Characterization CompareProcesses and Parameter of SS304 between Nitriding and Thin FilmDepositionofTiNby Reactive Magnetron Sputtering

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ABSTRACT

From last few decades we are using thin film coating and nitriding for the improvement of surface modification. This is also improving mechanical properties wear, corrosion and improving service life of the material, Nitriding is improve hardness of the material fromsurface to some depth and ultimately increase toughness, hardness and also Improve wear resistant of the material and increases productivity. Coating gives different different color depend on the what will be used target for the coating like TiN gives gold color, here we are using two Ti target and flow of reacting gas is Nitrogen . The TiN coating is improving hardness up to 24 Nano-meter thickness will the 1 to 7µ depend on time of experiment and substrate temperature. The uses of TiN thin film coating in universal use, coating on the tool material, forming like molds and dies and also uses for the decorating purpose.

We are using Ti Target and reactive magnetron sputtering process for the coating of TiN on the substrate material SS304 and also for the nitriding H_2 gas uses for the flushing and H_2+N_2 gas uses for the nitriding at a certain temperature and pressure inside a vacuum chamber. Here we are characterizing MicrohardnessTest, X-Ray Deposition, and Energy (XRD)Dispersive Spectroscopy (EDS) Field Emission Scanning Electron Microscopy (FESEM).

The purpose and conclusion of various analysis's is that improve the hardness, corrosion, wear resistance and toughness of the material ss304 by using of reactive magnetron sputtering (RMS) and Nitriding process.

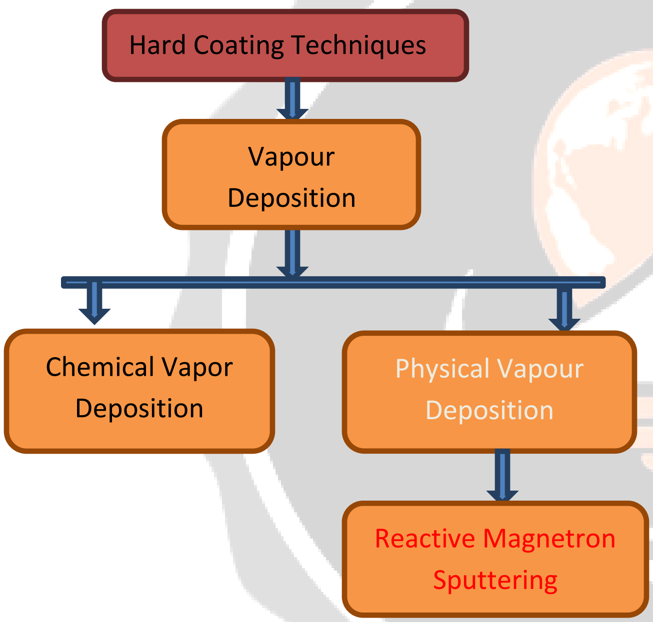
Keyword: *-RMSP1, Coating2, Nitriding3, EDX4, XRD5, Microhardness Test6*

1.1 INTRODUCTION

For the hard coating of material we are using many techniquesas shown in figure (1)[1] below but we are using reactive magnetron sputtering process. This is the classification of physical vapour deposition (PVD) technique. According to reactive magnetron sputtering process we are using Magnet of wheel on both side which have 1000 gauss on both side but at center where coating isperformed on the substrate sample it has to occurs nearly 300 gauss .The TiN coating uses for the commercial purpose for forming and decorating purpose, it has golden color so nice in looking and hard so we are using in many application like thermal barrier coating, cutting tool, watches.

So overall cutting performance and tooling life is increases and Cr-based coating are using for the industrial tools.[2] According to TiN coating we use two Ti target and substrate material SS304 by applying the process reactive magnetron techniques at the certain gases flow rate Ar and N_2under a certain temperature and pressure we are performing TiN coting and details described in the experimental work.

1.2 Nitriding is a heat treating process that N_2 diffuses into the surface of metal. [3]Initially material polishing process of nitriding is same as the thin film coating of TiN. Nitriding is done at high temperature as compare to thin film coating by reactive magnetron sputtering and nitriding is done at high pressure as compare to TiN coating by reactive magnetron sputtering .In a case of nitriding diffusion pump is not required but in TiN coating diffusion pump is required because it has to coating occurs at low pressure in 4×10^{-3} to 1×10^{-2}mbar. In the nitriding is hardness is obtained but in TiN coating occurs a thin layer 1 to 7 µ depend on time and flow rate of gasses. Plasma nitriding also known as ion, plasma ion, and glow discharge nitriding it is an surface hardening treatment for metallic materials hard layer to protect the metal.[4] Classification of nitriding described below as shown in the figure in figure 2.



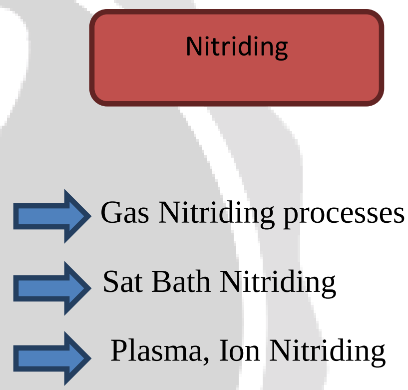


Fig 1 Classifications of Hard Coating Techniques Fig 2 Classification of Nitriding

In the above figure we are only focused on the reactive magnetron sputtering process as given in the hard coating technique’s and process. Also in figure2 nitriding is process diffuse nitrogen in to the surface of the metal under a high temperature and high pressure. Various nitriding process of comparison is given below in Table1. [1]Also described in figures in fig3 as shown in figure below how to vary temperature and processes. [1]

	Applicable Steel Types	Nitriding Performance		Low temperature	productivity	Load on the Environment	Costs
		Hardness Depth	Control White Layer				

Gas Nitriding	× Carbon steel Alloy steel	× Significantly affected by surface properties	× Nitriding capability is low and it is difficult to control the white layer	× Difficult	◎	○	◎ Mass-producibility is good and internal production is possible
Salt Bath Nitriding	○ Carbon steel Low and high alloy steels Stainless steel Cast iron	○ Significantly affected by shape and salt bath	× Batch-by-batch adjustment is impossible	○ Possible to a certain extent	◎	×	◎ Mass-producibility is good but internal production is very difficult
Ion Nitriding	○ Carbon steel Low and high alloy steels Stainless steel Cast iron	○ Significantly affected by shape of workpiece	○ Controllable to a certain extent	○ Possible	× Low	◎	× Mass-producibility is not good

Table 1 Comparison of Various Nitriding Processes

Heating Pattern and Treatment Process during Nitriding

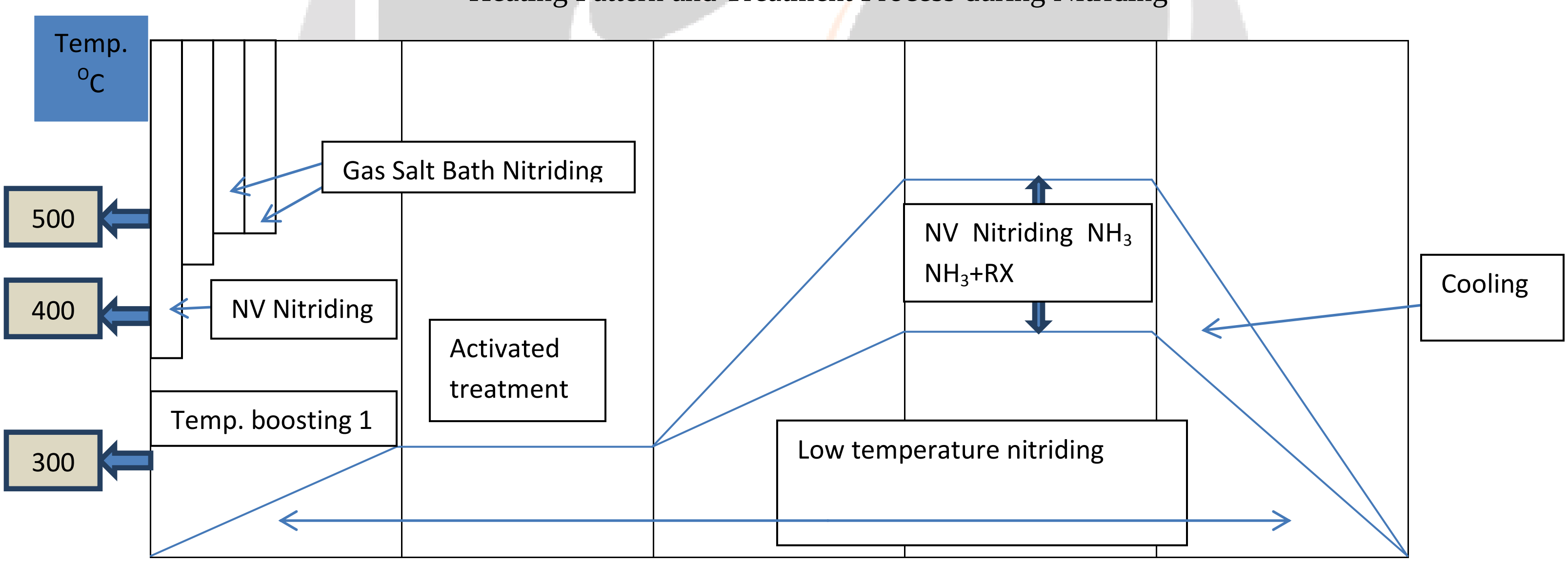


Fig 3 Heating Pattern and Treatment Process

1.3 REACTIVE MAGNETRON SPUTTERING

In the reacting magnetron sputtering process we are sing two titanium (Ti) target which have 60 mm diameter and 10 mm thickness and substrate material (Sample) have 20 mm diameter ,8 mm thickness .Flow of gasses are N_2 and Ar where N_2 use as a reacting gas and Ar use for flushing and for the sputtering. During coating working pressure is in range of 10^{-3}mbar pressure if pressure is between $4\times10^{-3}$to 1×10^{-2}mbar then it will gives best coating, And temperature varying in respect to what will be voltage and current provide. Generally voltage and current in between

-500 to-1000 volt and current uses 60 to 100 mA. This will gives best glow discharge during coating of TiN and radish color appear between target as show in figure 5 below. It will uses one side transparent glass and see from that side .Reactive magnetron sputtering process is done by setup like fig 4 as show in the figure below.

Fig 4

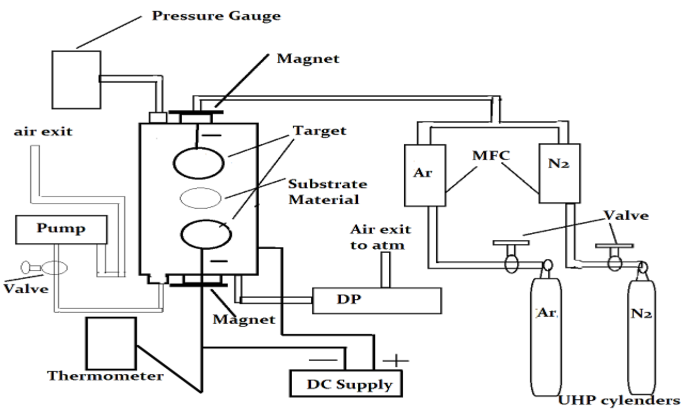
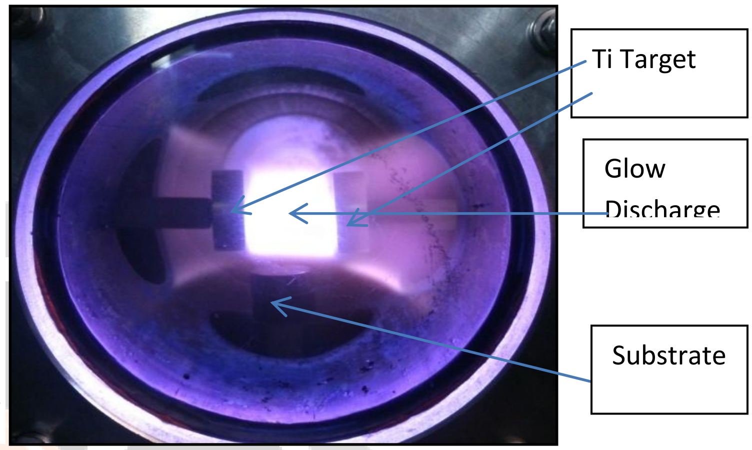


Fig. 4 Reactive Magnetron Sputtering

Fig 5



1.4 GLOW DISCHARGE NITRIDING

Nitriding is the process that diffuse nitrogen into the surface of metal .which have increase the life like after the nitriding of tool material increase the tooling life of the material . According to glow discharge nitriding have high pressure and temperature in between 300 to 500. Initially H_2 uses for the flushing for 1 hour it has nearly 48 ml/min it's have also control by mass flow controller. After that we use N_2 gas for the diffusion on surface it has under a pressure and temperature timing in between 3 to 5 hour. In the flushing glow discharge like blue color and when supply of N_2 is added then discharge color change into radish color. In the glow discharge nitriding is not necessary target material and diffusion pump remaining process is same like reactive magnetron sputtering. Diffusion pump uses for the pressure led to lower but in the nitriding have to done at high pressure so rotatry pump is enough for the nitriding. Glow discharge is similar to like fig 6 as shown in figure below. Figure 6 represent is Ar, N_2, and H_2 glow discharge.

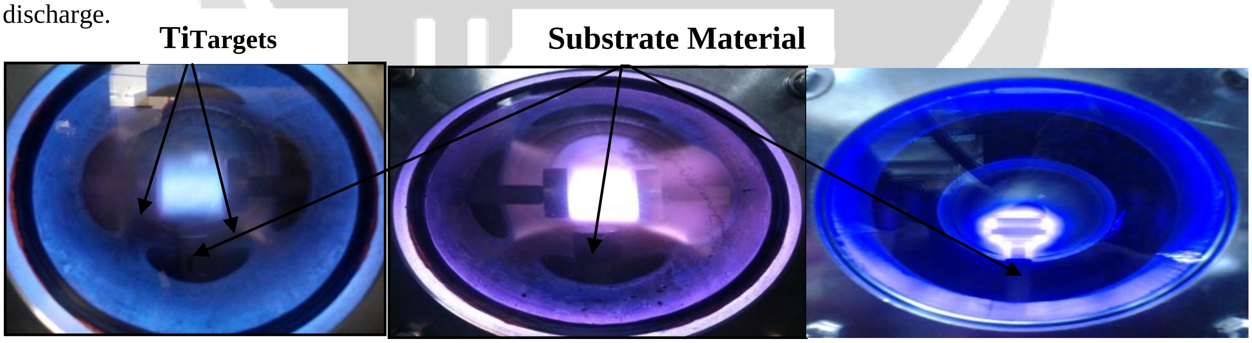


Fig6a Argon Magnetron Plasma Fig 6b Ar + N_2 Magnetron Plasma Fig 6c Hydrogen Plasma

2 EXPERIMENTALPROCEDURES

2.1 Selection of substrate materialand polishing processes:

We are using SS304 material which have easy to available excellent toughness better in forming and good quality of welding properties. It hasease of cleaning and fabrication as well as good deep drawing propersties. The application of SS304 also use as a nut, bolt ,spring watches, cutting tool, kitchen purpose, heat exchanger in tube boiler, screw, piping valve storage and hauling tank for kitchen purpose, tubing , and improve weldibility etc. The chemical composition table 2 is as given below of SS304 and mechanical and physical properties is given below Table number 2, 3, 4. [2]

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

Table 2: Chemical composition of SS 304

Grade	Tensile Strength (MPa)	Yield Strength 0.2%prof(MPa)	Elongation(% in 50mm)	Rockwell B (HR B) max	Brinell (HB) Max
SS304	515	205	40	92	201

Table 3: Mechanical Properties of SS304

Grade	Density	Elastic modulus(GPa)	Mean Coefficient of Thermal Expansion(µm/m/^{0}C)			Thermal Conductivity (W/m.K)		Specific Heat (J/kg.K)
			0-100^{0}C	0-315^{0}C	0-538^{0}C	at 100^{0}C	at 500^{0}C	0-100^{0}C
SS304	8000	193	17.2	17.8	18.4	16.2	21.5	500

Table 4: Physical Properties of SS304

Cylindrical samples with 8 mm thickness were cut from a rod of SS304.The substrate samples is prepared by grinding and polishing by the conventional procedure of sample preparation to achieve mirror polished surface. The polishing machine of MTI Corporation used is as shown in fig 2.1. Initially we use for polishing of sample SiC abrasive paper number 240,600,1000,1200,2000. This all scratch remove from the surface by using this abrasive paper finally 2000 number paper gives pre mirror polish. After that we use diamond pasteof 7µ and with uses 7µ cloth. Thus we obtained mirror polish sample. This process use for both reactive magnetron sputtering and plasma nitriding also. The polishing machine and diamond paste and abrasive paper as shown in figure 2.2 , figure2.3.

Fig 2.1 Polishing Machine

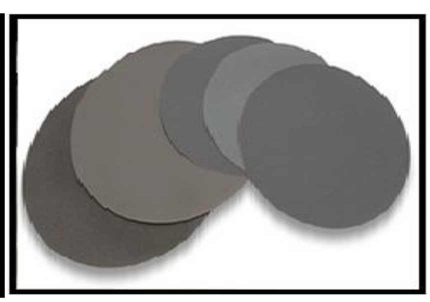
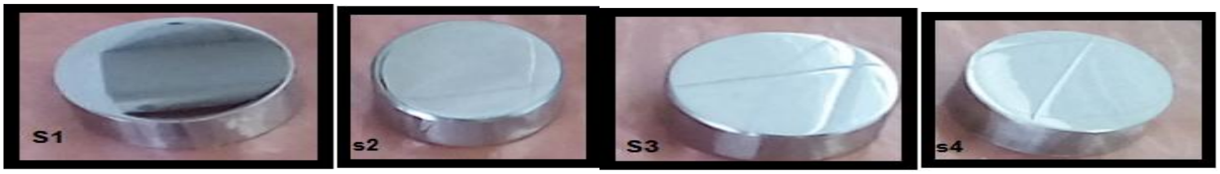


Fig 2.2 Abrasive Paper

Fig 2.3 Diamond Past

According toprocess above figure we use polishing machine abrasive paper and diamond paste figure 2.4.



2.2 Target Design and chemical composition:

In the reactive magnetron sputtering we use twoTi target which have chemical composition as given below in the table 5. The diameter of the target is 60mm and 10 mm thickness it's also has in the center 0.5mmdiameter and 5mm deep hole as shown in figure 2.5

C	Si	Mn	Cr	Ni	Fe	Cu	Nb	V	Al	Ti
0.009 %	0.008 %	0.006 %	0.011 %	0.006 %	0.098 %	0.025 %	0.024 %	0.003 %	0.276 %	99.529 %

Table 3.2 Chemical composition of Titanium target material

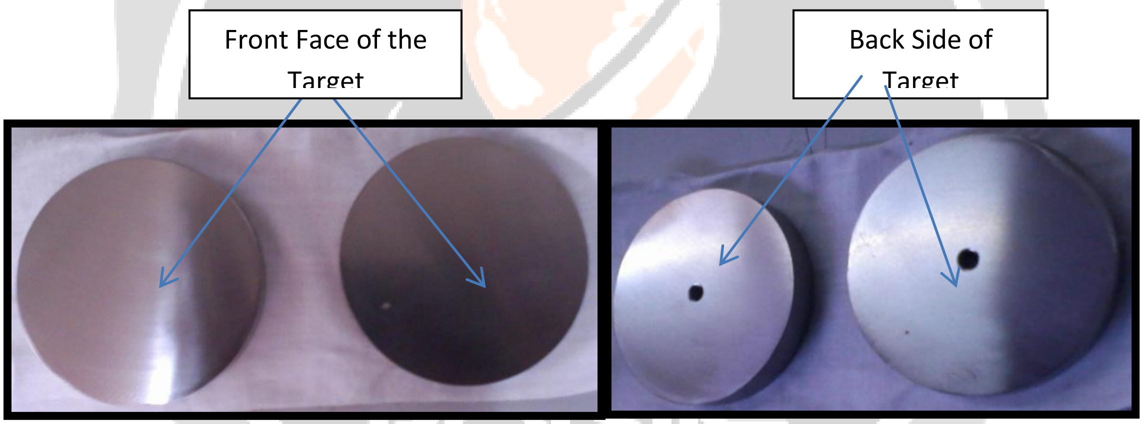


Fig. 2.5: Titanium targets of 60 mm diameter and 10 mm thickness

2.3 Working Procedure of Reactive Magnetron Sputtering:

Initially fit the sample inside the vacuum chamber and tight both side Ti target as shown in the figure 6a, b. Now closed the vacuum chamber and close all vacuum chamber valves and start the rotatory pump after few min later pressure of vacuum chamber is achieve in 1×10^{-2}mbar order .Now after achieving pressure 1×10^{-2}mbar order start cooling fan for cooling of water when water will cooled then start cooling pump which have function circulating water across a Diffusion Pump. Now after cooling of diffusion pump start the diffusion pump and after start diffusion pump pressure decreases and led into a 1×10^{-5}mbar order. Now after achieving pressure in 1×10^{-5}mbar order start gas flow of Ar for the cleaning and flushing of chamber this operation is nearly 30 min after N_2 gas and Ar both on for 30 min, Gas flow rate controlled by mass flow controller (MFC) for providing gas flow ration across the vacuum chamber. Now start the DC power supply voltage and current like -500 to 1000 Volt and current 52 to 100 mA without bias. Now Ti target react with Nitrogen gas and make TiN which is deposited on the on surface of material of SS304, Here N_2 uses as a reacting gas and Ar uses for the sputtering. Glow discharge look like a fig 6b. During experiment temperature rises and reading take in mV this is take corresponding value from temperature and mV relation chart Now after completing our experiment 1 hour for TiN coating going reversed process in last open chamber and clean target ,chamber by Petroleum Ether .after that continue our next experiment. As mentioned in figure below in 2.6 is complete processes.

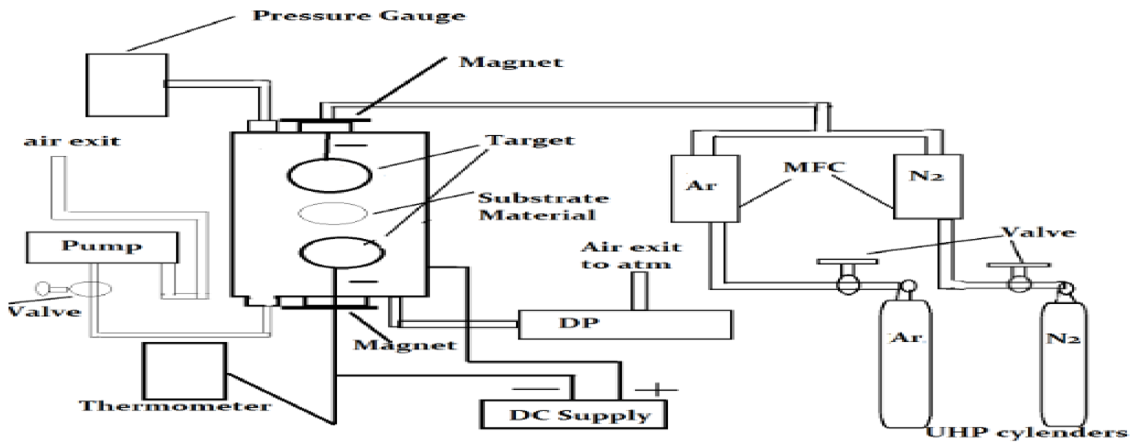
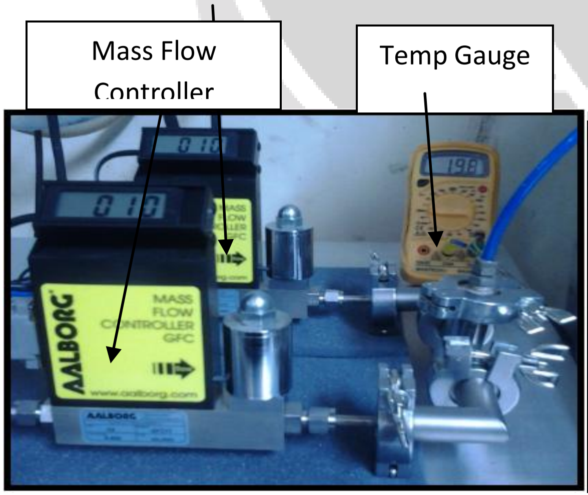


Fig 2.6 Reactive Magnetron Sputtering

2.3 Working Procedure of Plasma Nitriding: Sample preparing process is same like RMSP and mirror polished, here also uses mirror polish sample. In the plasma nitriding is not required DP, Target, and Magnet rather than remaining proses is same like reacting magnetron sputtering proses as show in the fig 2.6. Here uses H_2 for the flushing and N_2 uses for the diffuse N_2 on the surface of metal SS304. Initially start the rotary pump and pressure carried out in 1×10^{-2}mbar. After that on the cylinder of H_2 for the flushing nearly 1 hour and H_2 DC plasma for the cleaning as shown in the figure 6c H_2 plasma obtained before the N_2 diffusion after that both hydrogen and nitrogen gas cylinder on. On DC supply Voltage and current given depend on the what will be use temperature for the glow discharge for the nitriding purpose. Temperature controlled by Voltage as increases voltage correspondingly increases temperature. This condition maintained during experiment after completing our experiment going to reverse closed all on thing. Open chamber clean by petroleum ether and now this ready for next experiment. Mass flow controller and chamber as shown in the figure below in the Fig 2.7a, 2.7b.



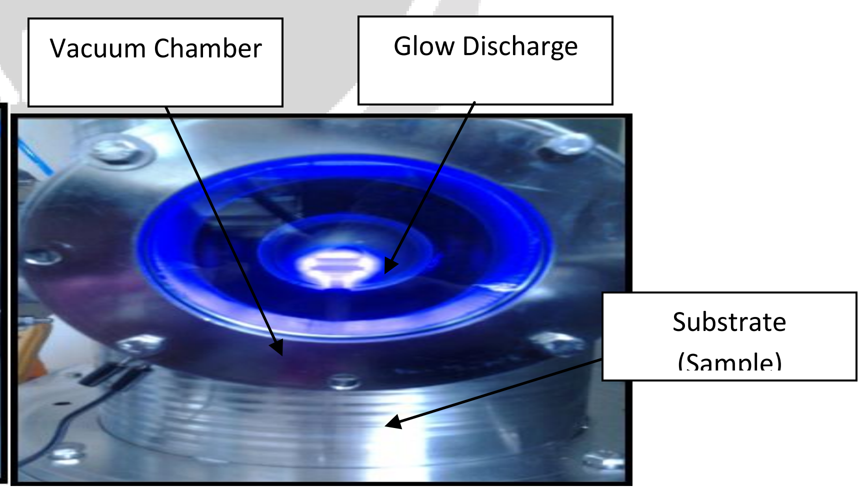


Fig 2.7 a Mass Flow Controller

Fig2.7b Vacuum Chamber

2.4 Parameter of RMSP: The magnetron sputtering process parameters of deposition of TiN on SS 304 substrates are as given in below in the table. The important parameters during the deposition are Ar:N_2 ratio, plasma sputtering by argon prior to deposition, working pressure and time duration of deposition. The TiN deposition on SS 304 by magnetron sputtering process were carried out keeping some of the variables like deposition duration (60 minutes), target-substrate distance (25 mm) and base pressure (approximately 1.3 x 10^{-5} mbar to 1.5 x 10^{-5} mbar) constant. To study the formation of TiN thin film based on the argon to nitrogen ratio during the process, the ratio Ar:N2was varied i.e. 95:5, 90:10, 80:20, 70:30. Table 2.4.1 shows this data. The detailed parameters at different Ar:N_2 ratios for TiN deposition by magnetron sputtering process are as given in Tables 2.4.2.

Sample	Ar:N_2 Ratio	Sputtering Power (W)	Time (minutes)	Temperature of substrate during deposition	Working Pressure (mbar)
S1	95:5	55.2	60	57 ºC	6 x 10^{-3}
S2	90:10	80	60	88 ºC	7 x 10^{-3}
S3	80:20	80	60	63 ºC	4 x 10^{-3}
S4	70:30	121.5	60	120 ºC	4 x 10^{-3}

Table 2.4.1: Variation in Ar:N_2 ratio during Magnetron Sputtering with other Variables

Parameter	S1	S2	S3	S4
Room Temp.(ºC)	22	24	25	25.5
Base Pressure(mbar)	1.48 x 10^{-5}	1.58 x 10^{-5}	1.30 x 10^{-5}	1.34 x 10^{-5}
Ar Flow Rate(ml/min)	18.8	18	16	14
N_2 Flow Rate(ml/min)	1	2	4	8.6
Voltage(V)	-690	-800	-900	-930
Current(mA)	80	100	100	100
Working Pressure(mbar)	6 x 10^{-3}	7 x 10^{-3}	4 x 10^{-3}	5 x 10^{-3}
Substrate Bias(V)	-100	-100	-100	---
Substrate-Target Distance(mm)	37.1618	25	25	25
Thermo-electric Voltage(mV)	1.4	2.5	3.7	3.7
Substrate Temperature(ºC)	57	88	3.7	115
Deposition Time (minutes)	60	60	60	60

Tables 2.4.2: Parameter of working reactive magnetron sputtering

2.5 Parameter of Nitriding Nitriding parameterof working procedure is given below in table 2.5.1.Initially supply of hydrogen flow across the vacuum chamber after that for hydrogen cleaning on DC supply. This is at a certain temperature as given in table below. Now flow of nitrogen is also add after that my nitriding is start this is happened a certain voltage and current as shown in table below. Now when DC supply on glow discharge like in a figure 6.c generally nitriding is occurs at 300 to550 ^{0}C and improve hardness of the material. This is generally uses for tooling in commercial purpose after the nitriding tooling life increases. When on the DC supply nitrogen diffuses on the surface of the metal. The working procedure is given below in table 2.5.1.

PARAMETER	SAMPLE 1	SAMPLE 2	SAMPLE 3	SAMPLE 3
Without DC H_2 Flushing time(hour)	1	1	1	1
Room Tem.	26	27.1	25.2	26.5
Voltage	-920	-910	-850	-930
Current	18	19	19	22
Cleaning Tem.	250	250	252	302
With DC Cleaning time	1	1	1	1
H_2 flow rate	20	20	20	20
N_2 flow rate	5	5	5	5
Nitriding Tem.	525	525	525	550
Pressure	2.58	2.6	2.6	2.59
Voltage	-640	-610	-590	-630
Current	47	47	46	49
Nitriding time (hour)	3	2	3	3

Table 2.5.1 Parameter of Nitriding processes of SS304

3. RESULT AND DISCUSSION

TiN gives gold color like in figure 3.1 it will gives hard coating of the material and nitriding also gives increases hardness of the material which is increase the service life of the material . These are method use for commercial purpose in increment of productivity. After the coating target material are sputtered like in fig 3.2 here it is clarify that DC power supply through the target.one thing is important for the coating that for the cleaning of chamber we uses petroleum ether . This parameter which is mentioned above give good result like in fig below in fig 3.1

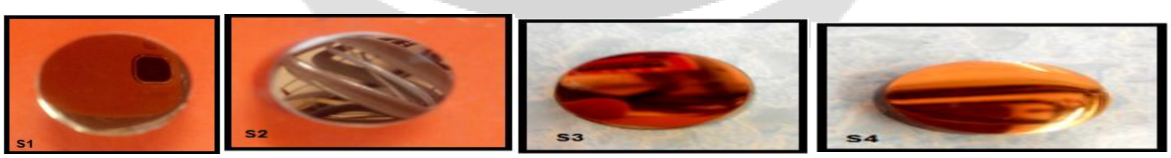


Fig 3.1 TiN Coting samples

After the sputtering target look like as fig 3.2

Fig 3.2 Titanium Target after use

3.1Comparing the hardness between Nitriding and thin film coating by reactive magnetron sputtering

According to this parameter of coating and nitriding to make hardness then now we are compare hardness of coating and nitriding of SS304 according of this parameter. The surface micro hardness of bare sample SS304 and samples S1, S2, S3, S4 and corresponding table 3.1, 3.2, 3.3, 3.4, 3.5.

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 3.1: Surface microhardness of Bare SS 304 sample

Similarly table 3.1 also calculates average hardness of the coated samples of S1, S2, S3, S4.

SAMPLES F=10g Load	S1	S2	S3	S4
Average Hardness (HV)	1027.41	2080.57	2080.99	2254.42
Deposition time in min	60	60	60	60
Gas flow rate Ar:N_2	95:5	90:10	80:20	70:30
Deposition thickness	1.8 µm	1.5 µm	µm	µm

Table 3.2 Result of Sample S1, S2, S3, and S4

Nitriding parameter and their hardness of sample 1, 2, 3, and 4 aregiven in table 3.3 as shown below.Bare sample hardness before measure in table 3.1

SAMPLE	S1	S2	S3	S4
Average Hardness (HV)	605.49	719	960	924
Nitriding time in (hour)	3	2	3	3
Flow rate in H_2:N_2	80:20	80:20	80:20	80:20

Table 3.3 Hardness of Nitriding sample of SS304

3.2 RESULT ANALYSIS OF HARDNESS NITRIDING AND RMSP: According to these hardness of SS304 after coating and RMSP (Reactive Magnetron Sputtering Processes). When compare hardness then we are obtaining that reactive magnetron is much harder than nitriding of SS304 according to above table 3.2 and table3.3. We have obtained best result in RMSP coating so characterize only TiN Film Coating only.

4 Characterization of deposited TiN Thin Films:

Figure 4.1 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). This result is common for both bare samples because we are doing work on the same material SS304 nitriding and coating.

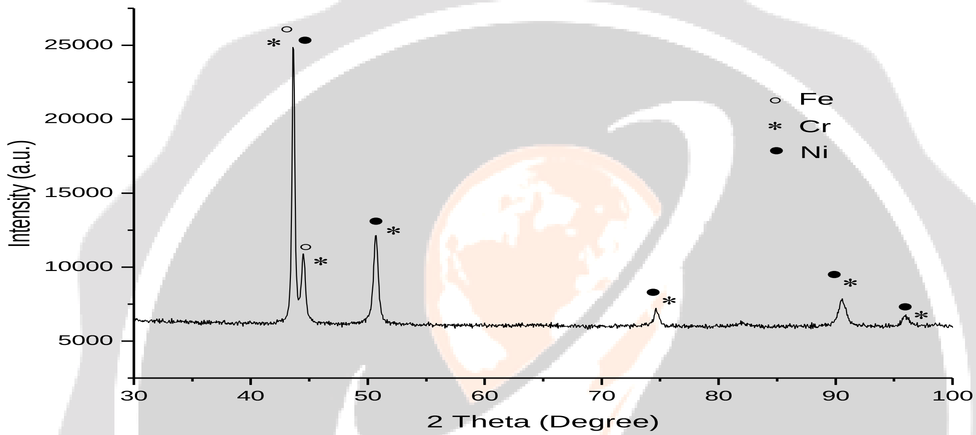


Fig. 4.1 XRD Plot for Bare SS 304 Sample

XRD Result of Sample S1, S2, S3 and S4 is given in figure 4.2

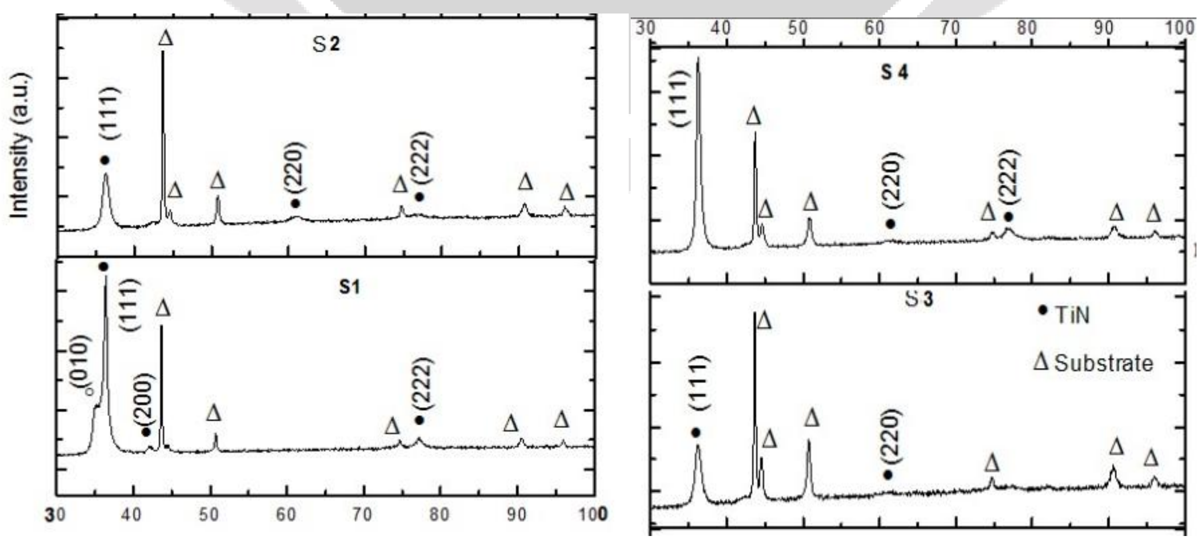


Fig 4.2 XRD Graph Of Sample S1, S2, S3 and S4 between angle 2 theta and intensity remaining parameter is given above.

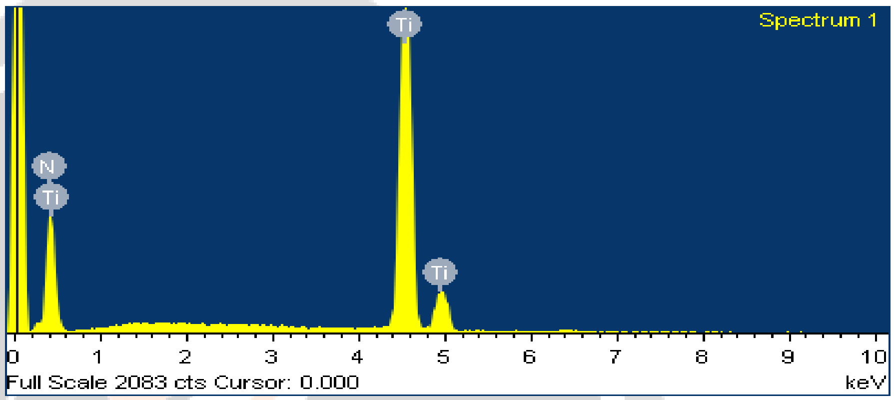
4.1 SEM-EDAX Results:

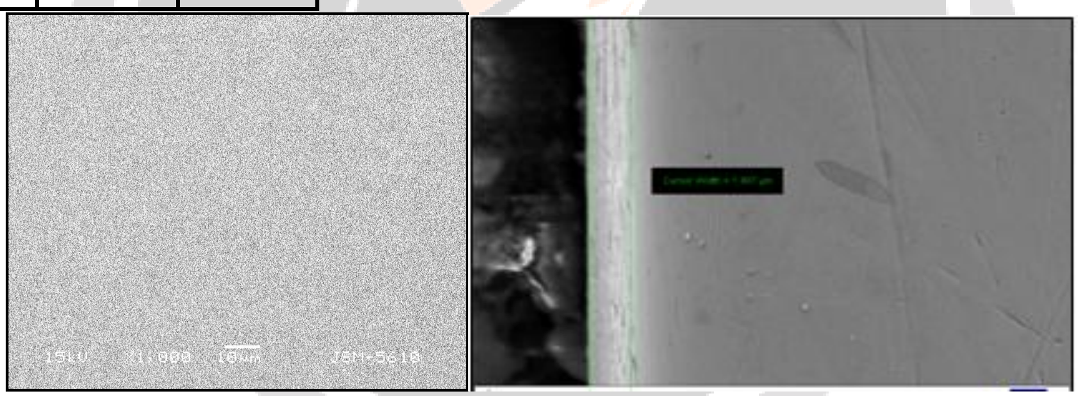
Sample S1: Ar:N_2 ratio 95:5 For sample S1, the film deposited was uniform and the SEM result shows no signs of cracks or voids in the deposited film. The microphotograph showing the surface features of the film at 1000 X magnification is shown in Fig. 4.1 (a) and Fig. 4.1 (b) shows the thickness of 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. 4.1(b)). Table 4.1 shows the chemical composition of the elements on the surface which clearly reveals the presence of Ti and N.

Table 4.1

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

Fig. 4.1: EDAX Graph for Sample S1





(a) **(b)**

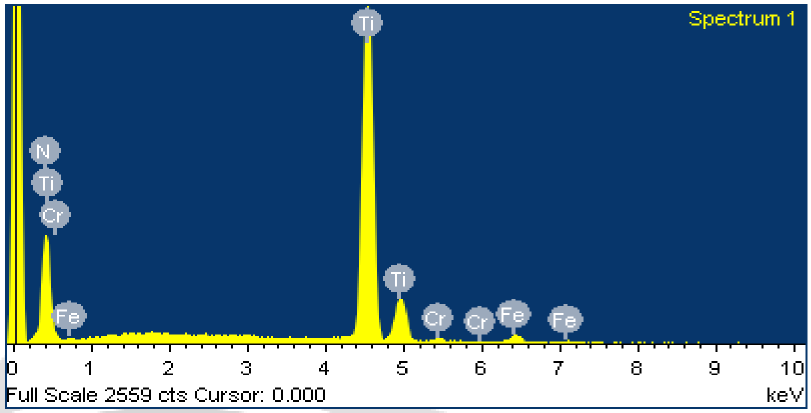
Fig. 4.2 (a) The top surface of the film (b) Cross section of sample S1 showing the thickness of the film

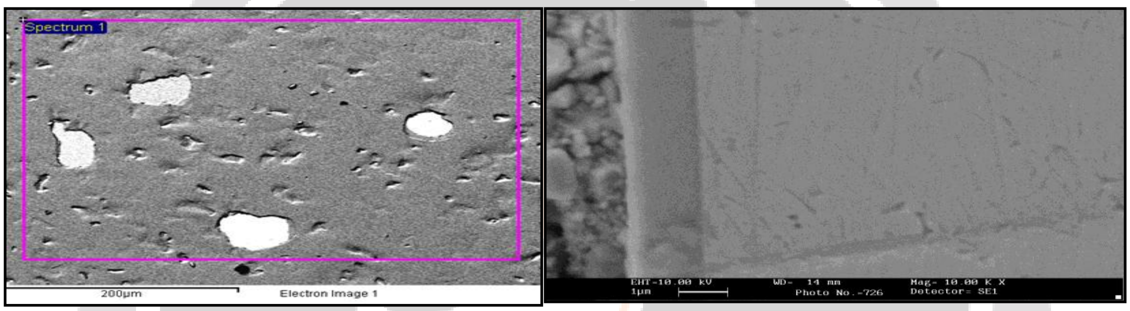
Sample S2: Ar:N_2 ratio 90:10 without Ar plasma sputter cleaning and 60 minutes deposition

Here, as shown in Fig. 4.4(a), the film was discontinuous and features like depressions and protuberances are seen. Furthermore, the substrate surface also shows voids. Figure 4.4(b) shows the cross-sectional SEM image and the thickness of the film was found to be 1.5 µm.

Table 4.2Fig. 4.3: EDAX Graph for Sample S2

Element	Weight%	Atomic %
N K	21.49	48.56
Ti K	73.20	48.38
Cr K	1.21	0.73
Fe K	4.11	2.33
Totals	100.00	





(a) **(b)**

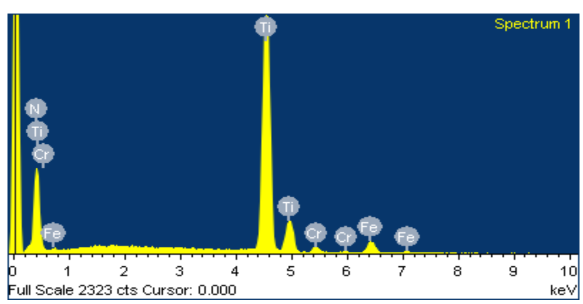
Fig. 4.4 (a) The top surface of the film (b) Cross section of sample S2 showing the thickness of the film

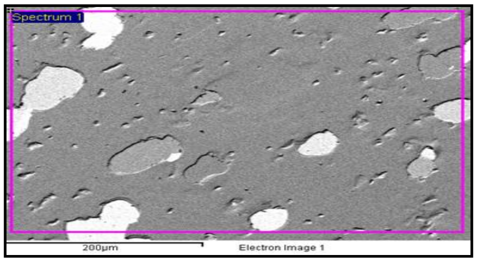
Sample S3: Ar:N_2 ratio 80:20, 60 minutes deposition

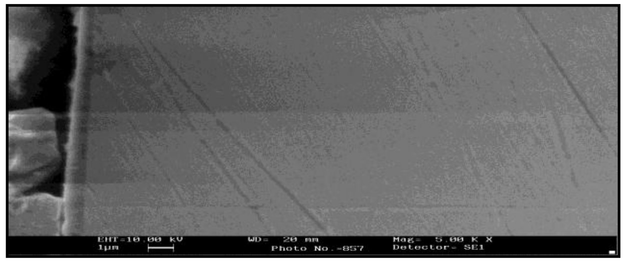
In case of sample S3, the film morphology is discontinuous and many cracks and voids are seen as in Fig. 4.6(a). Figure 4.6(b) shows the cross sectional SEM image and the thickness of the thin film was found to be 890 nm (0.89 µm). The EDAX graph and the Table 4.18 show the presence of the substrate elements because of the broken film.

Table 4.3Fig. 4.5: EDAX Graph for Sample S3

Element	Weight%	Atomic%
N K	23.36	51.43
Ti K	67.21	43.27
Cr K	2.11	1.25
Fe K	7.32	4.04
Totals	100.00	







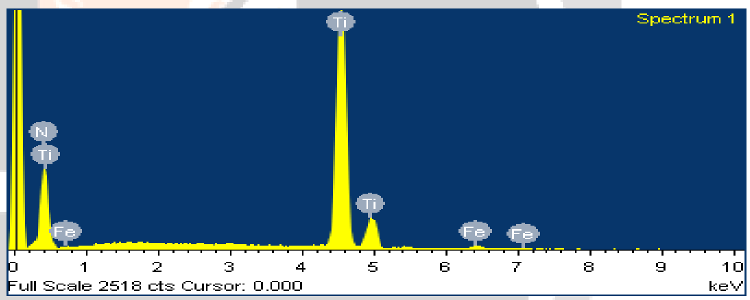
(a) **(b)**

Fig. 4.6 (a) The top surface of the film (b) Cross section of sample S3 showing the thickness of the film

Sample S4: Ar:N_2 ratio 70:30, 60 minutes deposition

The deposited TiN film on sample S4 in 70 % Ar and 30 % N_2 is discontinuous having voids in the structure. From the above SEM micrograph, it is revealed that many layers of TiN have formed and the white portions visible beneath the film show the substrate surface. **Table 4.4Fig. 4.7 EDAX graph for sample S4**

Element	Weight%	Atomic%
N K	22.59	50.04
Ti K	75.32	48.79
Fe K Total =100	2.10	1.17



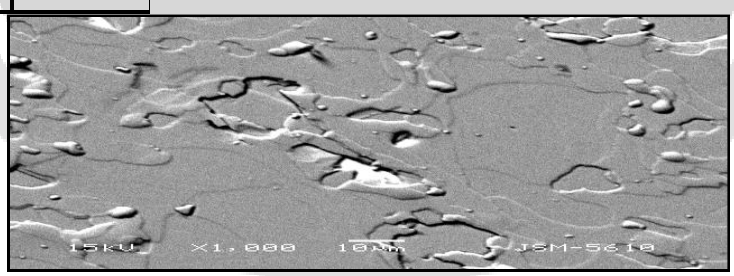


Fig. 4.8 SEM micrograph of top surface showing the deposited thin film for sample S4

5 Conclusions

- A new type of magnetron plasma source has been characterized in this work by depositing TiN thin films via DC reactive magnetron sputtering with different nitrogen concentration on SS 304 substrates.
- Reactive magnetron sputtering coating gives better hardness than nitriding.
- 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.

- 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 EDAX results indicate that at lower N_2 concentration, the atomic % of Ti and N are almost nearly 50% each, in agreement with the formation of stoichiometric TiN phase. However, at higher N_2 concentration, the nitrogen atomic % increases implying that the formation of other non-stoichiometric TiN_x phases may be possible which cannot be distinguished by XRD.
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