining curves were raised up to the level of 111<sup>0</sup> and 211<sup>0</sup> this whole operation is carried out with in the 2 theta The crystal structure formed by TaN at 400<sup>0</sup>c is hexagonal structure. The wave length of the TaN is  $1.540598\text{\AA}$ .

Fig:-2: XRD patterns of TaN coated at 400<sup>0</sup>c



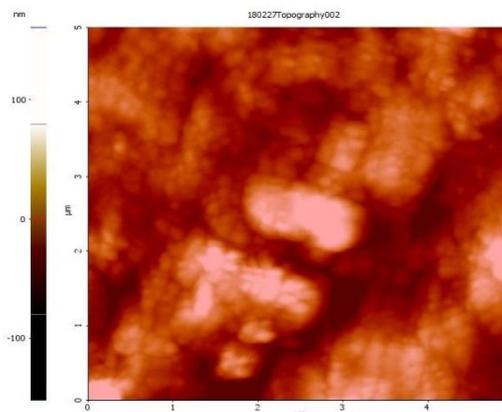
**Atomic Force Microscopy (AFM):**



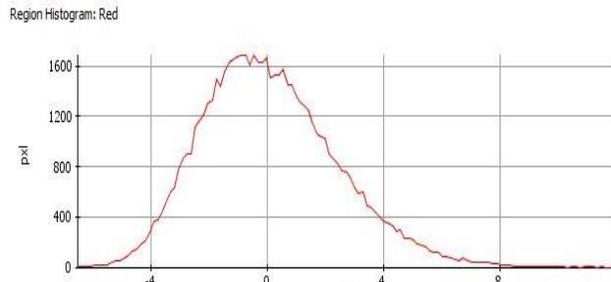
**Fig:3 Surface topography 3D view of samples obtained by AFM of samples (a), (b) sample coated at 200<sup>o</sup>c, (c), (d) sample coated at 400<sup>o</sup>**

Atomic force microscopy (AFM) is one of the portrayal strategies generally utilized for the surface investigation. This method is extremely appealing for surface imaging of different materials. The surface morphology of the movies was researched with AFM in the non-contact mode utilizing a silicon nitride cantilever. The nuclear power pictures demonstrate the adjustment in the morphology of the film with the adjustment in the temperature. The tantalum nitride film saved at 200<sup>o</sup>c shows harsh sporadic structure, while, the AFM picture of tantalum nitride film saved at 400<sup>o</sup>c shows littler surface highlights, joined with an expansion in the harshness (Ra) of the film (i.e. 0.264 nm to 42.769 nm). Surface projections at standard interims of a similar size and expanded unpleasantness are seen in tantalum nitride films kept at 200<sup>o</sup>c while test covered at 400<sup>o</sup>c had projections at unpredictable intervals. By contrasting the pictures we can state that as the temperature of the coating increases roughness and surface irregularities were increased.

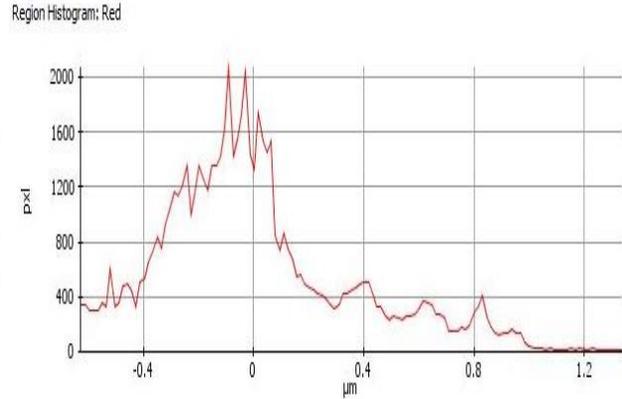
**ROUGHNESS**



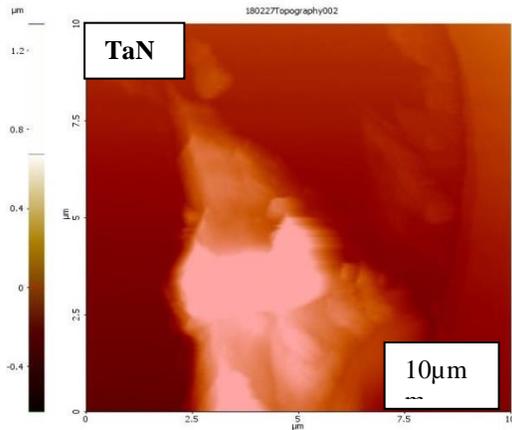
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**Fig.4: roughness image and line profile of 200<sup>o</sup> tan sample**



**Fig.5: roughness image and line profile of 400<sup>o</sup> tan sample**



The roughness estimations of 200<sup>o</sup> C tan example is 32.088nm, and in addition, the roughness estimation of 400<sup>o</sup>C tan example, is 42.769nm. By looking at both the qualities at various temperatures we watched that the roughness estimation of the 400<sup>o</sup>C tan example is higher than the 200<sup>o</sup> C tan sample. By this, can state that the unpleasantness esteem will increment as the temperature increments.

**Table-1: Roughness parameters observed:**

	200 <sup>o</sup> c		400 <sup>o</sup> c	
	a	b	c	d
<b>Min(nm)</b>	-151.945	-6.524	-0.628	-228.077
<b>Max(nm)</b>	160.694	11.921	1.335	191.057
<b>Mid(nm)</b>	4.375	2.698	0.353	-18.510
<b>Mean(nm)</b>	0.000	0.000	0.000	0.000
<b>Rpv(nm)</b>	312.639	18.445	1.963	419.134
<b>Rq(nm)</b>	40.625	2.310	0.345	55.446
<b>Ra(nm)</b>	32.088	1.840	0.264	42.769
<b>Rz(nm)</b>	300.73	17.685	1.957	412.31
<b>Rsk</b>	0.142	-0.489	-0.841	0.041
<b>Rku</b>	3.218	3.252	3.440	3.616

SCANNING ELECTRO MICROSCOPE (SEM)

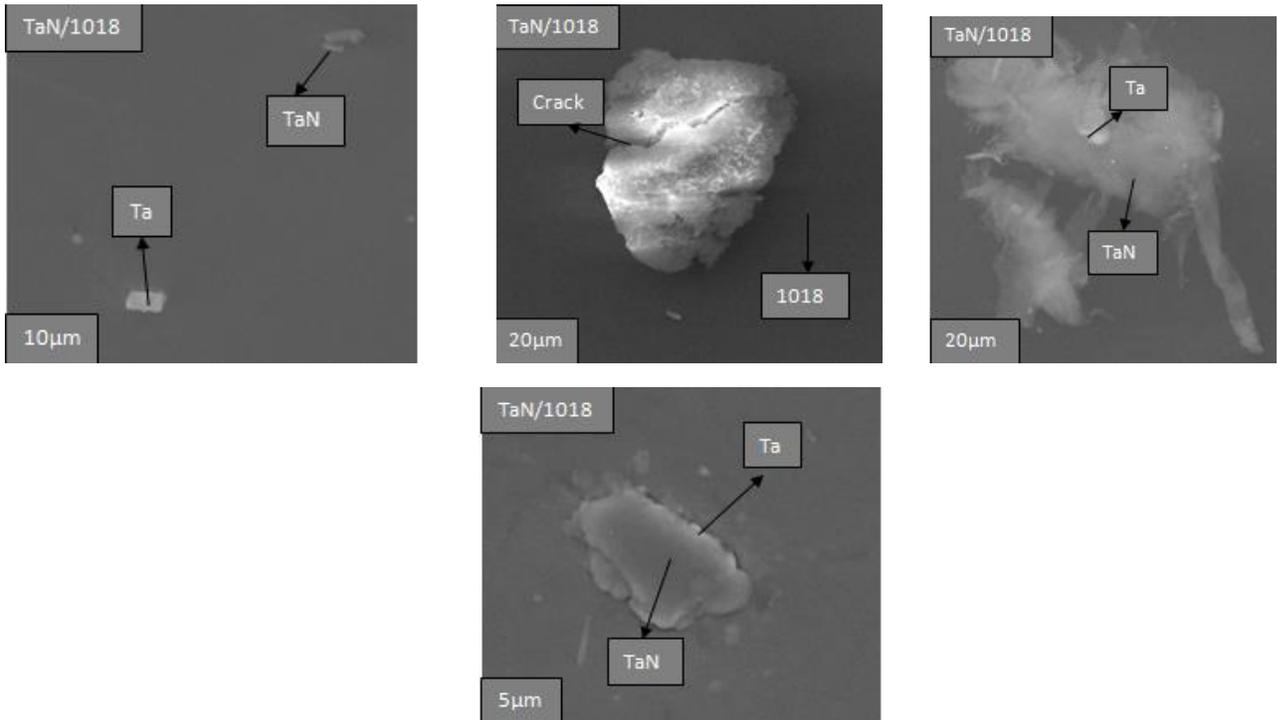


Fig.6: cross sectional SEM images of sample coated at 200<sup>o</sup>c

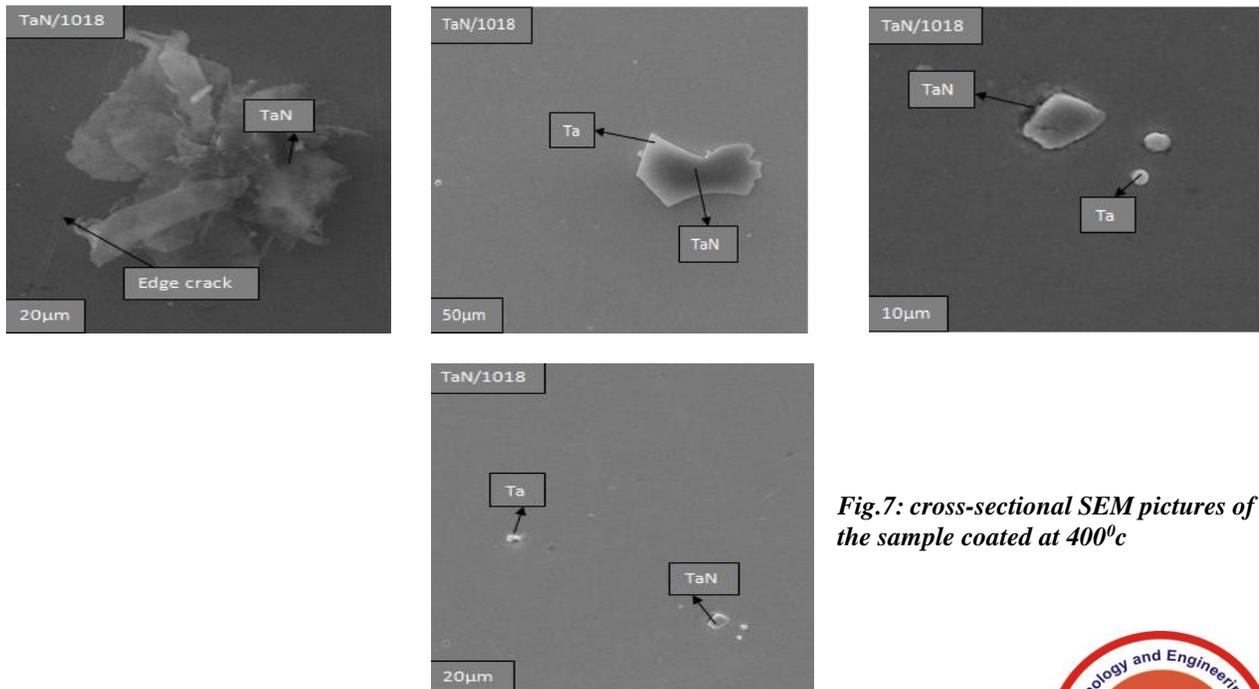


Fig.7: cross-sectional SEM pictures of the sample coated at 400<sup>o</sup>c

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Cross-sectional pictures of TaN coatings saved underneath numerous temperatures appeared in fig. 6 & 7 from fig.6 we watched broke apart at 20µm scale. Under different amplifications and scales, we watched unpredictable structures on the example. For tests covered at 200°c no. of damaged regions was higher when contrasted and the example covered at 400°c. Cracked structures were seen in test covered at 200°c, while test covered at 400°c had edge split structures. Cells were connecting on the surface of TaN gatherings with a good morphology. In addition, cells on TaN movies demonstrated more filopodia (appears

as a red bolt) at high amplification. Joined with the after-effects of fluorescence recolouring, it can be reasoned that the TaN film gave a remarkable condition to the connection and spreading. Numerous examinations have detailed that surface unpleasantness essentially influenced the cell reaction to the material, and the surface harshness affects cell adhesion. Combined the consequence of AFM pictures, the TaN film critical expanded the harshness of the substrates, which can be the purpose behind the better cell attachment on TaN gathering.

### Micro Hardness

Table-7: Mechanical parameters of samples (AISI 1018/TaN)

Vickers Hardness				
substrate	Hardness			
	Point 1	Point 2	Point 3	Mean
200°c	180	188	144	171
400°c	148	158	144	150

The hardness for TaN coatings at different temperatures was recorded in the table. The mean hardness taken at different focuses on the example 200°c and 400°c are 171 and 150 Gpa individually. From the diagram, the hardness of the example covered at 400°c is less when contrasted and a hardness of the example covered with 200°c. By this, presume as the temperature of the covering builds, hardness diminishes.

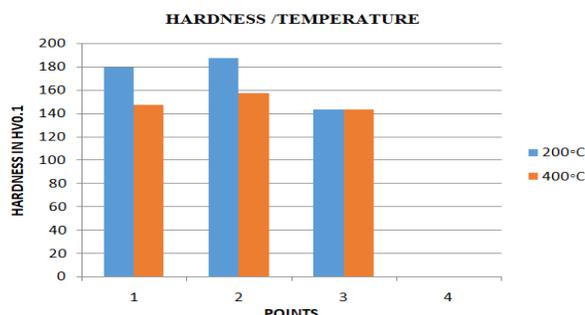
### CORROSION TEST

#### Corrosion Test with Salt Spray:

Corrosion test for tests was led at various parameters like 5.2% of sodium chloride focus and the temperature in the chamber was 34.0-35.10c. The PH of the salt arrangement was 6.7 and pneumatic stress was 15 psi and the accumulation of arrangement every hour was 1.3ml. The technique for cleaning of the example before stacking is example cleaned delicately preceding stacking and after fulfillment example washed tenderly in clean running water to expel of testing salt stores from their surfaces and afterwards dried promptly after this no consumption happened

Fig. 8: Micro Hardness Graph

until 12 hours so the introduction timeframe expanded



to 24 hours then red erosion watched.

Table-8: Corrosion (salt spray) test observations for 200°c

Sl.No	Time	Observation
1	At 12 Hrs	No corrosion
2	At 24 Hrs	Red corrosion observed

#### Corrosion Test with Humidity:

**Material:** round coin machined out of Low carbon steel was covered with Tantalum Nitride covering by D.C. magnetron sputtering process. This coin was subjected to salt shower chamber mist test according to ASTM benchmarks. All the surface of the covered example was subjected to Salt splash mist consumption test according to ASTM B-117 of every a salt shower chamber. The example was kept in holders with recognizable proof Nos.

In this sort of corrosion, the humidity was noted as 98% by hygrometer amid the test. The temperature of the test showed 330c-350c. The weight of air for atomizing was 2-3 bars consistently by a weight controller. The piece of the salt answer for 1 liter of arrangement is 5% of sodium chloride and 1% of magnesium chloride

De-ionized water is 94% PH of the arrangement is kept up at 7.5 by the expansion of cradle arrangement. Estimation of PH is estimated once in 8 hours. Kind of stacking of examples tied with plastic wires and hung in the holders the example was subjected to cleaning with non-watery dissolvable and which isn't assaulting base material steel with Tantalum nitride covering. The dissolvable cleaning was done just on the surfaces of the coin on every one of the sides.

**Observation of the specimen surfaces:**

The example surfaces were intermittently watched and the pictures of the surfaces were caught with high-determination USB camera at helpless zones of the example. The example following 24 hours was taken out and the water beads were wiped and watched outwardly and by USB camera. The surface states of the covered example were watched and derivations were observed. The beginning and the last weight of the example was weighed and the corrosion rate is given underneath.

**Results:**

**Table-9: Corrosion (Fog with Humidity) test observations for 400<sup>0</sup>c**

Sample ID	Initial weight in gm	Final Weight in gm (After 24 hrs.)	Increase in weight In gm	% Increase in weight
TaN coated	11.424	11.409	0.015	0.131%

## CONCLUSION

TaN films were set up on low carbon steel substrates at different temperatures (200<sup>o</sup>c and 400<sup>o</sup>c) using DC magnetron sputtering system. The prepared films were depicted by XRD, SEM, AFM, MICROHARDNESS and CORROSION strategies to consider the micro structural, mechanical & tribological properties of the films exclusively. The XRD examination showed that the films orchestrated at 200<sup>o</sup>c were exhibited the peaks identifying with cubic structure, while the films organized at 400<sup>o</sup>c showed the peaks contrasting with the hexagonal structure.

TaN coatings may achieve higher utilization assurance and almost stable corrosion potential than the uncoated low carbon steel (AISI1018). In this way, the secured illustrations would have a lower corrosion and therefore the substrate secured at 200<sup>o</sup>c, 400<sup>o</sup>c indicated best utilization corrosion for the coatings investigated inside the examinations (corrosion test with salt shower and corrosion test with fog humidity). The corrosion resistance of low carbon steel was multiplied by TaN coatings. TaN gives great assurance against consumption to the metal substrates.

The surface morphology of the films was depicted by AFM considers. It exhibits the smooth morphology of the films with a uniform dispersal of the valuable crystals and most of the grains had a comparative size. The roughnesses of the films are 32.088nm and 42.769nm at 200<sup>o</sup>c and 400<sup>o</sup>c independently. In the TaN thin films, the surface topography is made out of gatherings of a comparable size with irregular shapes. It is obvious from the AFM micrographs that roughness increases with increase in the temperature of the covering. Micro hardness of the illustrations was 171GPa and 150GPa for 200<sup>o</sup>c & 400<sup>o</sup>c independently. By this, we assume that as the temperature of the covering extends hardness decreases.

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3. Fabrication and tribological behavior of sputtering TaN coatings (Kun-Yuan Liu, Jyh-Wei Lee Fan-Bean Wu)
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