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"Characterization and Tribological Evaluation of Tin Thin Films Deposition by Reactive Magnetron Sputtering Process of SS304"

Mohd Aslam [1]
Gautam Budha University
Gautam budh nagar Greater Noida U. P. INDIA

Dr Satpal Sharma[2]
Head Of Mechanical Engineering
Gautam Budha University
Gautam budh nagar Greater Noida U. P. INDIA

Mohd Usman Ahmad[3]
Gautam Budha University
Gautam budh nagar Greater Noida U. P. INDIA

Anam Parveen[4]
Gautam Budha University
Gautam budh nagar Greater Noida U. P. INDIA

***Abstract*:- The purpose of TiN films coating is prevent from wear, corrosion and improve hardness of material and increase the life of material. This is more economical and cheap than radio frequency sputtering because here coating are possible at low voltage than radio frequency sputtering. The TiN is a group of meta stable hard coating consisting of metallic element Titanium target and supply of Nitrogen gas. TiN can be deposited either by cathodic arc deposition or magnetron sputtering. . Generally cathode is made of TiN of the required composition. However independent Ti targets (cathodes) have also been used for cathodic arc deposition. We have developed a balanced closed field dual cathodic magnetron sputtering system which is 1000 gauge at side and 300 gauss at center for the successfully deposited of TiN. In this project we will use independent two Ti cathodic Target in our reactive magnetron sputtering system to deposit TiN . By adjusting voltages independently on the two targets (cathodes) of Titanium it will be possible to deposit TiN of the desired composition.**

INTRODUCTION

The TiN thin film uses in many applications like thermal barrier coating for reduce heat loss during flow of heat through the barrier in the microelectronic devoice in microelectronic industry(1).This is hard and protective coating on the cutting tool also uses on solar cell,wear resistance coating on the steel. We are uses in many PVD techniques have been used to deposit the TiN thin films of different substrates.But we are using here Reactive magnetron cathodic arc sputtering process and without bias for the coating. Initilly prepare the subtract material ss304 and gives mirror polish by uses SiC abrasive paper at medium to high rotary speed as (shown in figure) as increases paper number after that uses diamond paste uses for the final mirror polish finishing. After that we start the rotatory pump and pressure carried in 1×10^{-2} mbar order in the vacuum chamber. Now start the cooling fan for cooing of water then flow of the cooling water passes through the DP .after the cooled DP start the DP and vacuum pressure carried out 1×10^{-5} mbar order .Now start flow of gas N_2 and Ar gas across the chamber again pressure increase for adding of gasses in vacuum chamber and pressure will obtained in 5×10^{-3} mbar to 1×10^{-2} mbar .Now Start the DC supply and glow discharge maintained for the 1 hour .

I. PROCESESS OF SAMPLE PREAPARING PROCEDURE

Initially prepare the substrate material we use 10 mm diameter and 8 mm thickness sample figure (2). first of all gives mirror polish of sample by using SiC abrasive paper and abrasive paper numbers like 240,600,1000,1200,2000 as shown in the figure (4).As increases abrasive paper number then decreases grain size of abrade particle. So remove all scratch from sample finally from 2000 paper gives like pre mirror then after the pre mirror polish using diamond paste as shown in figure (3) this is 7 micron white paste a little amount on cloth paper with drop of water on the rotating machine as shown in figure (1)which gives final mirror polish of the sample as shown in the figure (2). Thus the sample is ready for the reactive magnetron sputtering coating. We want to coating on the SS304 and chemical composition as shown in table(1).

Table 1

Fe	Cr	Ni	Mn	Si	P	S	C
Bal%	18-20%	8-12%	<2%	<1%	<0.045%	<0.03%	<0.08%

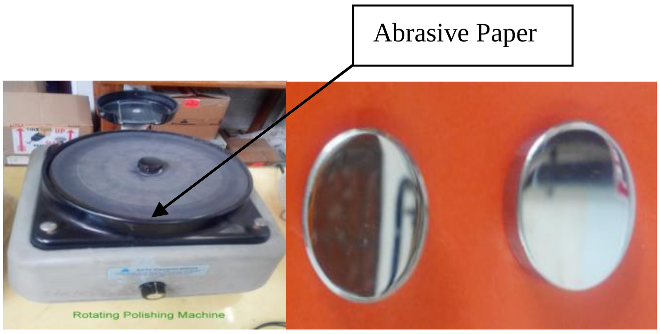


Figure 1 (Polishing Pad)   Figure 2 (Mirror Polish Sample)

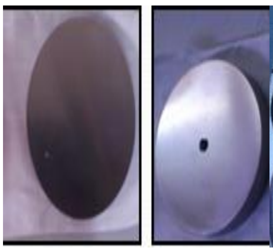
Figure (3) 7micron diamond paste Fig   (4)   SiC Abrasive Paper

II . TARGET AND MAGNET

During experimental work one important thing uses target which have 60mm diametre and 10 mm thickness and in the centre 0.5mm dia and which have 5 mm deep hole with thread for the supporting of both side and we use magnet 1000 guass both side and at centre 300 gauss .The purpose of magnet uses for in the experiment that experiment perform at low voltage and current as compare to RF sputtering processes as show in the figure A and B. Target is the made of Titanium Ti.

Chemical composition of Titanium target material is given table (2)

C	Si	Mn	Cr	Ni	Fi	Cu	Nb	V	Al and Ti
9%	8%	6%	1%	6%	8%	5%	4%	3%	6 ,9%



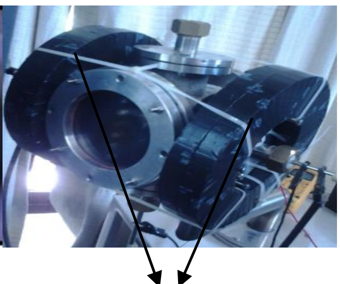


FIGURE (A)   FIGURE (B) MAGNETS

WORK EXPERIMENTAL PROCEDURE

Initially start the rotatory figure (5) pump and before start the pump close all valves. Thus the gas from vacuum is suck by pump and led in the space or environment and pressure in between 1 × 10^{-2} to 8×10^{-3}mbar depend on weather and condition of the pump as shown in figure(6).

Figure (5) Rotary vacuum Pump   Fig (6) Pressure Gauge

After the pressure come in order of 1×10^{-2} to 8×10^{-3} mbar start the cooling fan for the cooling of water .as soon as possible cooled water start the pump for circulating water inside the Diffusion Pump . After cooling DP Start the diffusion pump and after some time pressure come 1×10^{-5} mbar. When pressure comes around 1×10^{-5} mbar then start the flow of gass Ar for flushing after flushing Nitrogen gas also flow inside the vauume chamber and gas flow ratio controlled by MFC as show in the figure(7).High Pressure (UHP) gass uses in our experimental work as shown in figure (8).

Figure(7) MFC Figure   (8) UHP N_2 and Ar cylinder

When start the flow of gasses then pressure rises in between 5×10^{-3} to 1.20×10^{-2}mbar. One thing also included that before the start experimental work sample fit inside the vacuum chamber .Now supply DC current also and voltages gradually like current 100Amp and voltage 500 to 900 volt. After on DC
supply temperature of sample(substrate) increases gradually as show in the figure (9) .And temperature gauge connected with the substrate material and measure temperature in mV.

Figure(9) DC Supply   Figure (10) Temperature Gauge

GLOW DISCHARGE

As rises voltages more than 500 volt and current 100 Amp Glow discharge visible inside the chamber in radish color. When flow of gasses N_2 and Ar where Ar is non-reacting gass and N_2 uses for as a reacting gass . N_2 gass react with Ti and TiN coating on substrate material as shown in figure(11)

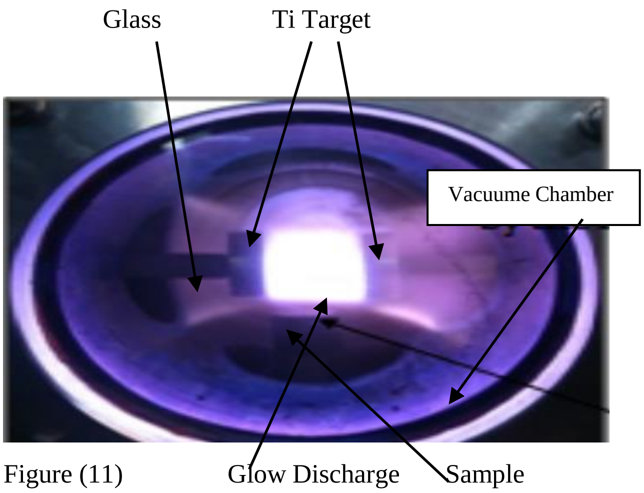


Figure (11)

DC supply on for 1 hour or one and half an hour during in this period temperature rises of sample (substrate). Voltage and current try to constant in this period then during glow Discharge also constant maintain pressure during experimental also. Now thus TiN coating done it will gives golden color as shown in figure (12)

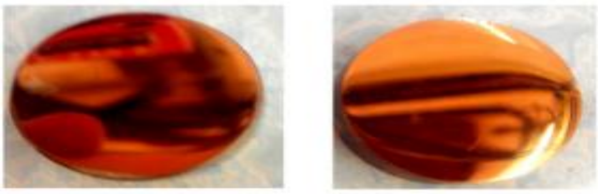


Figure (12)

IV. EXPERIMENTAL WORK SETUP

According to reactive magnetron sputtering initially sample fit inside the chamber and chamber closed . in this experiment uses magnet which uses for the experiment done at low voltage and current and electron and ions in the alignment but in the RF sputtering process and electron and ions alignment at high voltage so overall cost increases. First of all start the rotary pump and pressure occurred 1×10^{-2} mbar after that start the cooling fan for the cooling water then cooled water supply through the DP and cooled the DP .Now start the DP and after some time Pressure decreases and obtained 1×10^{-5} mbar .now start Ar supply for the sputtering and flushing also after that supply of N_2 add. Now pressure increases and obtained 1×10^{-3} mbar nearly half an hour flow continue then start the DC supply voltage nearly 500 to 900 volt and current 100 Amp without bias. As voltage reaches at 500 volt glow discharge appear in vacuum chamber maintain the voltage, current and pressure for one hour or more depend on the experiment during in this period pressure gauge and temperature gauge must be on in condition. Thus experiment have to done as shown in the figure (13).

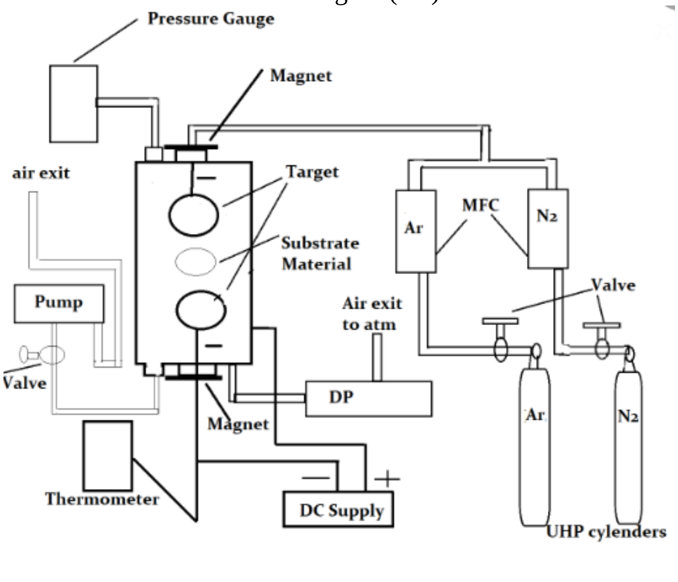


Figure (13) PAPVD SETUP

V. RESULT AND DISSCUSSION

As in mention below we uses processes and parameter in our experiment and obtained TiN coating as in the figure(12) this is a hard coating and golden color obtained. This is surface modification process improve corrode wear and tear as shown in the table

Table .3 Proses and Parameter

Experimental Data	**Process Parameters for the Sample**				
Ar and N_2 ratios	S1 95:05	S2 90:10	S3 90:1	S4 90:10	S5 90:10
Room temperature(^{0}C)	22	22	22	23.5	24
Base pressure (mbar)	1.48×10^{-5}	1.5×10^{-5}	1.30×10^{-5}	1.34×10^{-5}	1.58×10^{-5}
Ar flow (ml/min)	18.8	18	18	18	18
N_2 flow(ml/min)	1	2	2	2	2
Voltage (V)	690	900	600	590	800
Current (mA)	80	50	100	100	100
Work pressure(mbar)	6×10^{-3}	5.5×10^{-3}	7×10^{-3}	6×10^{-3}	7×10^{-3}
Substrate bias (V)	-100	-100	-100	-100	-100
Substrate target distance(mm)	37.1618	25	25	25	25
Thermo electric voltage (mV)	1.4	2.8	1.9	2.5	2.5
Substrate temperature(^{0}C)	57	91	70	85.5	88
Deposition Time (minute)	60	60	120	120	60

V.1. EFFECT ON THE TARGET

After the experiment we seen that target is sputtered by N_2 when N_2 atom strike on the Ti target a large number of atom and tear out from Ti target as show in the figure (14.a) .This is a plasma state and coted TiN thin film on the SS304 sample .

Target material Ti after the experiment like that as shown in the figure (14.a) front side and figure (14.b) is back side of the target. If i want to do another experiment then simply by uses SiC abrasive paper 240 number and remove sputtered layer from the surface. We uses petroleum ether after removing sputtered layer and for the cleaning of vacuum chamber also.

Figure (14.a)

Figure (14.b)

Thickness Measurement of the TiN Thin Films

For the thickness measurement the samples were cut with the help of a low speed precision cutting machine of a Chinese "Jinan Kason Testing Equipment's Co." make and model: DTQ-5 as shown in Fig. (15.a) The cutting speed can be varied from 30 to 60 rpm. During cutting operation, the cutting disc gets heated, and may break during the operation. A coolant is used for the smooth cutting action and to prevent breakage of the cutting disc due to overheating. The cut cross-sections of the samples were taken for mounting. The mounting press is of MTI Corporation make and type XQ-2B as shown in Fig. 15.b. The materials used for mounting the samples were red, green and black colored phenolic resins. During mounting, the temperature is set to 185 ºC, once the temperature is reached, the pressure is applied. The temperature and pressure conditions are maintained for about 12-15 minutes for obtaining a compact mount.

Figure (15.a)

Figure (15.b)

Figure (15.c)

Characterization of deposited TiN Thin Films

XRD RESULTS:

Figure (16) shows the XRD pattern for a bare SS 304 sample which is the substrate material. The substrate elements are observed which are Fe, Cr and Ni respectively. The peaks and d values match with the ICDD standard 01-071-7594 (Cr Ni) and JCPDS card for Fe-γ (austenite).

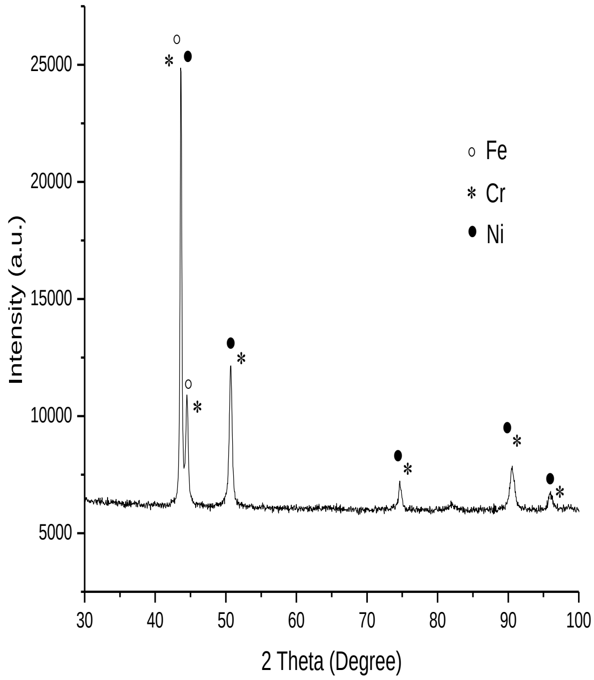


Figure (16.a)

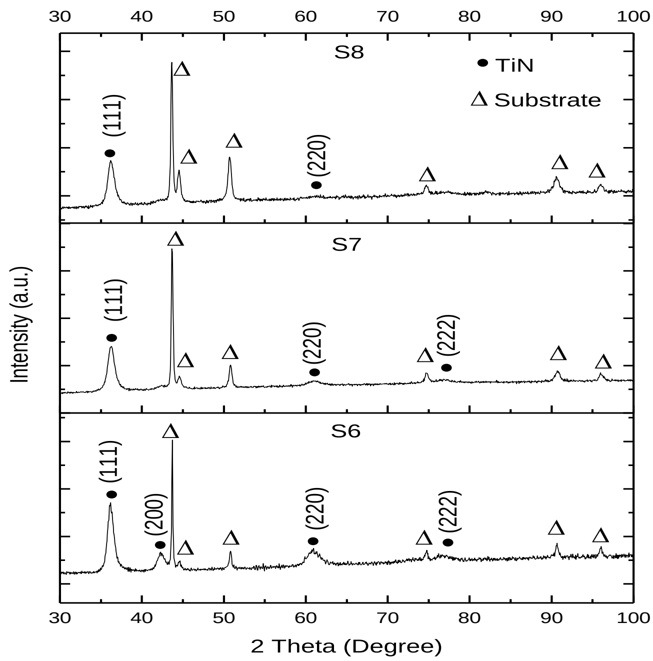


Figure (16.b)

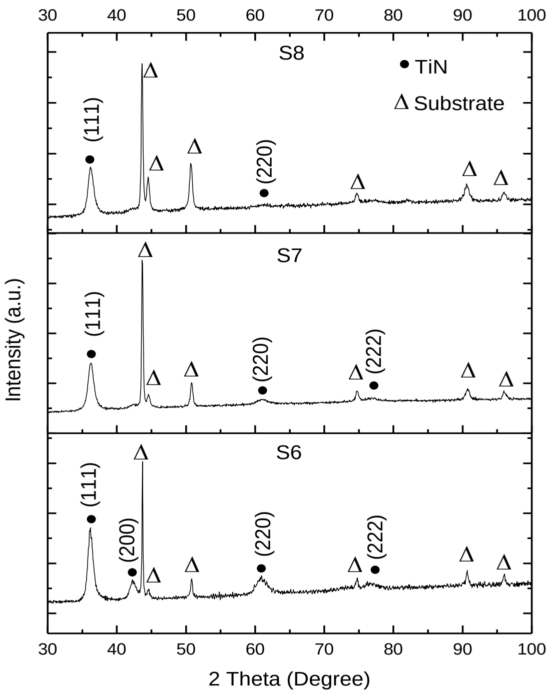


Figure 16.c)

Tables 16.a, 16.b and 16.c give the XRD data of samples S2, S4 and S6. The reflections from planes (111), (200), (220) and (222) with d spacing 2.48, 2.14, 1.53 and 1.23 matches with standard ICDD file number 00-038-1420 for TiN phase. Also the first peak having d-spacing of 2.54 matches with the JCPDS card of Ti showing reflection from plane (010) Thus the samples S1, S2 reveal the presence of both TiN phase with these different orientations.

Fig.(16) XRD Plot for Bare SS 304 Sample

By uses kind of parameter we can perform another experiment.

Table 16.a: S2: 95 % Ar – 5 % N_2, 60 minutes deposition					
No.	**2 theta**	**d observed**	**d standard**	**Plane**	**Phase**
1	35.17	2.54	2.55	(010)	Ti
2	36.40	2.46	2.45	(111)	TiN
3	42.08	2.14	2.12	(200)	TiN
4	77.17	1.22	1.23	(222)	TiN

Table 16.b: S1: 90 % Ar – 10 % N_2, 60 minutes deposition with Ar plasma Sputtering					
No.	**2 theta**	**d observed**	**d standard**	**Plane**	**Phase**
1	35.88	2.49	2.45	(111)	TiN
2	41.99	2.14	2.12	(200)	TiN
3	60.33	1.53	1.49	(220)	TiN

Table 16.c: S2: 90 % Ar – 10 % N_2, 120 minutes deposition without Ar plasma Sputtering					
No.	**2 theta**	**d observed**	**d standard**	**Plane**	**Phase**
1	36.25	2.47	2.45	(111)	TiN
2	77.07	1.23	1.22	(222)	TiN

SEM-EDAX Results

Sample S1: Ar:N_2 ratio 95:5

For sample S1, the film deposited was uniform and the SEM result shows no signs of cracks or voids in the deposited film. The microphotograph showing the surface features of the film at 1000 X magnification is shown in Fig. 17 (a) and Fig. 17 (b) shows the thickness of the TiN film at 10000 X which is 1.887 µm. The SEM image of the cross section shows surface oriented morphology of the film (Fig. 17(b)). Table 4 shows the chemical composition of the elements on the surface which clearly reveals the presence of Ti and N.

Table 4

Element	Weight%	Atomic%
N K	21.65	48.58
Ti K	78.35	51.42
Totals	100.00	

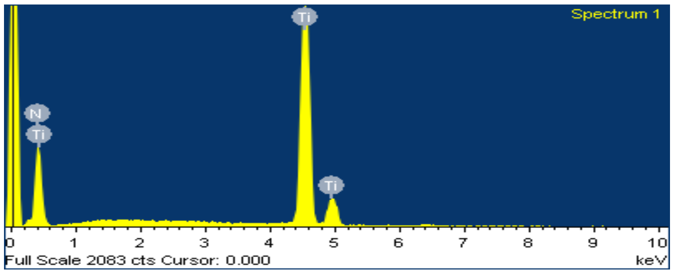
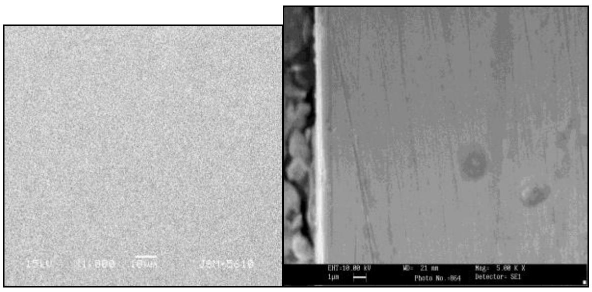


Fig.17: EDAX Graph for Sample S1



(a) **(b)**

Fig. 17 (a) The top surface of the film fig.17 (b) Cross section of sample S2 showing the thickness of the film

Sample S2: Ar:N_2 ratio 90:10 with Ar plasma sputter cleaning

In sample S2, the deposited film shows no sign of voids or cracks and it shows densely packed morphology as can be seen in Fig. 19(a) and the thickness of the film is around 1.428 µm shown in Fig. (b). Table 5 shows the chemical composition showing Ti and N elements revealing the formation of pure and dense TiN film.

Table 5

Element	Weight%	Atomic%
N K	20.51	46.88
Ti K	79.49	53.12
Totals	100.00	

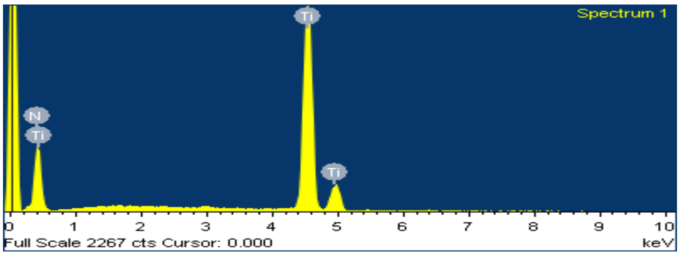
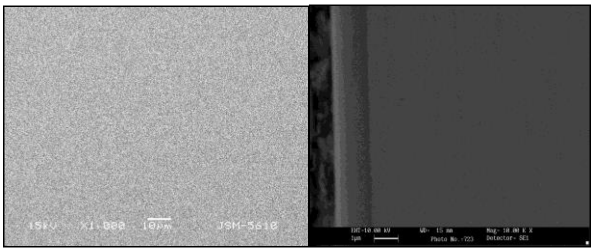


Fig. 18: EDAX Graph for Sample S2



(a) **(b)**

Fig. 19 (a) The top surface of the film (b) Cross section of sample S2 showing the thickness of the film

PROPERTY EVALUATION:

Surface Hardness Results:

The surface hardness results of all the TiN deposited samples and the bare SS 304 sample are shown in tables 6 to D_1 and D_2 are the diagonals of the indentation. D is the average of D_1 and D_2. Six to seven readings have been taken for the TiN deposited samples for measuring the average surface microhardness of the film. The observation of these results shows that the measured values are having minor differences and the microhardness values are quite uniform.

Table 6: Surface microhardness of Bare SS304 sample S1

Bare SS 304 Sample F = 10 g (load)	**D_1**	**D_2**	**D= (D1+D2)/2**	**HV = 1.854 F/D^2 (HV)**
-	6.93	7.4	7.17	361.14
	7.73	7.39	7.56	324.39
	6.95	7.23	7.09	368.82
	7.08	7.68	7.38	340.41
Average Hardness (HV)	348.69			

Table 7: Surface microhardness of sample S2

Sample S2 F = 10 g (load)	**D_1**	**D_2**	**D= (D1+D2)/2**	**HV = 1.854 F/D^2 (HV)**
-	4.4	4.97	4.69	844.68
	4.53	4.59	4.56	891.62
	4.12	4.22	4.17	1066.20
	4.33	3.97	4.15	1076.50
	4.27	3.92	4.10	1105.61
	4.24	4.08	4.16	1071.33
	4.25	3.83	4.04	1135.92
Average Hardness (HV)	1027.41			

Table 8: Details of the Results Obtained

Sample	S1	S2	S3
Ar:N_2	95:5	90:10	90:10
Time(min)	60	60	60
Voltage(V)	690	900	600
Current(mA)	80	50	100
Power (W)	55.2	45	60
Substrate Temperature (^{0}C)	57	91	70
Working pressure(mbar)	6 x 10^{-3}	5.5 x 10^{-3}	6 x 10^{-3}
Thickness **(µm)**	1.8	1.428	1.2
Average Micro-hardness	1027.41	2228.06	3120.57

CONCLUSION

Magnetron plasma source has been characterized in this work by depositing TiN thin films a new type via DC reactive magnetron sputtering with different nitrogen concentration on SS 304 substrates. The average deposition rate observed for different N_2 concentrations is about 1.20-1.25µm/hour. The SEM results show that up to 60% N_2 concentration, the TiN films deposited are thicker, denser and continuous showing a densely packed, void free and crack free morphology. The gas ratio 60% Ar and 40% N_2 resulted into a higher deposition rate of 1.45µm/hour and the average microhardness was also observed to be around 3000 HV. Also the XRD results reveal the presence of stoichiometric TiN. The films deposited with a low N_2 concentration are metallic golden in color whereas those deposited with a higher N_2 concentration are brass-golden in color.

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