



## **Wear Resistant Nanostructured Coating for Cultivator Shovels**

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### **Authors' contributions**

*This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.*

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## **ABSTRACT**

In Indian economy agriculture plays a principal source of income. Primary and secondary operations such as ploughing are the basic actions and extensive usage of cultivators for secondary tillage operation causes wear of the shovels because they are subjected to high tillage forces. An ample amount of material is lost due to wear and abrasion. Therefore, control and prevention of wear of cultivator shovels are vitally important for agro applications because wear of critical components has an economic significance. In this work CrN coating is deposited over cultivator reversible shovels using the DC magnetron sputtering technique and the structure, morphological, hydrophobic and wear characteristics are determined. It is observed that the working life of the agricultural tool is increased up to a significant level by depositing the desired coating on the substrate.

*Keywords: Wear; DC magnetron sputtering; tillage; secondary equipment.*

## **1. INTRODUCTION**

Agriculture is the backbone of the Indian economy, with agriculture providing primary

income to almost half of the Indian population. In India, farm mechanisation is regarded as one of the most significant aspects of agricultural modernization. From 0.636 t/ha in 1965-1966 to

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2.111 t/ha in 2013-2014, the country experienced extraordinary rise in food grain productivity. The usage of agricultural technology on farms is largely to blame for this expansion. In India, average farm power availability rose from 0.30 kW/ha in 1960-1961 to 2.02 kW/ha in 2013-2014 [1].

Ploughing is one of the most basic primary and secondary processes required for the cultivation of any crop. Farmers employ a variety of implements, such as mouldboard ploughs, disc ploughs, and cultivators, under a variety of field situations. Cultivator is a type of soil engagement tool that, because of its broad soil coverage area between two furrows, is employed as a secondary tillage implement. Because shovels are subjected to strong tillage forces when cultivators are used for secondary tillage, they wear out quickly [2-8]. According to an estimate of material loss in grain farming in Turkey, 9700 t of steel is lost owing to wear and abrasion and the energy equivalent of cultivating an area of 13422000 ha twice.

Surface modification techniques such as coating can help to reduce the wear and abrasion of agricultural tools which can increase the life shell of agricultural tools. It is highly desirable to deposit hard protective coatings to increase the cost effectiveness, life and performance of the shovel. These procedures offer strong process stability, are cost-effective, and have been adapted for industrial production, however they are not environmentally friendly, and the coatings they produce have poor characteristics. The deposition of ions and molecules of a material is vaporised and transported through a vacuum or low pressure gaseous environment before condensing on a substrate in the physical vapour deposition (PVD) process. PVD methods may deposit films with thicknesses ranging from a few angstroms to a few dozen nanometres [9-13]. Vacuum evaporation, magnetron sputtering, sputter deposition, arc evaporation, low voltage electron beam evaporation, cathode arc deposition, triode high voltage electron beam evaporation, and ion planting are some of the most extensively used PVD processes using sputtering and evaporation technologies. But nanostructured thin films have attracted considerable attention in several applications like optoelectronics, electronics [14-17].

Magnetron sputtering is a physical vapour deposition (PVD) coating technique that can overcome many disadvantages associated with

other techniques, such as low density, poor adhesion, and poor properties. Sputtering is a very flexible and effective deposition process for depositing various metallic and compound thin films for surface engineering applications. It holds many advantages like pure deposition, reproducibility, uniform deposition rate and good quality of film.

Hard protective coatings deposited by sputtering shows excellent physical properties such as high hardness, strength, toughness, chemical stability, wear and corrosion resistance [18-19]. It has been widely used as a thin protective film [20]. The sputter deposition technique can deposit coatings at low substrate temperatures or control the structure and properties of the deposited coatings by adjusting the deposition parameters [21].

Physical vapour deposition of chromium nitride in comparison to other nitride coatings through magnetron sputtering technique was a good alternative to other conventional surface treatments for protection against wear. CrN has better hardness, toughness and good tribological properties. Hard coatings are characterised by a relatively low friction coefficient, good wear resistance and a high corrosion resistance. The aim of this research was to deposit nanostructured coating material to enhance the life shell of the cultivator shovels [22-24].

In this study, we have deposited CrN over agriculture tool named reversible shovel on using the DC magnetron sputtering technique and investigated structure, morphological, hydrophobic and wear characteristics at different substrate temperatures as (100,150,200 °C). The coatings were characterized by X-Ray diffraction (XRD), Scanning electron microscopy (SEM) nanoroughness and hydrophobicity were determined using atomic force microscopy and contact angle goniometer. Wear test were carried out in a custom designed circular soil bin.

## 2. EXPERIMENTAL DETAILS

### 2.1 CrN film Processing

The CrN films were deposited by DC-magnetron sputtering technique on shovel (mild steel) as substrate in a custom designed 35 inch diameter chamber (Milman thin film systems). A 4 inch diameter and 6 mm thick chromium target of 99.99% purity was used for the sputtering. The substrate was thoroughly cleaned by rinsing in

acetone and methanol and dried under nitrogen gas. The chamber was initially evacuated by a turbo molecular pump backed by a rotary pump. During each deposition, the base pressure was kept less than  $5 \times 10^{-6}$  Torr and the distance between the substrate and the target was kept 110 mm. The CrN coating was fabricated at As-deposited and 200 °C substrate temperature. The sputtering was carried out in an argon (99.99% pure) atmosphere while nitrogen was used as a reactive gas. The flow rate of nitrogen and argon in the chamber was 20 and 20 sccm, respectively. The gas pressure was kept 10 m Torr for all depositions.

## 2.2. Characterization Details

The structure properties of -CrN films were characterized by X-ray diffractometer (XRD) with Cu K $\alpha$  radiation. Scan rate used was 2° min<sup>-1</sup> and the scan range was from 10 to 70°. The surface topography and elemental analysis of the film was studied using Scanning electron microscope (SEM) (Carl Zeiss) was carried out by using energy dispersive X-ray analysis (EDS). The surface morphology of the CrN films was studied using atomic force microscope (NT-MDT, Ntegra) operated in semi contact (tapping) mode. To determine whether the films are hydrophilic or hydrophobic by nature, contact angle measurement system (Kruss DSA 100 Easy Drop) was used to find contact angle of water with the films. Wear analysis was carried out in custom designed soil bin. A circular soil bin had an outer diameter of 5520 mm, inner diameter of 3490 mm and a depth of 900 mm.

## 3. RESULTS AND DISCUSSION

### 3.1 Structural analysis

X-ray diffraction analysis was carried out to determine the orientations and structural transformation of CrN films deposited at three different temperatures. Fig.1a and Fig.1b shows the XRD of the nanostructured films of chromium nitride deposited at 100, 150 and 200 °C and JCPDS 11-0065 details of the CrN. The XRD curve clearly reveals that CrN film deposited at different temperatures are polycrystalline in nature and dominant peak occurs at  $2\theta = 37.56^\circ$  which corresponds to (1,1,1) orientation of cubic phase of CrN (JCPDS 11-0065). At 200 °C temperature the deposited film exhibited an intense and sharper diffraction pattern. The average crystallite size of the thin film was calculated using the Debye–Scherrer formula [25].

$$t = \frac{0.9\lambda}{\beta \cos\theta} \dots\dots\dots (1)$$

Where t is the crystallite size,  $\lambda$  the wavelength of X-ray (1.54056Å ),  $\beta$  the line width at half maximum of the most dominant peak and  $\theta$  Bragg diffraction angle. The crystallite size for 100, 150 , 200 °C substrate temperature coating were 18.97, 20.44 and 25.47 nm, respectively. Crystal size was found to increase with the rise in substrate temperature. The enhancement in the crystal size of chromium nitride films can be associated with the change in the kinetic energy of the sputtered particles with the temperature.

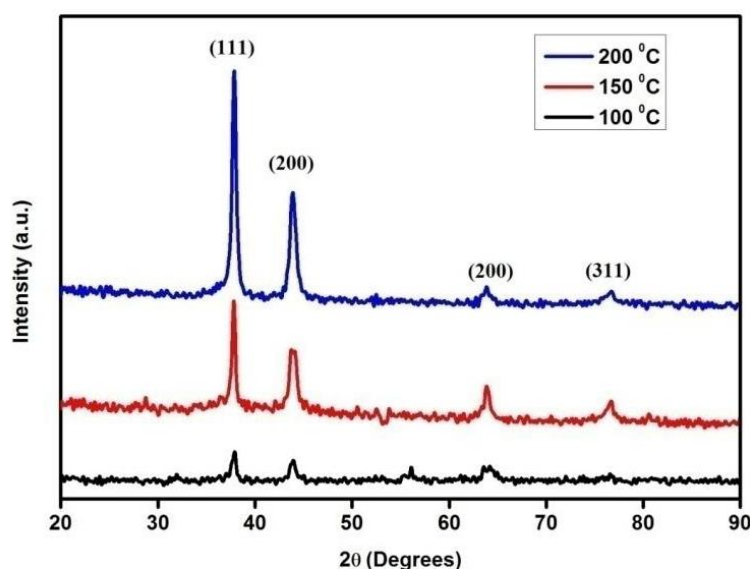


Fig. 1a. XRD patterns of CrN films deposited at different temperatures [26]

11-0065		Wavelength= 1.5418			
CrN	2 $\theta$	Int	h	k	l
Chromium Nitride	37.569	80	1	1	1
	43.774	100	2	0	0
	63.597	80	2	2	0
Carlsbergite, syn	76.226	60	3	1	1
	80.186	30	2	2	2
Rad.: CuK $\alpha$	$\lambda$ : 1.5418	Filter: Ni	Beta	d-sp: 9.0	
Cut off:	Int.: Estimation	I/lor.:			
		108.547	50	3	3
		112.714	60	4	2
Ref: Turkdogan, Ignatowicz, J. Iron Steel Inst., London, 188, 242 (1958)		131.352	60	4	2
		150.104	30		
Sys.: Cubic	S.G.: Fm $\bar{3}m$ (225)				
a: 4.140	b:	c:	A:	C:	
$\alpha$ :	$\beta$ :	$\gamma$ :	Z: 4	mp:	
Ref: Ibid.					
Dx: 6.178	Dm: 5.900	SS/FOM: F <sub>g</sub> = 11(0.092 . 9)			
Color: Light gray, pink-violet in reflected light					
Electron diffraction: a=4.139. Reference: Pinsker, Ambrosimova, Kristallografiya, 281, Sov. Phys. Crystallogr., 3 285 (1958). Cl Na type. Halite Group, nitride Subgroup. PSC: cF8. See ICSD 37412 (PDF 76-2494). Mwt: 66.00. Volume[CD]: 70.96.					


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Fig. 1b.JCPS 11-0065 details of the CrN (Chromium Nitride)

### 3.2 Surface Morphology and Chemical Composition

The surface topography and chemical composition analysis was done through EDS and SEM as shown in Table 1. It shows the atomic (%) of chromium and nitrogen of deposited film at different substrate temperatures. It was clear from that the elements of Cr and N that they were dependent upon temperature. SEM images of film deposited at substrate temperatures are shown in Fig. 2. The Cr/N ratio was approximately 1 for film deposited at temperature 200°C while that of As-deposited was less than one. The surface-nano roughness of the CrN films was studied using atomic force microscopy (AFM). The AFM micrographs correspond to a scan area of 2 $\mu\text{m}$ ×2 $\mu\text{m}$  and two dimensional AFM images obtained for CrN films deposited at 100, 150, 200 °C are shown in Fig. 3. It was clear from the Fig. 3 that CrN films are constituted with a large number of nanograins. The surface roughness was calculated using the software attached with AFM and as shown in Table 1. It was clear that as substrate temperature was increased from 100 to 150 and 150 to 200 °C roughness value was also increased from 6.35, 8.48, 11.17 nm respectively. This behaviour of roughness was very well correlated to the particle size which was earlier calculated through XRD. The increment in substrate temperature increases the particle size which in turn

enhances the roughness, while decrement in particle size reduces the roughness.

### 3.3. Hydrophobic Properties

The wettability of CrN coatings were evaluated by measuring the water contact angle goniometer at ambient room temperature. The distilled water droplet were dropped on the deposited coating surfaces using a micro syringe. Average value of the water contact angle was determined by experimental drop profiles at different positions for the same sample. The water contact angle as a function of sputtering temperature was measured and shown in Fig.4. A graph in Fig.4 shows relation between roughness, contact angle and substrate temperature. Water contact angle as measured of 100, 150 and 200 °C was 100, 102 and 105° respectively which confirms that CrN coating was hydrophobic in nature.

### 3.4 Effect of Coating on Wear of Cultivator Shovels by Abrasive Sand

The effect of coating on gravimetric wear of reversible cultivator shovels was observed in a circular soil bin by using sand as abrasive medium. Test shovels S<sub>1</sub>, S<sub>2</sub>, S<sub>3</sub> with substrate temperature 100, 150 and 200°C, respectively had almost equal hardness of 371 BHN and that were mounted on the tool frame for continuous

testing in the soil bin. The moisture content of sand was kept 9-10%. Operating speed and

depth of operation were 1m/s and, 100 mm, respectively.

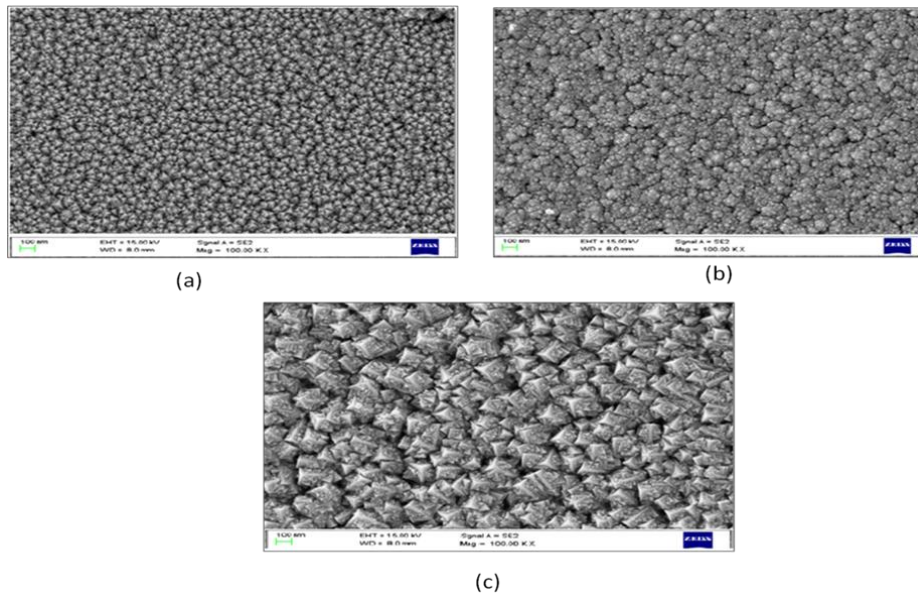


Fig. 2. SEM images of film deposited at different substrate temperatures, (a) 100°C (b) 150°C (c) 200°C

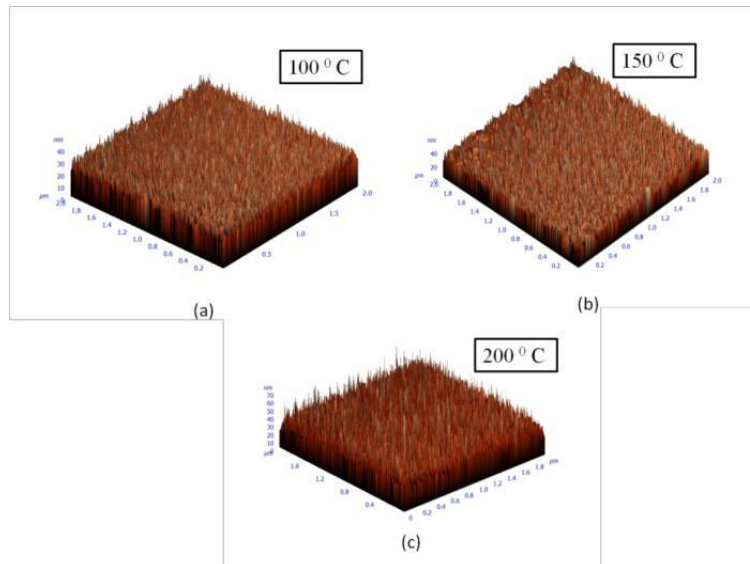


Fig. 3. AFM micrographs of film deposited at different temperatures

Table 1. EDS data of deposited CrN samples at different substrate temperatures

Substrate temperature ( <sup>0</sup> C)	Atomic % Cr	Atomic % N <sub>2</sub>
100	39.56	46.54
150	40.77	44.31
200	42.06	42.48

### 3.4.1 Effect of working period

The results obtained during the study are depicted in Fig.5. It is clear from the figure that the weight of shovels decreased with increase in working period for all tested shovels. Shovels with different substrate temperatures coating deposited after 100 h are shown in Fig.6. This result is with the findings of [27, 28] who also reported that wear increases with increase in period of work. Wear rate was gradually increasing with time. The higher average wear rate of 2.41 mg/min was observed during 100 h of operation in non-coated shovel, and the same was reduced for 100 °C, 150 °C and 200 °C that was 2.02 mg/min, 1.77 mg/min and 1.36 mg/min respectively. This may be due to the reason that as substrate temperature increases adhesiveness of the coating increases. The results are in agreement with (Lufitha,2001) who also reported that adhesion strength of the coating increased when substrate temperature was raised.

A statistical analysis was also carried out to find the significant effect of substrate temperature based coating and time on wear of shovels (Table 2). ANOVA shows that the main effects of

time and temperature were highly significant on wear loss. The analysis further reveals that the interaction S (Shovel) x t (Time) was also significant. Table 3 showing mean values interaction effect of substrate temperature based coating and time on gravimetric wear.

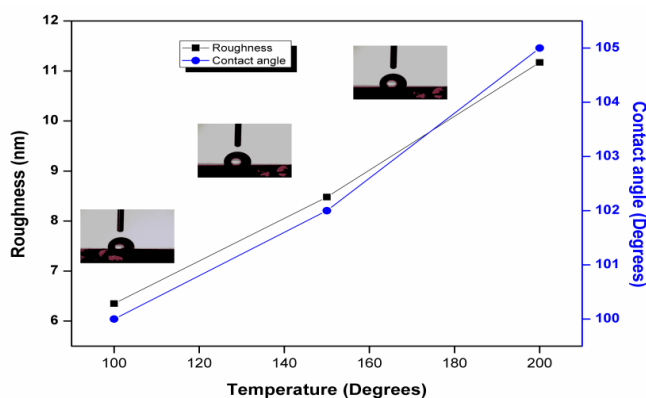
### 3.4.2 Effect of substrate temperature based coating on wear of shovels.

The cumulative wear observed was 14.48, 12.13, 10.59 and 8.15 gm for shovel with non coated and substrate temperature coating at 100 °C, 150 °C and 200 °C respectively after 100 hrs of working period. It was found that the wear loss decreased with increase in substrate temperature based coating. Minimum weight loss due to wear was observed in shovel with temperature 200 °C. Thus, with the increase in temperature from 100 °C to 200 °C the cumulative wear was decreased by 43.71 per cent. Further wear rate was calculated for the tested shovels (Table 4.). The wear rate was minimum i.e., 1.36 mg/min for shovel with 200 °C substrate temperature and for shovel 100°C it was 2.02 mg/min. These results are in agreement with the findings of [29-31]. Fig.7 shows graph between wear rate and time.

**Table 2. Analysis of Variance for the effect of substrate temperature based coating and time on wear of cultivator shovels**

Source of Variance	DF	SS	MSS	Computed F
Shovel (S)	3	63.42	21.14136	1170.688*
Time (t)	5	1145.58	229.1169	12687.18*
Sxt	15	47.31	3.154078	174.6548*
Error	48	0.87	0.018059	1.000
Total	71			
Grand Mean		11.33		
C V		5.51		

\* Significant at 5 % level



**Fig. 4. Contact angle and roughness of film deposited at different temperatures**

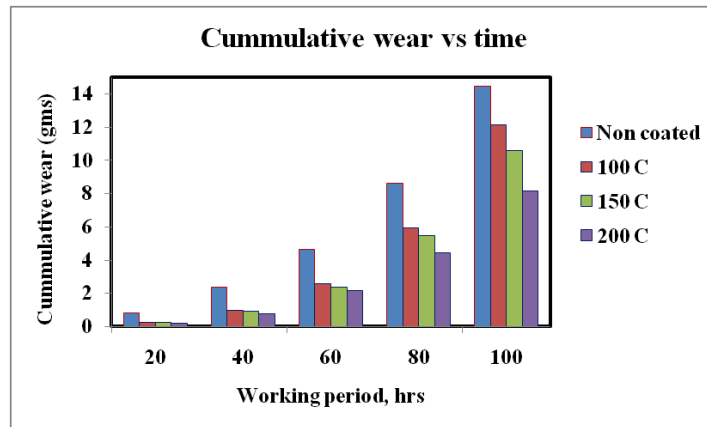


Fig. 5. Cumulative wear in shovels with different substrate temperature coating at different working period

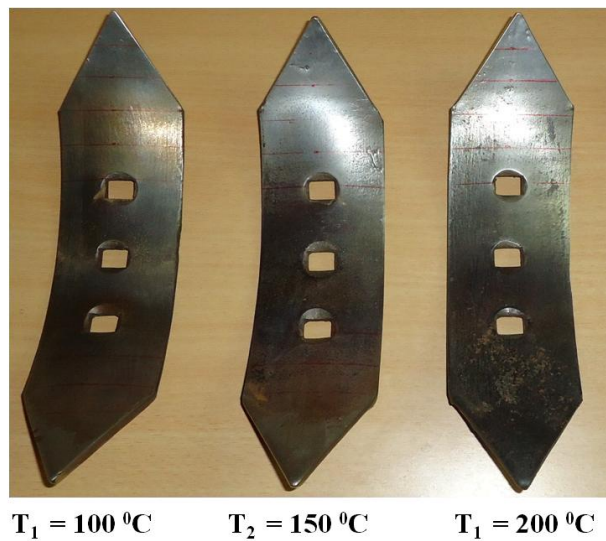


Fig. 6. Shovel with different substrate temperature coating after 100 hrs of working period

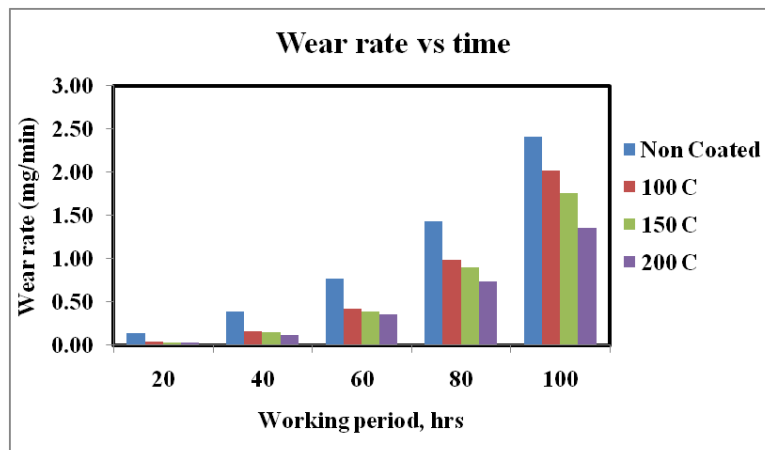


Fig. 7. Wear rate in shovels with different substrate temperature coating at different working period

**Table 3. Mean values showing interaction effect (two variables) of substrate temperature based coating and time on gravimetric wear.**

Time/Shovel	S <sub>1</sub> (Non coated)	S <sub>2</sub> (100°C)	S <sub>3</sub> (150°C)	S <sub>4</sub> (200°C)	Mean
0	0.00	0.00	0.00	0.00	0.000
20	0.82	0.25	0.22	0.17	0.367
40	2.38	0.95	0.89	0.75	1.243
60	4.65	2.55	2.36	2.14	2.928
80	8.60	5.92	5.44	4.41	6.090
100	14.48	12.13	10.59	8.15	11.338
Total	30.94	21.80	19.50	15.62	
<b>Mean</b>	<b>5.16</b>	<b>3.63</b>	<b>3.25</b>	<b>2.60</b>	

**Table 4. Wear rate of shovels with different substrate temperature based coating**

Sr.No.	Shovel (Temperature)	Wear rate (mg/min)
1	S <sub>1</sub> (Non Coated)	2.41 mg/min
2	S <sub>2</sub> (100 °C)	2.02 mg/min
3	S <sub>3</sub> (150°C)	1.77 mg/min
4	S <sub>4</sub> (200°C)	1.36 mg/min

## 5. CONCLUSIONS

The diffraction patterns confirms that the coating deposited on the shovel was chromium nitride (CrN) and exhibits cubic structure. Crystal size was found to increase with increase in substrate temperature. Cr/N ratio was approximately one for film deposited at temperature 200°C, while for other deposited temperatures at 100°C and 150°C and it was less than one. Roughness value was found to increase with temperature. Water contact angle were observed 100°, 102° and 105° at 100 °C, 150 °C and 200 °C substrate temperature respectively, which confirms that CrN coating was hydrophobic in nature. The effect of temperature was found significant and the wear loss decreased with increase in substrate temperature coating. Minimum weight loss due to wear was observed in shovel with maximum substrate coating temperature at (200 °C). The shovels with different substrate coating temperatures wore along the thickness. There was negligible change in length and width. The reduction in thickness in all shovels followed the same pattern. Wear loss was maximum at the tip of the shovel and it gradually decreased while moving away from the tip of the shovel. Minimum dimensional wear loss was observed in shovel with substrate coating temperature at (200 °C). On the basis of the above findings, it was concluded that the DC magnetron sputtering coating technique can be used to deposit antiwear, corrosion free and hydrophobic coating at 200 °C substrate temperature on agricultural tools. The working life of the agricultural tool can

be increased upto a significant level by depositing the desired coating on the substrate.

## DISCLAIMER

The products used for this research are commonly and predominantly use products in our area of research and country. There is absolutely no conflict of interest between the authors and producers of the products because we do not intend to use these products as an avenue for any litigation but for the advancement of knowledge. Also, the research was not funded by the producing company rather it was funded by personal efforts of the authors.

## COMPETING INTERESTS

Authors have declared that no competing interests exist.

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