

# EFFECT OF HEAT TREATMENT OVER THE ELECTROCHEMICAL, MECHANICAL AND TRIBOLOGICAL PROPERTIES OF THE CR-DLC COATING

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Diamond Like Carbon (DLC) with both sp<sup>3</sup>- and sp<sup>2</sup>-bonded carbon in DLC coatings is equipped with superior mechanical, tribological, electrical, and optical qualities. Depending on their composition and synthesis method, DLC films can be amorphous, hard, or strong. High residual stress in the film, however, limits its potential uses in many fields and results in poor adherence to the substrate materials. Increasing the deposition temperature, post-synthesis annealing, and vacuum furnace heat treatment are some popular techniques to lower residual stress. In this work, a DC magnetron sputtering method was used to create Cr-DLC thin films on silicon (100) substrates. Atomic force microscopy (AFM) and electrochemical testing, and nanoindentation test were used to assess the mechanical attributes of the thin film prior to production. According to the corrosion test, the annealing temperature increased corrosion resistance. The coating's young's modulus (E) and nanoindentation hardness (H) were computed. The internal stress of the produced coating was determined using Stoney's equation. The Cr-DLC coating was heat treated in a vacuum furnace at temperatures ranging from 270 to 360°C. Following heat treatment, nanoindentation was used to characterize the coating once more in order to evaluate its mechanical properties, such as H and E. The findings demonstrated that raising the heat treatment temperature to 360°C considerably reduced the coatings' residual stress. In contrast to the coatings' H and E decreasing, the residual stress of the coating heat treated at 300°C was somewhat reduced. From the electrochemical analysis, it was clearly understood that by increasing the temperature the corrosion resistance (CR) of Cr-DLC

coatings' is increased up to 330°C. However, with the rise in temperature to 360°C the CR started decreasing.

## KEYWORDS

DLC, Cr/DLC, Sputtering, residual stress

## 1 INTRODUCTION

These days, a unified technology called diamond-like carbon (DLC) coatings is used on a different substrates, including metals and ceramics [Sui 2018; Cui 2019]. Due to the advantageous combination of characteristics, DLC coatings have been used on a variety of items, including tools [Santiago 2020], blades [Jelinek 2015], bearings [Guo 2015] optics [Wang 2023], and automotive components [Khodayari 2023]. This includes the capability to resist abrasive and adhesive wear, high hardness, high density, low friction coefficient, high thermal conductivity, optical transparency, and chemical inertness. DLC is an extremely amorphous and stable form of carbon that exhibits a special hybridization of sp<sup>2</sup> and sp<sup>3</sup> bonds [Shahsavari 2020]. The high residual compressive stresses of pure DLC coatings are one drawback. This is because the hybridization of sp<sup>2</sup> and sp<sup>3</sup> bonds causes a divergence in the bond angle. Because of their broad variety of applications, these coatings are both highly significant and fascinating from a scientific standpoint for many industrial applications with remarkable mechanical, chemical, tribological, and physical qualities [Shahsavari 2020]. Their outstanding chemical inertness protects them from oxidative and corrosive attacks in saline and acidic conditions. With variable degrees of diamond-like characteristics, DLC is amorphous and heavily reliant on the ratio of diamond sp<sup>3</sup> to graphitic sp<sup>2</sup> C-C bonds. But because of its high internal stress, the DLC coating has a drawback: it adheres poorly to metal and metal alloy substrates. This can be fixed by utilizing interlayers and dopants [Dai 2016]. The deposition of various metal doped These transition metals alter the structure of DLC coatings and contribute to bettering their mechanical and tribological qualities since their d orbitals prefer to overlap with carbon's p orbitals [Lakhonchai 2022; Zhu 2020]. DLC coatings has been tried recently to address the high residual stress and poor adherence of pure DLC coating. When metal atoms are joined with carbon atoms or integrated into the DLC matrix, a nanocluster may be created. It has been noted that metals that create carbides, such as W, Ti and Cr, and carbide free constitutes, such as Pt, Cu and Ag form metal doped DLC coatings [Xiao 2016; Shao 2020]. Because of its high adhesion and low stress, the Cr-doped DLC coating among these dopant metals shows outstanding tribological capabilities when compared to the undoped DLC coating. Numerous groups have conducted research on chromium-doped and chromium- interlayered DLC coatings, examining their electrical, mechanical, tribological, magnetic, and thermal characteristics [Shahsavari 2020]. Although a diamond is completely crystalline, DLC coatings are usually created using low-temperature plasma for chemical vapor deposition (CVD) or physical vapor deposition (PVD). Depending on the raw materials used for the deposition (such as graphite or hydrocarbons), the chemical composition of the coating may contain hydrogen. Ion beam deposition, pulsed laser deposition, plasma-enhanced CVD (PECVD), Magnetron sputtering [Duminica 2018; Muthuraja 2019], plasma-assisted chemical vapor deposition (PACVD) [Khodayari 2023], and cathodic vacuum arc deposition are some of the deposition techniques used to create DLC carbons. In this study, we report on the synthesis of Cr-doped DLC coating using magnetron sputtering procedure. Additionally, the impact of doping chromium with DLC on mechanical and corrosion behaviour is covered. From the above literature it has been observed that Cr can play a crucial intermediate layer between the coating and

substrate. Also heat treatment can enhance the mechanical and tribological properties of any kind of coating. There is very less work has been done by considering heat treatment of the bilayer of thin film coatings. More specifically the heat treatment of Cr-DLC is unexplored. So, in the present work a through mechanical analysis have been done for the study of heat-treated Cr-DLC coatings.

## 2 MATERIALS AND METHOD

### 2.1 Sample Preparation

A DC magnetron sputtering system was used to synthesis the Cr/DLC bilayer thin film over Silicon (100) substrates. Before synthesis, the substrates underwent an ultrasonic cleaning using acetone for 10 minutes, followed by rinsing with isopropyl alcohol. After that, the cleaned substrates were mounted onto the holder and inserted into the load chamber of the sputtering system. For the Cr/DLC coating, Chromium and Carbon targets, having a 3 inches diameter, were positioned at valve positions 1 and 3, respectively. These targets were affixed to water-cooled magnetron cathodes with a purity of 99.99%. The process parameters and their respective values ate given in table 1. Post deposition the sample were heat treated in a vacuum furnace at different temperatures the details of which is presented in table 2.

Process Parameters	Values
Ar gas flow rate (sccm) for the Carbon coating	50 sccm
Bias power (Watt)	70 watts
Deposition time of Cr	20 minutes
DC power Chromium target	100 watts
DC power Carbon target	200 watts
Substrate used	Si (100), Glass
Distance between substrate and target	40 mm
Substrate rotation	60 rpm
Substrate Temperature	Room temperature
Deposition time of the Carbon layer	100 minutes
Argon gas flow rate for the Chromium layer	50 sccm

Table 1. Deposition conditions for Cr/DLC thin films

Sample	Temperature (°C)
Sample 1	270
Sample 2	300
Sample 3	330
Sample 4	360

Table 2. Heat treatment of Cr/DLC coating with various temperature

### 2.2 Coating Characterization Details

The CHI608E equipment was used to assess the DLC coating's resistance to corrosion. After obtaining corrosion current ( $I_{corr}$ ) and corrosion potential ( $E_{corr}$ ), CHI software was used to create the  $E_{corr}$  vs.  $I_{corr}$  figure. For corrosion testing a 1M NaCl solution had been prepared by incorporation of distilled water with reagent-grade reagents, keeping the NaCl concentration at

3.02 mol/l. The Cr-DLC sample was used as the working electrode in these room- temperature, open-air tests, while platinum wire was used as counter electrode and Ag/AgCl served as reference electrodes, respectively. Using nano-hardness tester (NHTX 55-0019), the mechanical properties of the Cr- DLC coatings were evaluated by means of the nano- indentation method. An indenter (designation B-I 93) with a 20 nm radius of curvature was attached to this apparatus. Ten load cycles, a loading-unloading rate of 10 mN/min, a maximum load of 2 mN, and a dwell time of two seconds were all applied. The mechanical properties of the Cr-DLC coatings were investigated through the calculated values of hardness (H) and Young's modulus (E), which served as standard metric.

## 3 RESULTS AND DISCUSSION

### 3.1 Morphological Analysis using Atomic Force Microscopy (AFM)

As seen in Figs. 1(a) and 1(d), respectively, the surface morphology of the Cr-DLC thin film coatings heat treated at different temperatures was investigated using AFM. The corrosion test results are consistent with the patterns seen in the coatings that were created at various annealing temperatures. As the annealing temperature increased, the coatings showed less surface roughness with smaller peaks, valleys, and flaws at temperatures ranging from 270°C to 360°C [Fig. 1(a) & 1(d)]. At 270°C, 300°C, 330°C, and 360°C, the heat-treated coatings' surface roughness ( $R_a$ ) values were found to be 6.026, 5.017, 4.882, and 3.981 nm, respectively. Raising the annealing temperature enhanced the densification and smoothness of the film, which may have increased atomic mobility without leading to graphitization. Roughness can be decreased by surface atoms rearranging into lower-energy forms due to increased atomic mobility. Additionally, granular coalescence and the smoothing of nanoscale asperities may result from increased surface diffusion with rising annealing temperatures.

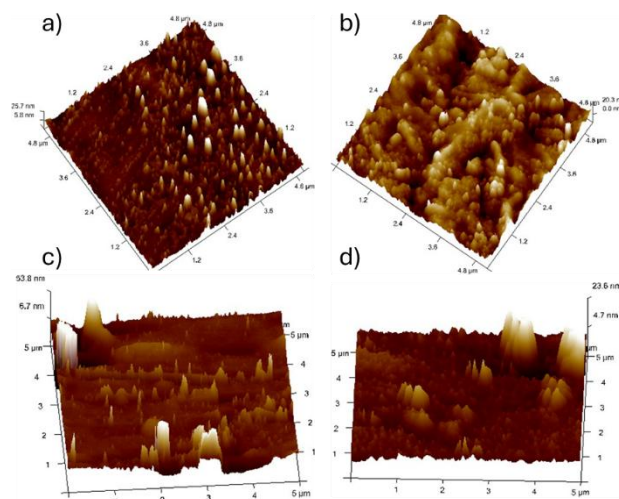


Figure 1. AFM images of heat-treated Cr-DLC at a) 270°C, b) 300°C, c) 330°C, and d) 360°C.

### 3.2 Electrochemical Analysis

The resistance to corrosion of Cr-DLC coatings coated and heat treated at various furnace temperatures was investigated using electrochemical potential (V) and current density potentiodynamic polarization curves shown in Fig. 2. It was clear that the Cr-DLC coatings' resistance to corrosion rose as the temperature rose. A boost in corrosion resistance was indicated by the Cr-DLC coatings' corrosion potential values moving to the positive side of the X-axis. Table 3 displays the  $E_{corr}$  and  $I_{corr}$ .

values for the samples. The development of more electrochemically active sites may be the cause of the corrosion resistance increase that occurs with successive temperature increases [Viswanathan 2017]. A slight change in the hybridization of carbon within the DLC matrix may occur during heat treatment. Both  $sp^2$  (graphite-like) and  $sp^3$  (diamond-like) bonded carbon are present in the coatings. Thermal energy can help  $sp^3$  bonds partially change into  $sp^2$  bonds as the temperature rises, particularly over 300 °C. By altering the surface chemistry, this controlled rearrangement improves stability. By limiting excessive graphitization and preserving the coating's hardness while increasing its chemical inertness, which improves corrosion resistance, chromium helps maintain equilibrium. Furthermore, during heat treatment, chromium in the DLC matrix may experience surface oxidation, particularly if there is even a little quantity of oxygen present in the air. A thin coating of chromium oxide ( $CrO_3$ ) is created as a result of this oxidation. Because of its thick, solid, and self-healing characteristics, chromium oxide is renowned for having exceptional resistance to corrosion. The total corrosion resistance of the coating is increased by this passive layer, which serves as a protective barrier by drastically lowering the interaction of the underlying DLC material with corrosive conditions. Additionally, heat treatment helps to cure microstructural flaws like voids or microcracks that may have developed during the sputtering process and reduce porosity. Atoms can reorganize into more stable structures when their mobility increases with thermal energy, thereby sealing minor flaws in the coating atmosphere. This results in a less porous and more homogeneous structure, which improves the coating's longevity and resistance to corrosion by reducing the passageways for corrosive chemicals and creating a smoother surface. The electrochemical test findings and the AFM results covered in section 3.1 are in good agreement with the estimated CR and comprehend how variations in process temperature impact Cr-DLC coatings' ability to withstand corrosion (Eq. 1) [Das 2021; Chen 2023].

$$CR = K \frac{E.W. \times I_{corr.}}{d} \quad (1)$$

Where  $d$  is the test sample's density (Cr-DLC-2.8) in  $gm/cm^3$  or  $2800 Kg/m^3$ , and  $E.W.$  is the Cr-DLC Equivalent weight, its value is computed as  $86.67 g/equivalent$ .  $E.W.$  is equal to molecular weight times the number of cations or anions times the charge. The phase composition of Cr-DLC, which were found by X-ray diffraction tests, have been taken into consideration while determining the Cr-DLC equivalent weight. Fig. 3 illustrates the anodic and cathodic slopes for greater clarity.

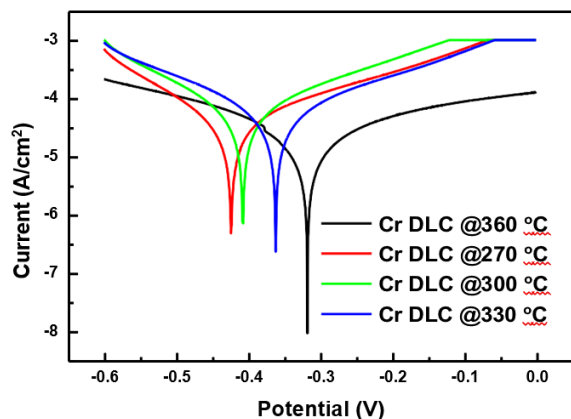


Figure 2. Tafel plot of heat-treated Cr-DLC.

Exp. No.	$E_{corr.}, V$	$I_{corr.}, A/cm^2$ [Antilog values]
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Sample 1	-0.424	-6.29
Sample 2	-0.408	-6.12
Sample 3	-0.362	-6.64
Sample 4	-0.317	-8.06

Table 3. Corrosion resistance of Cr doped DLC

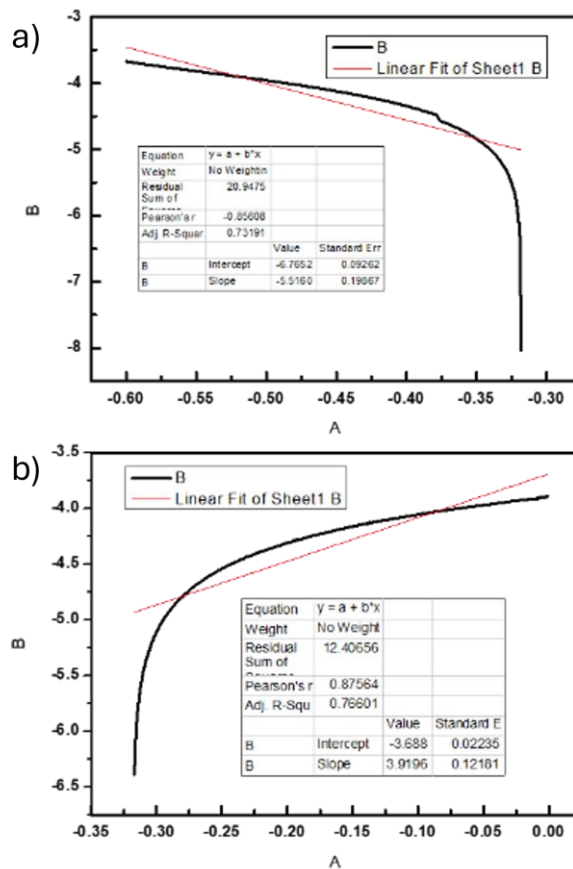


Figure 3. (a) Anodic slope calculation of Cr-DLC thin film coating, (b) Cathodic slope calculation of Cr-DLC thin film coating.

For Cr-DLC, the molar mass has been evaluated as follows:  $(1 \times Cr_{molar-mass}) + (1 \times C_{molar-mass}) = (1 \times 51.996 g/mol) + (1 \times 12.011 g/mol) = 64.007 g/mol$ .

Since Cr-DLC has one mole of Cr, the equivalent weight would be = Molar mass of Cr DLC/1 =  $64.007 g/equivalent$ . Thus, the approximated weight of Cr DLC is calculated as  $64.007 g/equivalent$ . Corrosion current density is highlighted in Table 4.

Temperature	$I_{corr.}, A/cm^2$ Log values	$I_{corr.}, A/cm^2$ [Antilog values with base 10]	$I_{corr.}, \mu A/cm^2$ [Antilog values with base 10]
270°C	-6.29	$5.128 \times 10^{-7}$	0.512
300°C	-6.12	$7.58 \times 10^{-7}$	0.758
330°C	-6.64	$2.29 \times 10^{-7}$	0.229
360°C	-8.06	$8.70 \times 10^{-9}$	0.0087

Table 4. Corrosion current density measurement of Cr-DLC coating

The CR of Cr-DLC coatings heated to various temperatures is shown in Table 5. According to the statistics, there is no discernible pattern in the CR values as the annealing

temperature rises. This unpredictable variation implies that the CR of Cr-DLC coatings is also influenced by variables other than deposition temperature, such as film thickness, coating surface roughness, and coating morphology. According to Table 5, the Cr-DLC coating that was annealed at 330°C had the highest CR, whereas the coating that was annealed at 300°C had the lowest CR. In an abrasive medium, a coating's crystallinity influences both its corrosion resistance and rate of corrosion. Higher crystallinity coatings usually have better corrosion resistance due to their higher densities and well-organized atomic packing. The corrosive media are successfully prevented from reaching the covered surface by the tight atomic packing.

Temperature	$E_{corr}$ , V	$I_{corr}$ , $\mu\text{A}/\text{cm}^2$	CR (mm/year)
270°C	-0.424	2.5	0.253
300°C	-0.408	0.588	0.0595
330°C	-0.362	9.33	0.944
360°C	-0.317	3.31	0.335

Table 5. Corrosion Rate of Cr-DLC coating

### 3.3 Nano-Mechanical Properties by Nanoindentation (Hardness [H] and Youngs modulus [E]).

Measurements of nanohardness have been performed at 2 mN stresses on Cr-doped DLC-coated silicon samples. Youngs modulus and hardness variations with different heat treatment are shown in Table 6. The Cr-DLC coatings have hardness levels ranging from 10.5 to 21.77 GPa. Similarly, the Youngs modulus of as deposited Cr-DLC and heat-treated Cr-DLC at different temperature observed within the range from 270.36 to 121.24 GPa. Compared to as Cr-DLC coating without heat treatment the hardness of heat-treated coatings is decreasing gradually with increase in heat treatment temperature. The decrease in hardness with increase in temperature could be due to the reason that, as annealing entails a number of diffusion-governed processes along with certain other features like material softening, coarsening, and grain development. The mechanical and electrical properties of the materials undergo changes in their microstructure along with annealing. The material becomes more ductile and tends to have less hardness with reduced internal stress post annealing process. Internal stress for unit deformation or resistance against the deformation reduces when dislocations can move freely within the coating; in other words, hardness also lowers.

Sample	Hardness (H) GPa	Youngs Modulus (E) GPa
Cr/DLC without heat treatment	21.77	270.36
Sample 1	19.6	222.4
Sample 2	16.4	282.5
Sample 3	14.3	165.2
Sample 4	10.5	121.4

Table 6. Hardness and Youngs modulus of Cr/DLC coating with respect to heat treatment temperature

In order to investigate the various mechanical properties of Cr-DLC (as deposited and heat treated) coatings more thoroughly, an analysis of the plastic deformation index ( $\frac{H^3}{E^2}$ ) value has been conducted. One measure used to assess the coating's plastic deformation is the plastic deformation index. Here,  $E^*$  is referred to as the coating's effective modulus, and it has been calculated using Eq. 2

$$E^* = \frac{E}{1-\nu^2} \quad (2)$$

Here,  $\nu$  is Poisson's ratio of Cr is 0.23 [Hossain 2022]. And the Poisson's ratio of DLC the coating is  $0.22 \pm 0.33$  [Cho 1999]. Therefore, in the present study the  $\nu$  of Cr-DLC is taken by considering the average which is approximately: 0.225. Changes in plastic deformation index is displayed in Fig. 4. The plastic deformation index of heat-treated Cr-DLC decreases with increase in annealing temperature, similar to hardness and Young's modulus; this suggests a reduced toughness value of the deposited film with increasing temperature. A critical phenomenon that has been observed while evaluating the plastic deformation index of as deposited and heat-treated Cr-DLC coating is that the ( $\frac{H^3}{E^2}$ ) value of Cr-DLC heat treated at 270°C is higher than the ( $\frac{H^3}{E^2}$ ) value of as deposited Cr-DLC coating [Ghadai 2018; Zhao 2022; Wang 2024]. The internal stress for the Cr-DLC coating was calculated using the Stoney equation, and the results are summarized in Table 7 [Chen 2015; Ghadai 2018]. The as-deposited Cr-DLC coating exhibited a compressive residual stress of 2.1 GPa, which gradually decreased with increasing heat treatment temperature. This reduction in internal stress is attributed to the relaxation of intrinsic stresses induced during the synthesis, as well as possible structural rearrangements and interfacial stress relief during thermal treatment of the developed coatings. Sample 1 and Sample 2, which heat treated at moderate temperature, showed reduced stress values of 1.8 GPa and 1.6 GPa, respectively. Further heat treatment in Sample 3 and Sample 4 led to a more pronounced decrease in stress, reaching a minimum of 1.1 GPa. The observed trend of the results suggest that post-deposition heat treatment is an effective strategy for minimizing residual stress in Cr/DLC coatings, thereby potentially improving their adhesion and mechanical stability in service applications.

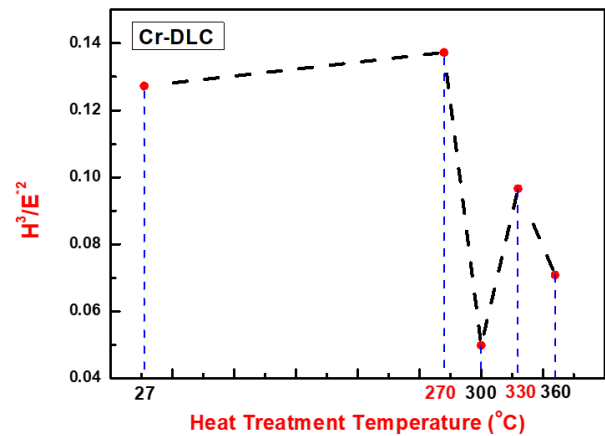


Figure 4. Change in plastic deformation index of as deposited and heat-treated Cr-DLC coatings.

Sample	Residual stress, GPa
Cr/DLC without heat treatment	2.1
Sample 1	1.8
Sample 2	1.6
Sample 3	1.53
Sample 4	1.1

Table 7. Residual stress of Cr/DLC coating with respect to heat treatment temperature

## 4 CONCLUSIONS

The present work shows that heat treatment has a significant effect on the mechanical and electrochemical properties of Cr-DLC coatings prepared using DC magnetron sputtering. The internal stress of the films, initially high due to the deposition process, was effectively reduced through vacuum furnace heat treatment, with the lowest stress of 1.1 GPa observed at the temperature 360°C. While the H and E of the coatings decreased with increasing heat treatment temperature, the overall corrosion resistance improved, suggesting a trade-off between mechanical integrity and electrochemical performance. The Cr-DLC coating heat treated at 270°C possesses the highest hardness of 19.6 GPa and Young's Modulus of 222.4 GPa respectively. Specifically, the film heat treated at 330°C exhibited the highest corrosion resistance, whereas the film heat treated at 300°C showed the lowest. However, the corrosion resistance did not follow a consistent trend with temperature, indicating the influence of other factors such as film thickness, surface roughness, and microstructural morphology. These above results show the importance of optimizing post-deposition heat treatment parameters to balance mechanical properties and corrosion resistance in Cr-DLC thin films for various industrial applications.

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