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Performance of Cr-DLC Thin Film Annealed at Different Temperatures

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Abstract

Diamond-like carbon (DLC) combines a range of significant physical and chemical properties that makes it suitable for several coating applications. In this work, Cr-DLC thin film is deposited by using RF magnetron sputtering with a pure chromium target, argon (Ar), and acetylene (C_2H_2) gases, as potential materials for thermal solar selective absorbers. The thin film consists of chromium (Cr) nanoparticles deposited inside DLC matrix. After deposition, the samples were exposed to temperatures of 500 and 800 ̊C for 2 hours in air (annealing). The deposited Cr-DLC thin film was characterized and tested in both as- deposited condition and after annealing. The microstructure and the optical properties (absorbance and emittance) of the sputtered Cr-DLC thin films were studied before and after annealing. It was found that the optical absorbance of the sputtered Cr-DLC thin film was about 97% for the first time reported. After annealing at 500̊C the absorbance decreased to 91% in UV, visible light, and IR ranges. The microstructure of the deposited Cr-DLC is transformed after annealing into chromium oxide, graphite oxide, graphene oxide.

Keywords: PVD; Thin film; Cr-DLC; Microstructure; Optical properties.

1. Introduction

The generation of energy from sunlight still faces many challenges such as low efficiency and high cost. Advances in material science and engineering are the key word towards enhanced generation of solar energy with high capacity [1]. Concentrated solar power (CSP) is one of technologies used to convert solar energy into thermal energy, where solar radiation is focused on receiver tube by concentrator and then the absorbed thermal energy is transferred to the working fluid in the receiver tube. Solar collector efficiency can be raised by developing this technology and using available radiation. High performance of solar collectors is achieved by increasing radiation absorbance and reducing the heat losses to surrounding. Also, the high efficiency of solar energy conversion is related to the intrinsic properties of the materials used in the manufacturing of photovoltaic or solar thermal collector systems. In this aspect, thin films and nanomaterials used in solar energy conversion applications for developing solar systems with higher efficiency and lower cost are significant [2,3,4]. Due to their excellent physicochemical properties, carbon- based nanomaterials are

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promising candidates which still are not extensively studied.

This work focuses on thermal solar energy conversion applications, especially CSP's. The solar selective absorber (SSA) coating is considered as a core for solar thermal energy conversion. SSA should capture as much as 99% of the solar spectrum at $\lambda \leq 2.5 \mu m$ and should show low emittance at $\lambda \geq 2.5$-$25 \mu m$. As the SSA absorbance increases the efficiency of the solar plant increases [1]. SSA thin films can be fabricated (deposited or plated) by many techniques such as chemical methods (sol gel & wet chemical), plasma assisted chemical vapor deposition techniques CVD, or physical methods such as ion beam deposition method, pulsed laser deposition, RF plasma assisted deposition, and sputtering techniques (physical vapor deposition). Thin films deposited by sputtering techniques have high adhesion to the substrate, homogeneity, and good performance at high temperatures. There are many types of SSA such as Multilayer absorbers, and metal-dielectric composites, etc. The targeted types of SSA should have good thermal stability at high temperatures .

Different types of materials have been investigated in recent years to be used as SSA coatings [5,6,7]. Metal-dielectric composite coatings or cermet have proven high absorbing efficiency as selective absorbers when deposited on metal. They consist of fine particles of metals embedded in a ceramic matrix .[1]

The diamond-like carbon (DLC) films have a wide variety of optical applications [8,9], corrosion protection [10], and wear resistance [11,12]. The good performance of DLC is due to its low friction, chemical inertness, wear resistance, and favorableoptical characteristics. DLC thin films, with their optical, chemical, and thermal characteristics are used widely as protective hard coatings in electronic devices [13]. Occasionally, DLC has been investigated to enhance the absorption of solar radiation in selected wave lengths to conduct heat to working fluid in solar collectors.[14,15]

DLC thin films can be developed by different techniques & compositions, but to obtain the DLC metastable structure films are deposited by plasma-enhanced chemical vapor deposition (PECVD) or physical vapor deposition (PVD) techniques [16]. A previous study, focusing on the same field, investigated the microstructure and different properties of DLC thin film deposited by pulsed laser deposition and showed that it can be used for solar plants applications [17]. The optical properties of DLC were investigated before and after annealing at 290̊C and the absorbance was shown not to be affected by annealing at the investigated temperatures [18]. In another investigation, DLC was deposited by RF Plasma from a gas mixture of acetylene (C_2H_2) and hexafluoroethane (C_2F_6) to form (F-DLC) and it was found that by increasing the fluorine content, the optical band gap energy increased [19]. Nitrogen & oxygen addition to DLC was also reported to affect its band gap, nitrogen increased the optical band gap, while the addition of oxygen decreased it [20]. The optical properties of DLC were also investigated [15-26], and the significant optical properties and stability at high temperatures up to 500̊C [27] make it a good candidate for medium temperature solar energy applications. However, few studies have explored the suitability of metal-DLC composite coatings for selective solar absorbers.[27]

Recently, metal-doped DLC (Me-DLC) have gained interest as their superior properties in presence of metal nanoclusters or nanocrystalline metallic carbides implanted in the carbon network were shown [27]. Moreover, Me-DLC exhibits high hardness, high magnitude of conductivity, and good adhesion to the substrate. The properties of Me-DLC thin films are usually related to the type of the transition metal which is integrated in the DLC thin film, such as Cr, W, Ti, Pt [27]. DLC is investigated with other thin films as a coating for triboelectric nanogenerators. They found coated specimens with DLC had a higher performance than uncoated[28]

Previous work has shown that the structure of the deposited DLC is problematic in case of DLC layers, which resulted in an interest in control of the structure of the deposited material [29]. Furthermore, the deposition rate which depends on the deposition techniques and systems, mainly controlled by the flow rate of gases pumped into chamber, the rotation speed of the holders carrying the substrate, temperature, and pressure.

Literature review has shown that DLC thin films exhibit the desirable properties for CSP applications, such as controlled optical properties, chemical inertness at a wide range of temperatures and high hardness. Moreover, DLC is not investigated enough for the use as solar selective absorber coating [25,26]. The optical and other properties of DLC thin films with different metals embedded were investigated by a few researchers and promising results were announced [25,26]. It was concluded in earlier research that they have a strong potential for application in passive electrical devices [18]. However, up to the author's knowledge, no work is reported that investigates SSA coatings composed of Cr nanoparticles embedded in DLC as an absorber layer by PVD sputtering. In thermal solar applications, heat transfer through the absorber is of great importance. Graphite and Cr nanoparticles are good conductors, but their optical absorbance and emittance properties and stability at higher temperatures are not reported for thermal solar applications. Corrosion properties of DLC are investigated and exhibit a high corrosion resistance for a long period of time[30]

Aslan et al deposited DLC with Ge by DC magnetron sputtering to use as solar simulator [31]. They found that thin film was responsive to light and increasing the density of Ge in thin film increasing the photosensitivity of it.[31]

The study is based on the promising optical properties (absorbance and emittance) of the Cr-DLC thin film, which suggests it is a candidate solar selective absorber coating for medium temperature solar energy applications. DLC is used as the matrix and chromium is the metal embedded inside this matrix. Cr metal target was used as an integrated transition metal into the studied DLC thin film. Acetylene and argon gases were used with chromium target in RF magnetron sputtering equipment to deposit Cr-DLC thin films. The Cr-DLC thin film microstructure and its effect on the optical properties will also be investigated.

2. Experimental

RF magnetron sputtering Protec NanoFlex 400 with 100 kHz frequency and maximum output power 2.5 KW was used to deposit Cr-DLC thin film on stainless-steel 304L (St. St.) substrates. Pure chromium target (99.999%) with size (30 x 10 cm2) was used for the deposition of thin films in reactive medium using Ar and C_2H_2 gases at flow rates 300 Sccm and 250 Sccm respectively. The substrates dimensions were 4 x 4 cm2 with 3mm thickness. They cleaned with acetone and isopropanol then dried well before being fixed in the deposition chamber. Six substrates were assigned in the chamber. The bias voltage and rotation velocity of deposition chamber were fixed at 150V and 3 r.p.m respectively. Sputtering power was preserved at 1.15 kW during the deposition. The chamber was pumped down to 10^{-4} Pa (the base pressure). Chamber dimensions about 50 cm diameter with a circular geometry. Distance between target and substrates is about 10 cm. During sputtering chamber temperature reached 160 ̊C. The deposition time was 90 min.

After deposition, six substrates were divided into three groups, where group 1was left in the as-deposited condition and groups 2 and 3 were subjected to annealing in ambient air at 500 and 800 ̊C for 2 hours (to investigate the effect of higher temperatures) in an OTF-1200X-II-UL furnace.

The optical properties were assessed by measuring the reflectance of the thin films at room temperature (absorbance & emittance). The reflectance of UV, visible and near IR was measured by spectrophotometry. In this work Shimadzu UV-3600 spectrophotometry is used to measure reflectance wavelength in range (200-2500) nm. FTIR spectrometer Jasco FT/IR-4100 was used for measuring long wavelengths (2.5-25 µm).

The thin films microstructure was observed by SEM (Scanning Electron Microscopy) model Quanta 250 FEG (Field Emission Gun) attached with EDX Unit (Energy Dispersive X-ray Analyses), with accelerating voltage 30 K.V. The surface topography of the deposited Cr-DLC thin films was measured by Keysight 5600LS AFM (Atomic Force Microscope) (N9480S) before and after annealing.

XRD (X-ray diffraction) Panalytical B.V Co., Netherlands was used to characterize the structure of the deposited thin films, using a Cu target operating at 40 kV and 30 mA with a continuous scanning type. The crystallographic phases and the structure of the thin films were determined by geometry configuration of the incident angle 2Ө.

3. Results and Discussion

3. 1 Microstructure&Structure Characterization

3.1.1 XRD Structure Characterization

XRD analysis was used to identify the phases present in the thin film. Fig.1 shows the XRD results of the as deposited and after annealing DLC layer. As shown in Table 1 graphite is found in the as-deposited DLC thin film which was oxidized after annealing at 800 ̊C to form graphite oxide (GIO) & graphene oxide (GO). Chromium carbide in two chemical compositions is found in the as-deposited thin film and after annealing indicating the presence of CrC& Cr_2C phases. Chromium carbide is a ceramic compound of high hardness, strength, corrosion resistance at high temperatures. It was not affected by annealing at 500 & 800̊C and maintained its state. Chromium oxide formed after annealing at 500 & 800°C.

Table 1. XRD Planes & Phases of DLC before & after annealing

Condition	2Ө	Phase	Planes	Reference
As deposited	30, 45, 62	Graphite	(111), (200), (220)	[32]
Phases found before & after annealing	38, 44, 51 76	CrC Cr2C	(420), (110), (200) (211)	[33,34]
Phases formed after annealing at 500̊C & 800̊C	24, 33, 42, 53	Cr3O4	(220), (202), (400), (242)	[35]
Phases formed after annealing at 800̊C	7-10	Graphite oxide (GIO) & Graphene oxide (GO)	(002)	[36]

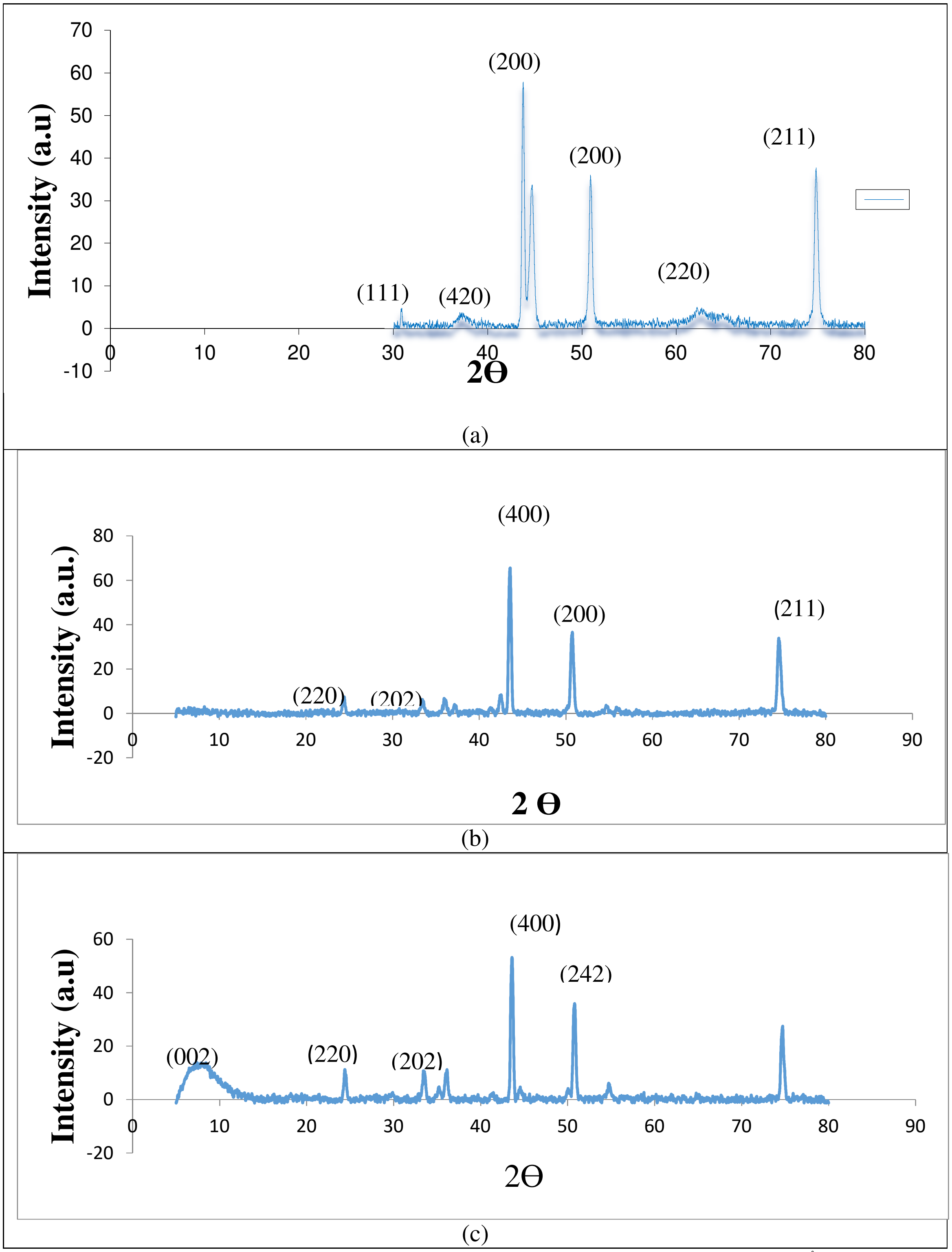
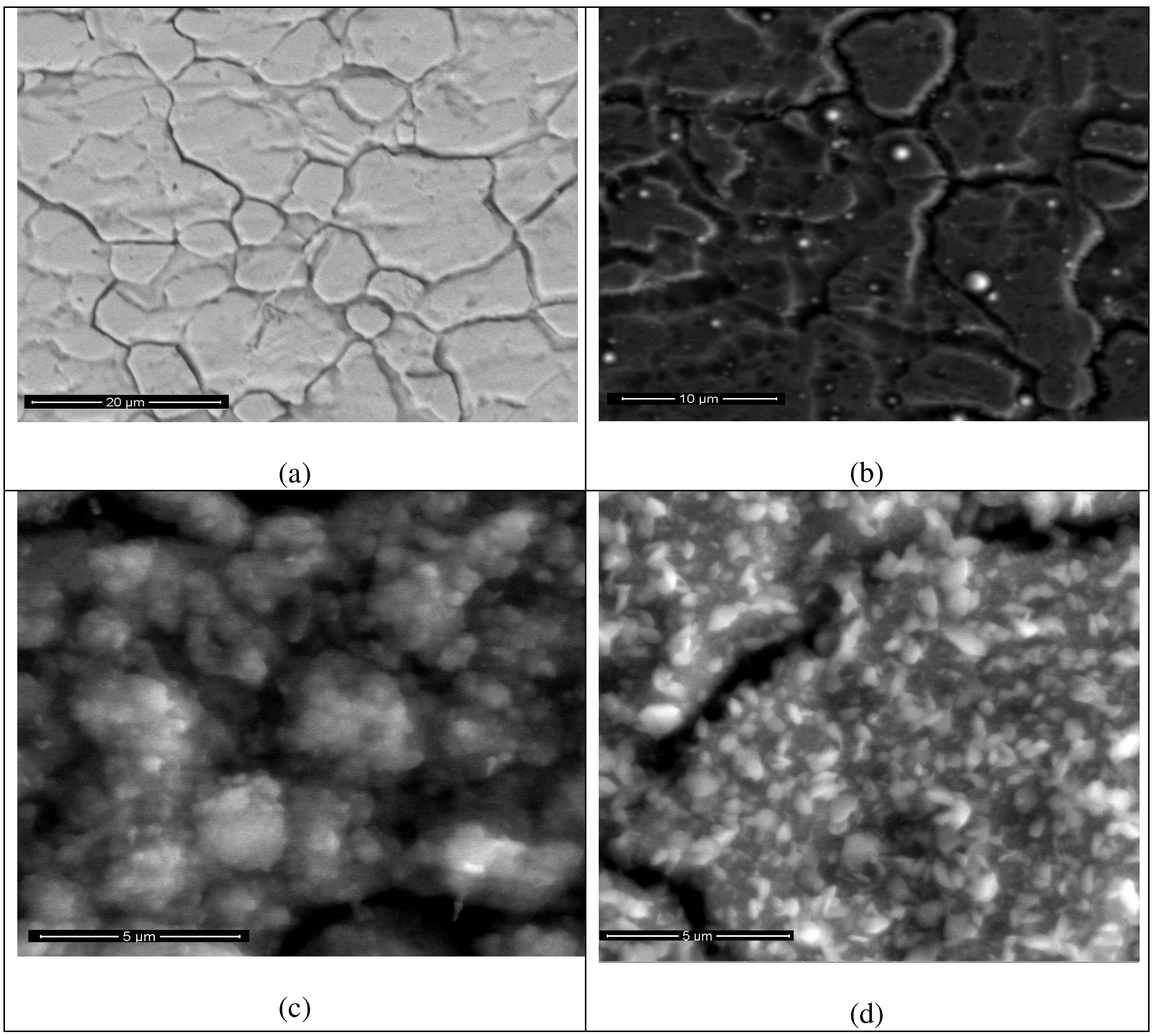


Figure 1. XRD Cr-DLC thin films before & after annealing. (a) As deposited Cr-DLC, (b) Cr-DLC after annealing at 500̊C, and (c) Cr-DLC after annealing at 800̊C

3.1.2SEM and AFM Structure and Topography Examination

As described in the experimental section, Cr-DLC is synthesized using pure chromium target and C_2H_2 gas environment in reactive medium. The

SEM microstructure examination of the deposited thin film is shown to be composed of metallic chromium nanoparticles embedded inside the DLC matrix (Fig. 2). Fig. 2b shows the as deposited epitaxial layer of Cr-DLC with chromium nanoparticles. It is shown in Fig. 2b that the chromium nanoparticles have different sizes and are distributed along the whole thin film, with different sizes varying between 70 nm – 1.2 µm.
Figs. 2c & 2d illustrates the Cr-DLC thin film after annealing at 500 ̊C and 800 ̊C which are represented the transformation of the polycrystalline microstructure from well-defined homogenous coarse grains to a porous structure containing different types of oxides. Also shown the agglomerated nano clusters occurred after annealing. The formed oxides and phases after annealing were identified through XRD examination (Fig. 1 and Table 1) to be Cr_3O_4, Graphite oxide (GIO) and Graphene oxide (GO).
Fig. 3a shows SEM image of as-deposited DLC with high magnification to clarify the deposited nanoparticle in thin film. EDX is investigated in two positions first one in the white nanoparticle appeared in Fig. 1a and second beside it. Fig. 3b represents the EDX in first position which is the composition analysis of the nanoparticle. The EDX analysis in second position beside the nanoparticle is found by Fig. 3c. The carbon weight increases from 18% to 23%. High percentage of Cr% appeared in EDX is evidence of deposition of Cr nanoparticles in DLC thin film. The EDX values in Fig. 3b show that the Cr content reaches about 67% while Fig. 3c shows a lower percentage of about 53% Cr beside the nanoparticle. The small Ni and Fe peaks are from the stainless-steel substrate.



(a) (b) (c) (d)

Figure 2. SEM of Cr-DLC thin films before and after annealing. (a) St. St. substrate, (b) As deposited Cr-DLC, (c) Cr-DLC after annealing at 500̊C, and (c) Cr-DLC after annealing at 800̊C.

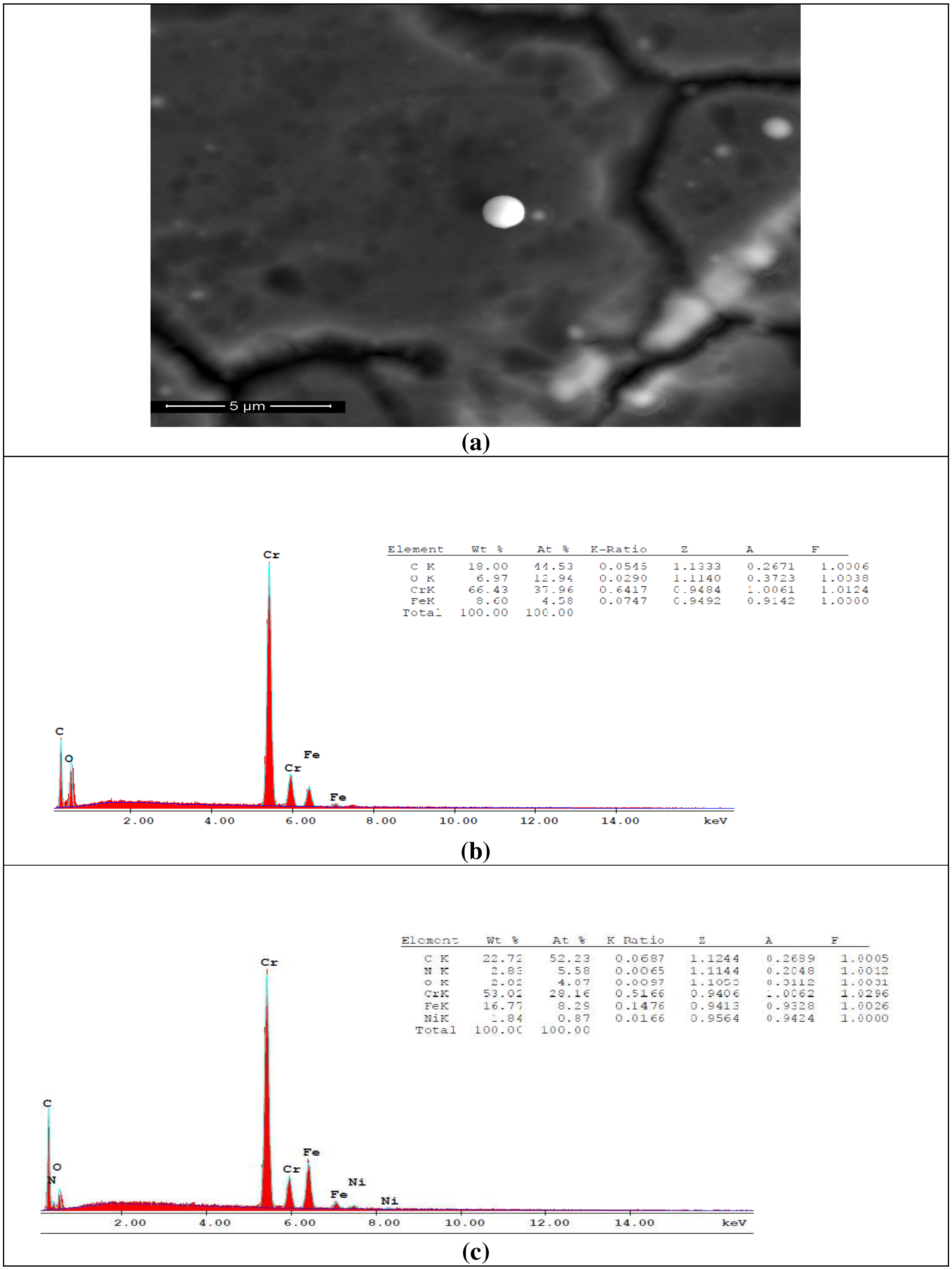
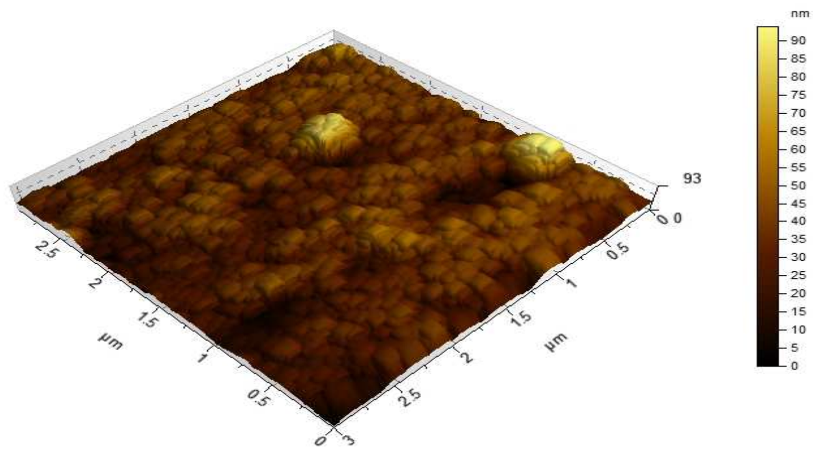


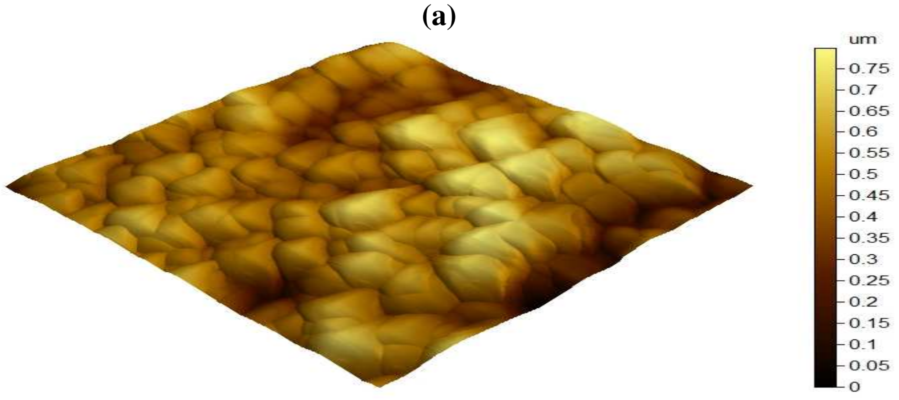
Figure 3. EDX of Cr-DlC as- deposited thin film at two different positions. (a) As-deposited thin film SEM with higher magnification, (b) EDX of first position, and (c) EDX of second position

SEM analysis

SEM analysis did not reveal the surface topographical changes. AFM investigations are applied to explore the surface topography. Figure 4 shows the AFM images of the thin film before and after annealing (Figs. 4a and b) with the substrate reference for comparison (Fig. 4c). The thickness of the deposited thin film is about 270 nm, measured by step hight method. The deposited smooth DLC layer consists of graphite diamond like grains with the metallic nanoparticles of Cr showing up as rounded particles embedded [36] at the edges of the image in Fig. 4a. The roughness of the as-deposited thin film is about 150 nm while after annealing at 500°C the roughness decreased to 93 nm, and after annealing at 800°C the roughness decreased to 44 nm. After annealing at 500°C the DLC layer shown in Fig. 4b exhibits change in the morphology compared to Fig. 4a for the as-deposited layer. Comparing Figs. 4b and c showing both the AFM microstructure of both the deposited coating and stainless-steel substrate after exposure to 500°C illustrates the difference in the matrix and the change in the surface morphology of the stainless-steel substrate after depositing the DLC. The decrease in the roughness of the thin film influences the optical properties, as will be shown later. The shape of the grains in the microstructure is changed after annealing at 800°C as shown in Fig. 4d, as it takes the shape of lima bean and becomes incomplete. The significant drop in the roughness indicates that exposure to temperature as high as 800°C results degradation in the deposited layer, which is expected to result unfavourable effects on microstructure, roughness, and optical properties of the thin film [37].



(a)



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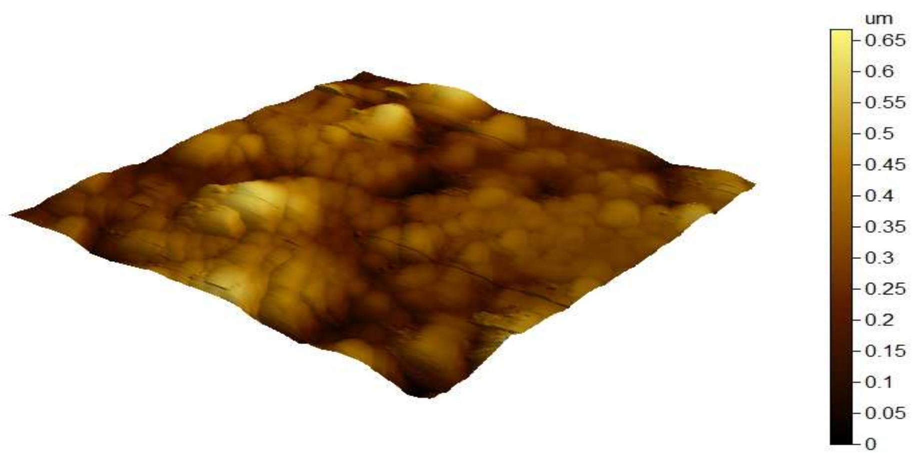

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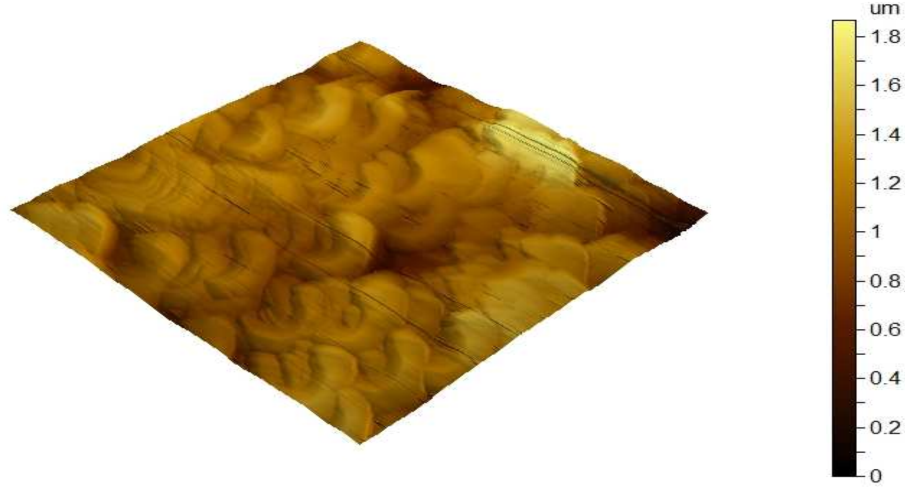
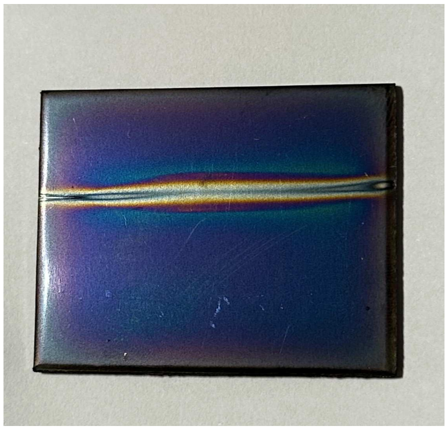


(d)

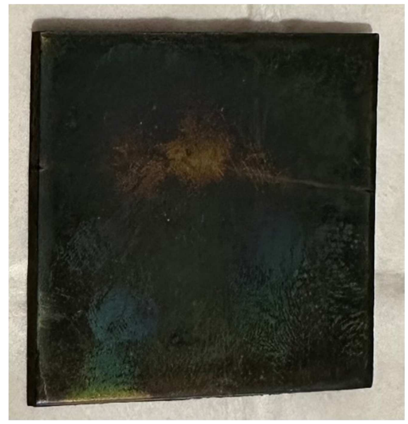
Figure 4. AFM of Cr-DLC thin films before & after annealing. (a) As deposited thin film, (b) Cr- DLC after annealing at 500°C, (c) Reference St. St. substrate after annealing at 500°C, and (d) Cr- DLC after annealing at 800°C.

3.2 Optical Properties

The formation of chromium oxide after annealing results changing the colour of the coating to dark green, instead of the purple surface obtained in the as-deposited state as shown in Fig. 5. Fig. 6 shows the absorbance of the as-deposited Cr- DLC thin film before and after annealing at 500°C & 800°C. The as deposited thin film shows the greatest absorbance about 97% in IR range. After annealing at 500°C the absorbance decreased to 91% in UV, visible light, and IR ranges. Annealing the thin film at 800°C resulted drop in absorbance to about 88% in UV, visible light ranges, and IR ranges. The high absorbance of the as deposited thin film is attributed to the metallic chromium nanoparticles in DLC matrix acting as high efficiency cermet selective absorber. Also, the high roughness of about 150 nm contributes to increasing the absorbance. The drop in the optical absorbance of the thin film exposed to annealing at 500°C indicates that the thin film starts degrading at this temperature. Surface topography is an important factor in determining the optical properties of the surface. As shown in Fig. 4a uniform topography is appeared with high roughness leading to higher absorbance. The high roughness increases the absorbance by dispersing incident light inside thin film. Figs. 4b&4d show that the uniformity of topography and roughness after annealing are decreased which resulted in decreasing of the absorbance.



(a)



(b)

Figure 5. Thin film images. (a) as deposited and (b) after annealing at 500̊C

The decrease in roughness of the annealed thin film at 800 °C to (44 nm) and the formation of GIO and GO oxides contributes to the drop in the absorbance. Fig. 7 Shows the emittance of the Cr-DLC before and after annealing at 500 & 800°C, from which it is shown that the annealed thin film at 500°C has the lowest emittance.

Based on the previous results, this work suggested SSA configuration for mid- to high temperature thermal solar applications. The suggested configuration integrates Cr- DLC thin film as cermet selective absorber on a stainless-steel substrate. The suggested selective absorber is expected to have good performance and can be commercialized due to ease of manufacturing and low cost.

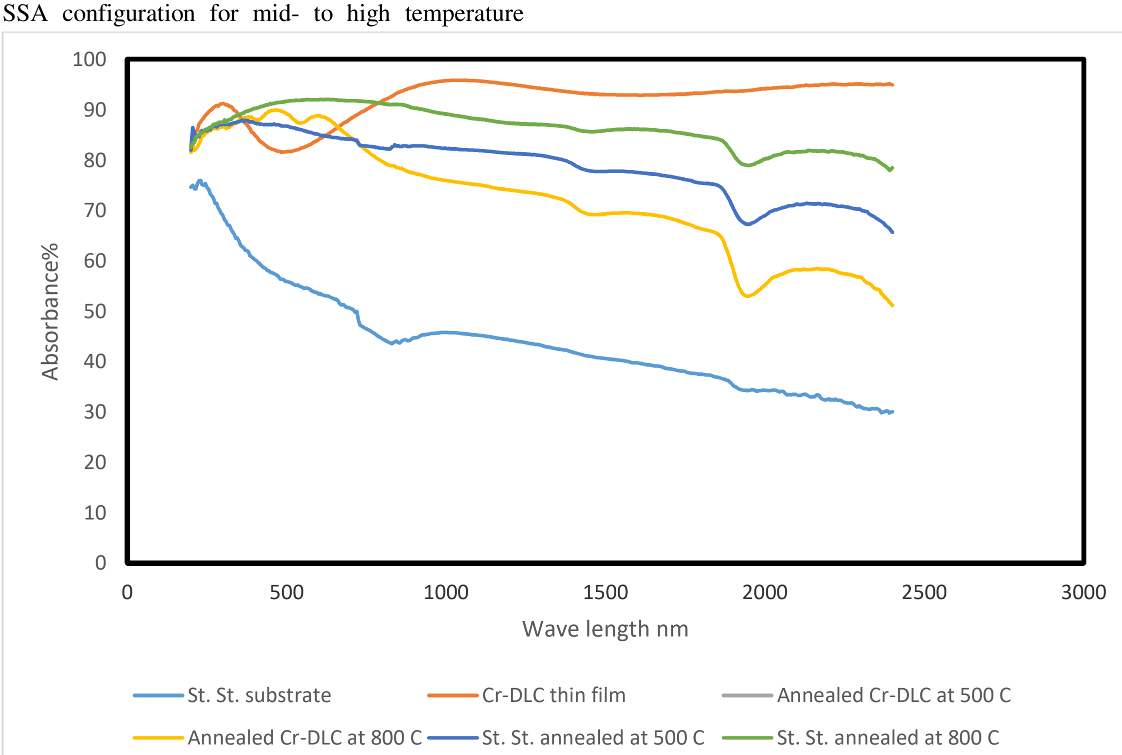


Figure 6. Absorbance of St. St. substrate and Cr-DLC thin films before & after annealing at different temperatures.

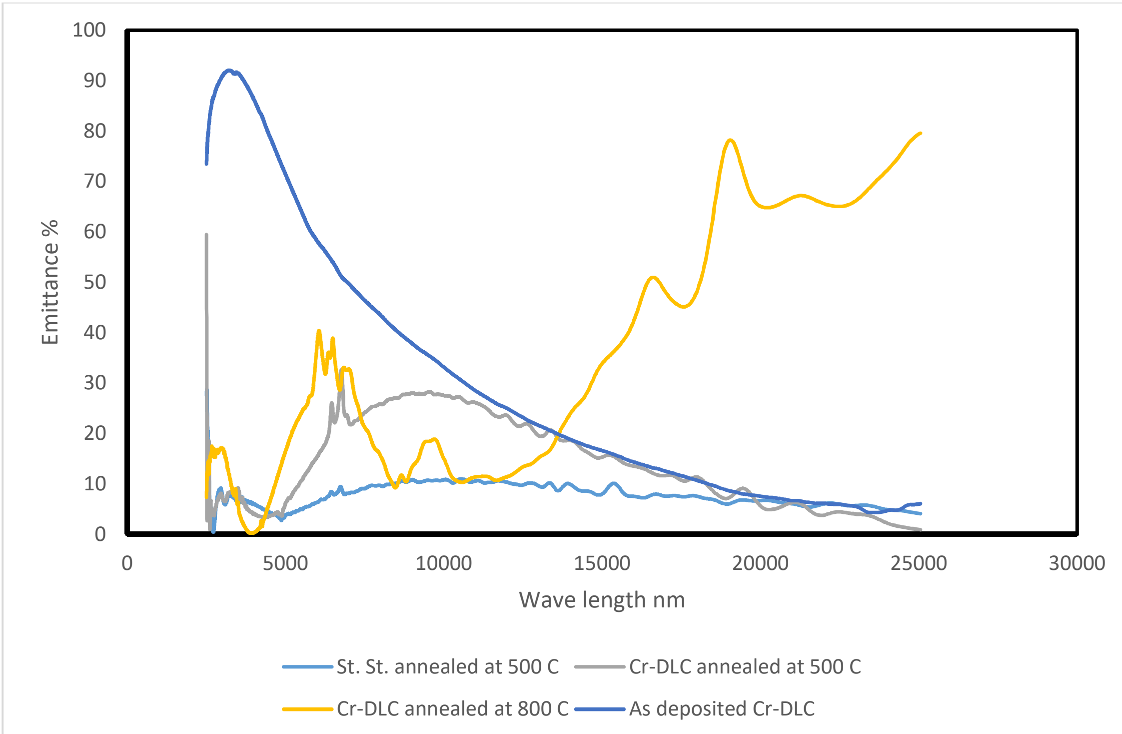


Figure 7. Emittance of Cr-DLC thin films and St. St. substrate before and after annealing at different temperatures.

This work has explored the opportunities for DLC coating to be applied for thermal solar selective absorbers. DLCs have been investigated for different biological, tribological, medical applications during the last 30 years. Moreover, DLC has a large scale of sp3 hybridized C molecules [38]. Cr/DLC films are extensively reported in literature for applications requiring good stability and wear resistance [39]. They have been studied for SSA very rarely during the last 10 years [25,26]. However, the recent interest in studying DLC thin film with embedded metals Me-DLC [31, 40], (Ge-DLC and Cr-DLC) focusing on its structure, microstructure, topography, and optical properties encourages further research on opportunities for selective solar absorbance applications. On the other hand, High cost of fabrication method (PVD) acts as an obstacle in the way of production and using Cr-DLC thin films for practical applications. In this research, enhancement has been achieved in the surface absorbance to reach 0.97, which means more useful energy to be harnessed and consequently higher working fluid final temperature that might be passed directly to have high pressure and temperature steam or stored in thermal energy storage systems. Cao [41] et al described a weighing factor to determine which is the dominating factor that affects the absorber efficiency. In case of a small weighing factor, the solar absorbance is overwhelming the thermal emittance, conversely, if it is big, the case is the other way round. Considering CSP systems, for higher fluxes and a surface temperature approaching 500 degrees Celsius, the weighing factor looks to be less than 0.1, which means higher superiority of solar absorbance versus thermal emittance. As per the concluded results for the enhanced solar absorbance up to 0.97, the thermal emittance will be expected to increase up also, a result which from the first sight looks unfavourable in general thermos fluids, but for CSP systems, the higher fluxes at high surface temperatures do not have an adverse effect on the absorber efficiency.

4. Conclusion

This work presents Cr-DLC thin film as a cermet selective absorber. The optical properties and the microstructure results for Cr-DLC thin film obtained by RF sputtering and annealing of the deposited thin film in air at 500 & 800̊C for 2 hours are investigated. The presence of Cr nanoparticles in DLC matrix is confirmed. The formed oxides and new phases after annealing or high temperature exposure are identified to be chromium oxide, graphite oxide and graphene oxide. The thin film structure and surface topography before and after annealing are correlated with the optical absorbance and emittance in the whole solar spectrum range. The as-deposited thin film has the highest roughness and absorbance of about 97%, indicating that Cr-DLC is suggested as a selective absorber for medium temperature ranges only up to 500̊C. This will contribute to the provision of the SSA application with low cost and good stability up to 500̊C.

5. Conflict of Interest

The authors declare that they have no conflicts of interest.

6. Acknowledgments

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