CHARACTERIZATION AND OPTIMIZATION OF CrN COATINGS ON TOOL STEELS (6959)

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Abstract: **Surface coating is one of the most useful and economical way to improve the surface properties of the materials. For this circumstance, Physical Vapor Deposition (PVD) is a unique method for coating of thin films. In this work coating is performed by magnetron sputtering deposition method. The properties of coating are varied with some of the process parameters like bias voltage, total gas pressure, volume of nitrogen gas, coating time, substrate temperature, substrate rotation and substrate roughness. Hence in this present work the characteristics of Chromium nitride (CrN) thin films on tool steel (grade: 6959) were investigated as function of Substrate temperature and Nitrogen gas flow rate. The CrN deposited films were categorized with X-ray diffraction (XRD) to disclose the development of different phases. The adhesive strength of the coating was studied by scratch tester and the microhardness of the thin film was determined by microhardness testing machine. Further, Optimization of the thin film coating is done by Response Surface Methodology (RSM).**

Keywords: **CrN thin films, Magnetron sputtering, PVD, Tool steel (grade 6959), XRD**

I. INTRODUCTION

Transition metal nitrides based coatings such as TiN and CrN are extensively used in coating industries due to its excellent strength, wear, and erosion resistance. CrN has been identified as a better substitute for TiN coatings for tribological applications due to its superior wear resistance and corrosion resistance. Among the various techniques available for the deposition of CrN thin films, reactive magnetron sputtering could be used effectively to tailor the features of the films as function of sputtering variables such as substrate temperature, gas pressure, power, target to substrate distance, reactive gas flow rate, deposition time and gas ratio. The main advantage associated with deposition of CrN thin film is its low deposition temperature.

Usually the durability of films intensely, depends on the grain size and the deposited film densities are prejudiced by collective effect of the temperature, working pressure and power of the sputtering process. At low working pressure and substrate temperature residual stress built up between coating and substrate interface which leads to poor adhesion of coating [1]. The CrN films deposited at low nitrogen flow rate showed that the electrical resistivity correspond to a metallic-like behavior and the films prepared at higher Nitrogen flow rate and above showed a semiconducting behavior. While increasing flow rate the percentage of pure CrN phase will decrease due to rapid solidification of highly energetic particles [2]. TiN, TiAl and CrN coatings can provide significant corrosion and erosion resistance to molten aluminium [3].CrN coating deposited by plasma activated physical vapor deposition on metal substrate by varying process parameters, the bias voltage is determined to have greatest influence on coating properties [4]. The adhesiveness of the coating on the substrate is dependent on substrate hardness [5].The highest stripping rate recorded for magnetron CrN coatings could not be explained by changes in coating structure/morphology and surface roughness. The variances in the stripping were renowned to the amount of CrN phase present in the coatings [6]. The literature review on influence in process parameters of the reactive magnetron sputtering technique on the characteristics of CrN thin films is very limited. It is very essential to substantiate the role of different sputtering condition for achieving the desired characteristics of thin films. Therefore the present work has been focused to study the features of CrN thin film as a function of substrate temperature, nitrogen gas flow rate, and optimizing the CrN thin film coating using RSM.

II. EXPERIMENTAL DETAILS

The 6959 tool steel substrate with a dimension of 50x25x4 mm was prepared for the deposition of CrN. The chemical composition of substrate used is shown in Table 1.

Table 1. The Chemical composition of substrate material

S.NO	Substrate	Carbon (%)	Manganese (%)	Silicon (%)	Chromium (%)	Nickel (%)	Molybdenum (%)
1	6959	0.35	0.75	0.3	1.5	3.5	0.65

Prior to the deposition the substrate must be cleaned. The specimens were cleaned by the ultrasonic effect for removing oil and other contaminations and liquid honing is performed using fine adhesive powder for obtaining better surface roughness. The coating was done in Planer RF magnetron Sputtering machine and the deposition parameters are shown in Table 2. Before coating the machine was pumped to $3X10^{-6}$ Torr. The environment in sputtering chamber was a mixture of Ar+N2gases. The plasma was generated by introducing Argon gas, after which the Nitrogen gas was introduced for reaction. The coating system has a cathode Chromium. A Cr (99.99%purity) target of 4inch diameter was used during the sputter deposition. Usually, the substrate was rotated at constant speed to face the target.

Table 2. The Operating conditions

S.NO	DEPOSITION PARAMETERS	VALUES
1	Pulsating parameter	D.C
2	Working pressure	3X10^-6 Torr
3	Substrate Temperature	200°C,300°C,400°C
4	Target to substrate distance	60 mm
5	Deposition Environment	$Ar+N_2$
6	Deposition time	40 min
7	Target power	100 W
8	Nitrogen flow rate	5 sccm,10sccm, 15sccm

For optimization the RSM is used. Response surface methodology or RSM is an assembly of mathematical and statistical practices that are useful for modeling and analysis of problems in which reaction of interest is prejudiced by numerous variables and the objective is to optimize the response. Response surface method consists of several methods. In this paper we have discussed 2 factor 3 level design method which is shown in Table 3. Here the substrates to target distance, power, deposition time and pressure were held constant during deposition. While substrate temperature, (reactive) Nitrogen gas flow rate were set to different values. For this method the region of exploration for the fitting of first order model should be $(200,300,400)^0$C and (5, 10, 15) sccm. After the coating process with varying parameters, the specimen is tested for its characterisation. The structure of the deposited films was studied by X-ray diffraction, hardness was measured by micro hardness tester and the adhesive strength of the film was detected by scratch tester.

Table 3. The 2 Factors and 3 Levels

CONTROL FACTORS	LEVELS		
	1	2	3
1.substrate temperature (^oC)	200	300	400
2.N_2-flow rate (sccm)	5	10	15

III. RESULTS AND DISCUSSION

Fig.1, 2 & 3 shows the XRD patterns at different N_2 flow rates, which indicates that the crystallography structure of sputter-deposited CrN thin films depends on the nitrogen content in sputtering process. For the thin films deposited at 5 sccm mixed phases of CrN, Cr_2N and Cr_2O_3 is observed. At 10 sccm the film contains mainly CrN with low intensity of Cr_2O_3 as reported in the literature (B.Subramanian et al). No purely single phase CrN films were obtained when the flow rate is increased. This is due to unbalanced growing conditions of magnetron sputtering, which were due to rapid solidification of highly energetic particles (Zhang et al). The mean free paths of gas particles are shorter and they decrease with increase in nitrogen flow rate. The decrease of mean free path also means less energy and momentum delivery on substrate by ion impingement. This leads to lesser extent of surface damage and fewer nucleation sites, which may result in larger grain size. The intensity peaks in the graph occurs in between 37 to 40, 2Ɵ angle ensures that CrN coatings exist. It is clearly seen that the purity of CrN phase is disturbed by higher nitrogen flow rate.

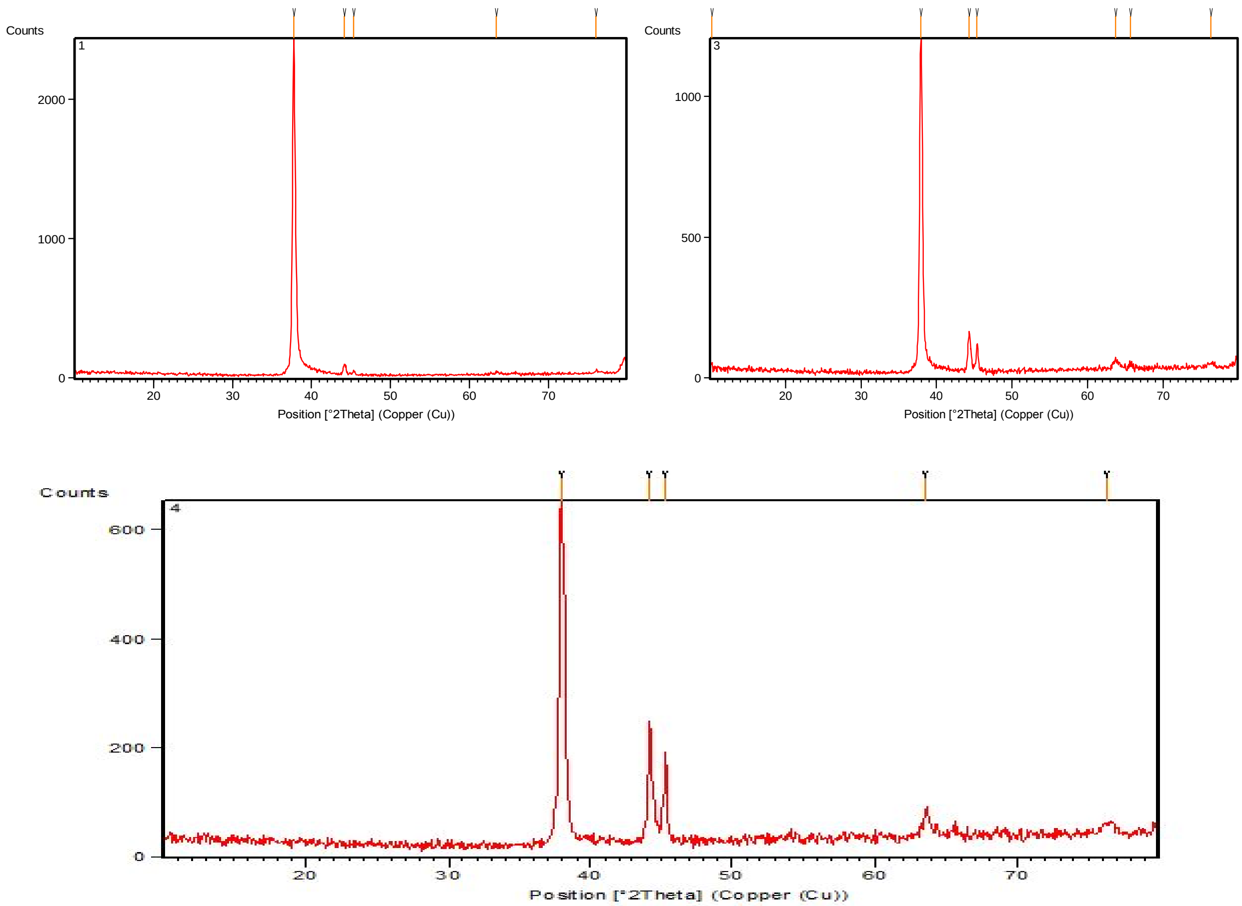

Figure 1. (a),(b),(c) shows the XRD patterns at different N_2 flow rates

Measurement of Microhardness and Adhesive Strength:

The microhardness and adhesive tests were carried out and the results are shown in Table 4. Microhardness of the thin films were measured by using Mitutoya HM113 machine. Vickers hardness is preferred to measure the thin film micro hardness. The indenter made up of diamond is used. Adhesive strength of the thin films was measured using scratch tester (make-Ducom). Here diamond indenter of 200µm is used. The initial load of 12N is given first and it is made to increase 2N for every 1mm Pile. During the testing, there is an occurrence of notable change in acoustic emission curve and corresponding to that change the change in traction force curve is noted. From that the load that completely scratched the thin film was calculated.

Table 4. shows the tested values

SI.NO	NATURAL VARIABLES		CODED VARIABLES		ADHESIVE STRENGTH (N)	MICROHARDNESS (HV)
1	200	5	-1	-1	26	409.6
2	300	5	0	-1	26.7	422.3
3	400	5	1	-1	26.8	456
4	200	10	-1	0	26.4	398.1
5	300	10	0	0	27.2	535
6	400	10	1	0	28.4	798
7	200	15	-1	1	24.8	464.4
8	300	15	0	1	26.4	498.8
9	400	15	1	1	27.8	629.2
10	300	10	0	0	27	665.6
11	300	10	0	0	27.6	677.5
12	300	10	0	0	27.2	637.5
13	300	10	0	0	27	653.9

Figure 2a & 2b shows the Contour plot of microhardness strength VS flow rate, temperature and Contour plot of adhesive strength VS flow rate, temperature. From the RSM result, the optimum value are shown in Table 5 in which we come to know that the adhesive strength and microhardness of the thin film is maximum when the substrate temperature is high and the nitrogen flow rate is medium.

Table 5. Shows the Optimized values

Temperature	Flow rate	Microhardness HV	Adhesive strength N
400°C	11.58 sccm	736.404	28.312

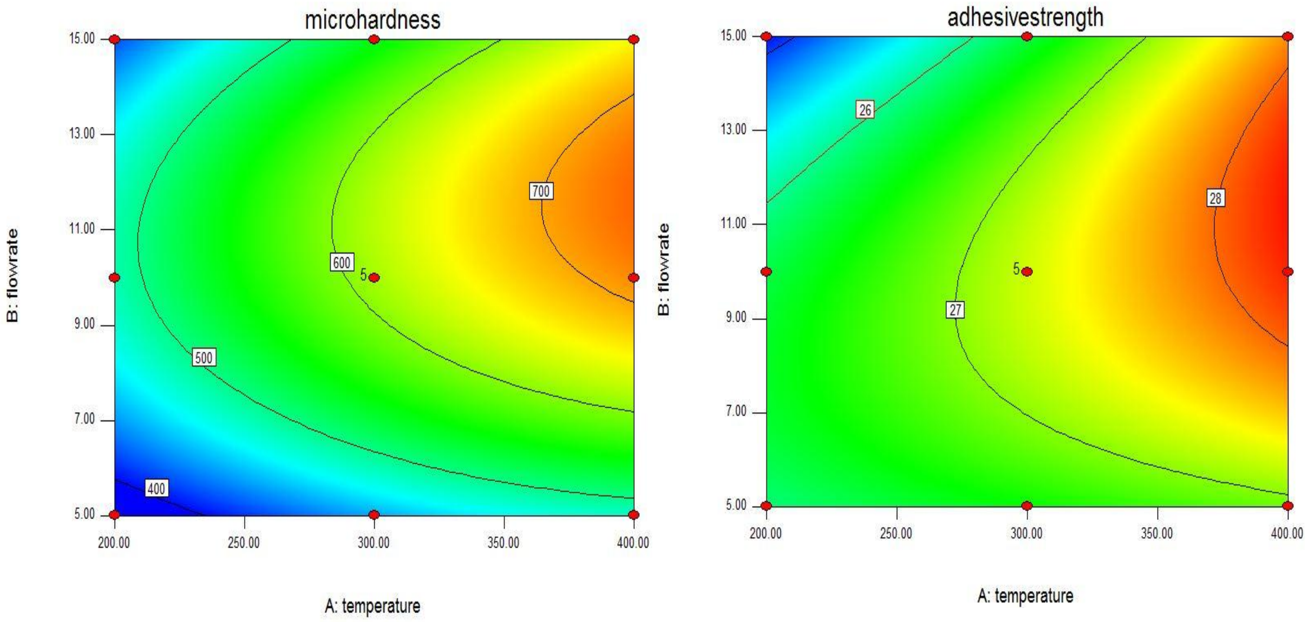

Fig 2 (a) & 2(b) shows the contour plot of micro hardness, adhesive strength VS flow rate, temperature

IV. CONCLUSION

The CrN thin films deposited on 6959 grade steel by magnetron sputtering process has been investigated in present work. The effect of substrate temperature and nitrogen gas flow rate on adhesive strength and microhardness were analyzed. From the results it is found that the micro hardness and adhesive strength of the thin film shows increasing trend with increase in substrate temperature and decrease in nitrogen flow rate. The higher adhesive strength is due to the grain size effect and highly textured grains.

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