

RESEARCH PAPER

## Synthesis of Faceted Thin Diamond Film on High Purity Nickel Substrate by PECVD Technique

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### ABSTRACT

In the present study, Nano-crystalline Diamond (NDs) coatings were deposited on nickel substrates by a DC-plasma enhanced chemical vapor deposition (DC-PECVD) deposition technique using methane/hydrogen ( $\text{CH}_4/\text{H}_2$ ) gas mixtures. Due to the extremely high surface energy of diamond, the nucleation and growth of diamond phase on non-diamond substrate is difficult. In order to improve the diamond nucleation density to form continuous films, a good pretreatment using suitable etching gas and magnetic field (MF) were used. The effects of MF pre-treatment on the characteristics of deposited coatings are discussed in view of experimental studies using analytical techniques such as Raman spectroscopy, X-ray diffraction pattern (XRD) and field-emission scanning electron microscopy (SEM). It was found that the quality of the diamond films grown with MF pre-treatment much higher than the film grown without MF pre-treatment process. The research indicates that the new technology of diamond growth using MF may have incredible effect on CVD diamond production at low temperature and few hours.

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### INTRODUCTION

Over the past few years, carbon-based materials such as diamond, carbon nanotubes (CNTs), diamond like carbon (DLC), fullerene and graphene have aroused intensive research interest from both scientific and engineering communities due to the unique superlative properties. Among these materials, diamond films,  $\text{sp}^3$ -bonded carbon have been considered as strategic nanomaterials for applications in a variety of current and future systems owing to their extraordinary properties including: high hardness, optical transparency, thermal conductivity, wear resistance, lowest compressibility, unique semiconductor characteristics, inertness towards most chemical

and reagents intrinsically low friction [1]. These outstanding features of diamond motivated the exploitation of this carbon nanostructure in various applications ranging from wear resistant coatings, cutting tools for non-ferrous materials, as heat spreaders for electronic devices, flat panel displays and transparent windows for infrared light [1-5], and so on. Furthermore, compared to the microcrystalline diamond (MDs) thin film, Nano-crystalline Diamond (NDs) films with smaller grains have attracted great attention in different fields such as a promising cold cathode field emission material, beholden to its lower turn-on field, high power at high temperature, high stability and reliability on service and high current

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density [6, 7]. This fact increases the power of NDs films for valuable applications in vacuum electronic devices like microwave amplifier and field emission displays [7]. However, it is noticeable that the practical use of diamond films in many scientific and industrial sectors is limited owing to its high-cost and scarcity [5]. Nowadays, numerous techniques have been developed for producing diamond films, including: pulsed laser deposition (PLD) [8], microwave plasma chemical vapor deposition [9], plasma enhanced chemical vapor deposition (PECVD) [10, 11], hot filament chemical vapor deposition (HFCVD) [12-15] and etc. Based on the unique structure of diamond with fully  $sp^3$  bonded form of black carbon, it has some challenges to growth on CVD techniques. Among these methods, plasma enhanced chemical vapor deposition has been widely used in diamond synthesis, due to its large surface deposition, easy processing, low capital cost and film uniformity control over deposition parameters [16, 17]. All parameters are so sensitive and should be rather balanced to form a high crystalline quality of this structure. Diamond growth starts when carbon atoms on the surface begin to nucleate and form  $SP^3$  bonds in the tetrahedral lattice.

There are two types of diamond growth which called Homogeneous and non-Homogeneous growth. In homogeneous type, a diamond base substrate or diamond seed is used. Then, the only thing that is necessary for the creation of a tetrahedral network is the diamond network which placed on the surface atom by atom in the vapor deposition process. In non-homogeneous type, a non-diamond substrate is used for growth, so the carbon atoms have no pattern on the substrate to follow and won't come together atom by atom like in the previous case. Therefore, after hitting the surface of the substrate, carbon atoms immediately cause surface etching and then return to the gas phase and react with hydrogen atoms. To avoid this problem, before the diamond growth process begins, the substrate must be pre-treated for the reaction. Using diamond base substrate or diamond seed is not commercial and also limits the type of substrate and applications. Therefore, using different type of substrate without diamond seed and improving the synthesis quality is one of the scientist's problems during recent years [1-7,9-17].

Furthermore, it is noticeable that due to the extremely high surface energy of diamond, the

nucleation and growth of diamond phase on non-diamond materials is difficult [18, 19]. In order to improve the diamond nucleation density to form continuous films, a good pretreatment of the substrates is usually needed [18]. Over the years, different methods for surface pretreatment have been developed, including scratching, seeding, electrical biasing, laser processing, various types of chemical and physical etching and chemical treatments, to improve substrate surface qualification before diamond deposition [18]. But none of them is not absolutely ideal method for perfect quality of produced diamond.

In addition, it has been reported that the nucleation density and the grain growth rate of the diamond besides viscosity are also affected by the magnetic field [19-22]. In this study, a DC-PECVD system were used to obtain a uniform deposition with a low cost of production. In addition, to make a high-density diamond film without using diamond powder for seeding, an etching treatment were used on Ni substrate using  $H_2$  plasma with combination of Magnetic field in pre-treatment step. This manuscript is organized as follows; following the introduction in Section 1 the experimental details are described in Section 2, Section 3 is dedicated to results and discussion and finally conclusion is presented in Section 4.

## MATERIALS AND METHODS

### Substrate Pre-Treatment

Nickel wafers (Ni) with 99.99% purity were used as the substrates in this experiment and pre-treated in two steps: All the substrates were polished with diamond paste ( $1\mu m$ ) to obtain a smooth surface. Before loading into deposition chamber, the Ni substrates ( $5mm \times 5mm$ ) were ultrasonically cleaned in bath of acetone, ethanol and de-ionized water for 15 minutes. The samples called #1 and #2 were kept in desiccators in vacuum. Sample #2 was kept in the vicinity of two magnets for 48h before loading the reaction chamber. The Magnetic Field (MF) applied in this pre-treatment process was 1.2T. A rectangle cubic magnet was used. The Magnetic field was parallel with surface.

### CVD diamond growth

The experiment is performed on the DC-Plasma Enhanced chemical vapor deposition (PECVD) system. Fig. 1 shows the schematic diagram of the PECVD system.

The main central chamber of the system is a cylindrical box with a diameter of 28cm consisting of metallic base and a cover. The DC source is flanged on the top of the system cover. In order to insert the substrates inside the chamber, an Aluminum cover open from top. The door handling by vertical shifting the cover on top of the system. The chamber is pre-evacuated by rotary pump up to the base pressure of the  $10^{-2}$  Torr. All substrates were placed on a furnace right under the anode. The temperature of the substrates is monitored by a sensitive thermocouple. In both experiments, for pre-treating the substrates, the etching process was used. An argon gas was inserted to the system for 5 minutes to clean the system and removing the oxide layers. The substrate temperature, flow rate, and etching pressure were: 550°C, 50 sccm, and 5 Torr, respectively. For creating the suitable sites for the first nucleation, the hydrogen gas was used about 20 minutes in 600°C with the flow rate of 20 sccm. The etching pressure in this case was 8 Torr. For growing process, a mixture of methane and hydrogen with~ 8% flow ratio were used for growing the NDs. The applied current and voltage in this experiment were 50 mA and 400 kV respectively.

## RESULTS AND DISCUSSION

### Raman Spectroscopy

Raman spectroscopy is a standard non-

destructive nano-materials characterization technique that is widely used for the analysis of structural typicality of carbonaceous materials, such as Diamond [23], [24], [26-27], Diamond-like carbons (DLCs) [28,29], nanotubes (CNT) [30] and [31]. The Gaussian deconvoluted Raman spectra in the frequency range from of 1000-1800  $\text{cm}^{-1}$  of the deposited films magnetic field is shown in Fig. 2. The fitted spectra exhibited many overlapped features. It is clearly observed that the obvious features at 1332  $\text{cm}^{-1}$ , 1351  $\text{cm}^{-1}$  and 1578  $\text{cm}^{-1}$ , exist in the Raman spectra of deposited coatings which are attributed to diamond of  $\text{sp}^3$ -bonded carbon ( $T_{2g}$  mode) [32], D and G band of  $\text{sp}^2$ -bonded carbon [33], respectively. The vibrational band of D peak originated from the breathing modes of  $\text{sp}^2$ -bonded carbon atoms in aromatic rings. This mode represents disorder in  $\text{sp}^2$  sites belonging to carbon framework [34, 35], while the G peak for carbon materials arises from the bond stretching of all pairs of  $\text{sp}^2$  atoms present in both rings and chains [34].

In addition, the characteristic peak located at 1440  $\text{cm}^{-1}$  as a major sign of NDs is belong to the vibration of C-H bonds in trans-polyacetylene (TPA) structure which provides indirect evidence that the hydrogen has been incorporated into the samples, specifically, at the grain boundaries [23, 36]. Furthermore, the presence of other feature at 1245  $\text{cm}^{-1}$  in these spectra is also associated with

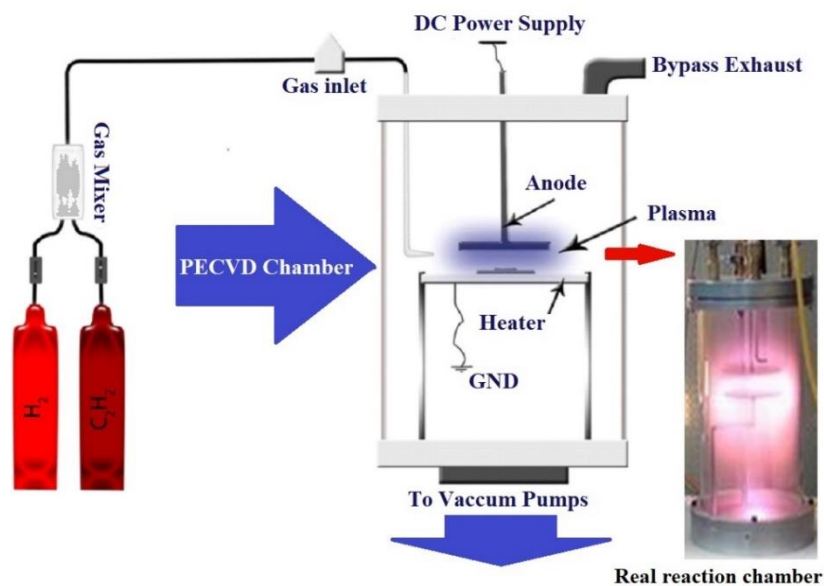


Fig. 1. Schematic diagram of the DC-PECVD system

NDs [37]. Moreover, the obvious characteristic peak locating at  $1540\text{ cm}^{-1}$  characterizes the diamond-like carbon structure [38]. The peak at  $1607\text{ cm}^{-1}$  is assigned to nanocrystalline graphite [39]. It is clear that Raman spectrum of the diamond film deposited with MF pre-treatment, as shown in Fig. 2b, has a sharp peak at  $1332\text{ cm}^{-1}$ , which indicates. On the other hand, a weak peak at around  $1332\text{ cm}^{-1}$  appears on the Raman spectrum of the diamond film deposited without MF (Fig. 2a), implying that poor quality. It is generally considered that the area intensity ratio of diamond peak to G peak ( $I_{\text{Dia}}/I_{\text{G}}$ ) could be used to assess the relative amount of  $\text{sp}^3$  phase [7]. Through multi-Gauss fitting, the  $I_{\text{Dia}}/I_{\text{G}}$  ratio of the diamond films deposited with the MF is found to be 8.7, while that of the diamond films deposited without MF is around 7.3. The former is higher than the latter, indicating that the quality of diamond film deposited with MF is much better than that deposited without MF pre-treatment.

#### Crystallinity Analysis

X-Ray diffraction (XRD) is one of the powerful techniques which contributes detailed information about the chemical composition and crystallographic structure of the materials [40-42]. The XRD patterns of deposited coatings at with and without MF pre-treatment are shown in Fig. 3. In the sample 3(a), we can see reflection peak at  $2\theta=27.98^\circ$  belongs to methane phase. Moreover, the other diffraction peaks at  $2\theta$  angles of  $38.60^\circ$  and  $39.66^\circ$  are detectable that are attributed (020) and

(-102) methanol phase, respectively. In addition, several reflections at  $2\theta=23.49^\circ, 40.66^\circ, 42.50^\circ, 48.05^\circ, 69.34^\circ$  and  $74.35^\circ$  corresponding to the plans of (009), (0 0 24), (012), (1 0 13), (1 0 19) and (0 0 42) characteristic of graphite structure can be clearly observed. Furthermore, Rhombohedral diamond phase has two diffraction at  $2\theta=41.46^\circ$  and  $46.40^\circ$  corresponding to (101) and (107) originations, respectively, the other reflection peaks assigned to graphite  $26.06^\circ$  and Ni,  $45.02^\circ$  are also detected in this sample. According to results in 3(b), we can see that the XRD pattern of the deposited sample with MF pre-treatment dramatically changes. Diffraction peak with strongly sharp intensity at  $2\theta$  angle of  $44.62^\circ$  attributing to the (103) reflection plane of hexagonal diamond can be seen in this sample. Furthermore, the other reflection peaks at  $2\theta=51.62^\circ$  and  $93.11^\circ$  belong to (0 1 17) and (200) graphite planes also is clearly observed. In addition, three diffraction peaks appear at  $2\theta=76.49^\circ, 98.40^\circ$  and  $122.03^\circ$  assigned to (220), (222) and (400) reflection planes of Ni, respectively. XRD spectra indicate that the phase transition occurs and the preferred orientation of the Rhombohedra diamond films transforms from (101) and (107) to (103) hexagonal diamond phases.

#### SEM Analysis

Fig. 4 shows typical SEM images of the films deposited MF at  $600^\circ\text{C}$  in PECVD reactor. It can be seen in Fig. 4 that the morphology of the surface mostly covered by Graphite and amorphous

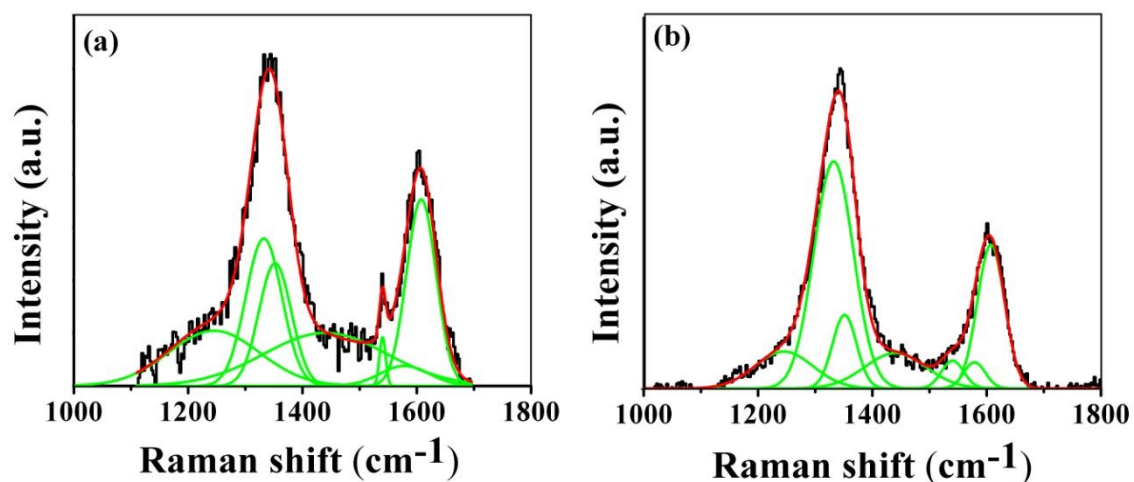


Fig. 2. Raman scattering spectra of the deposited coatings on Ni substrates (a) without MF and (b) with MF pre-treatment

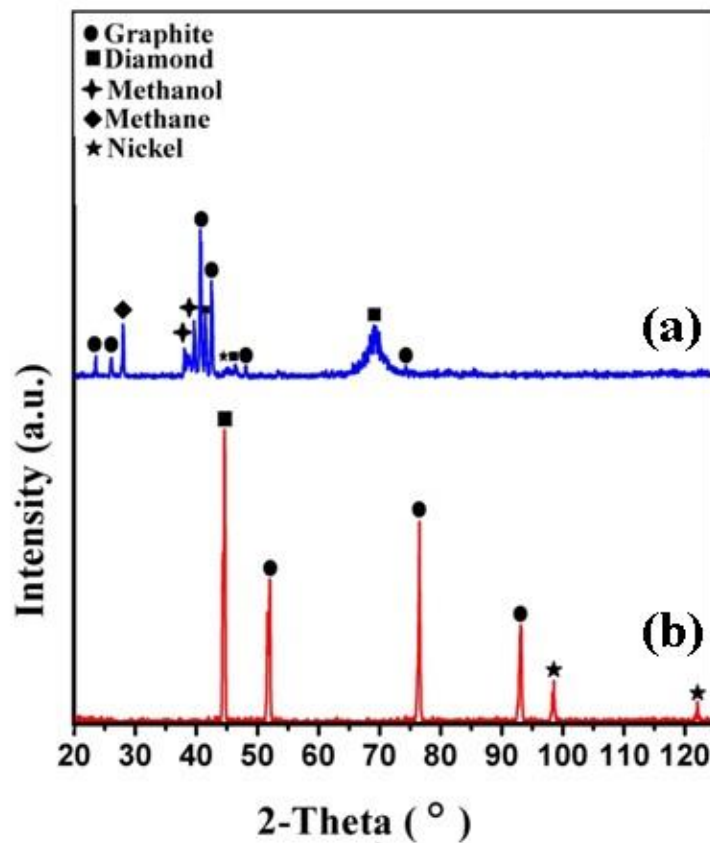


Fig. 3. X-ray diffraction patterns of the deposited coatings (a) MF and (b) with MF pre-treatment

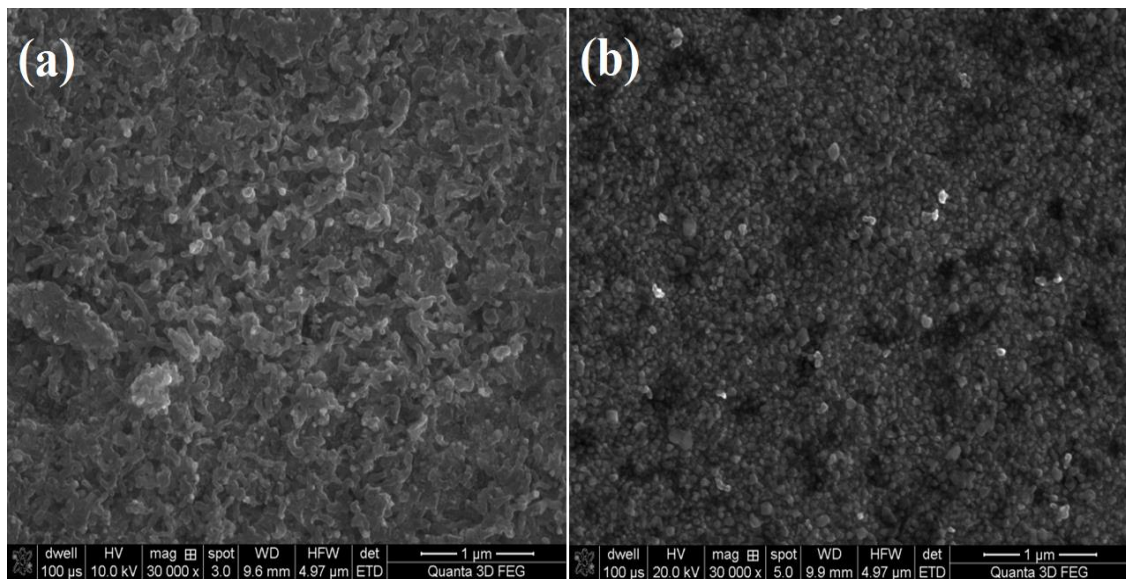


Fig. 4. Typical SEM images of deposited films on high purity Nickel substrate with PECVD system pre-treated (a) without MF (b) with MF



phase of carbon. However, high quality crystalline diamond was observed in sample #2.

The surface images were recorded using FEI Quanta 3D FEG 200/600 Scanning Electron Microscope Dual Beam: FEB and FIB. The morphology of the samples was studied here. In order to investigate the scenario of what happened in the sample #1, the cutting process was performed by using the ions beam (30 pA). After cutting with the ions beam, it was performing the scanning by using the electrons beam in order to obtain a better image. The elemental analyses was achieved with an EDAX Genesis device attached on the FEI scanning electron microscope. It can be observed that the sample #1 content preponderantly carbon according with the diamond phase identified in XRD pattern and a small amount of oxygen. For a more detailed morphological analysis there was performed a cross section using high-current ion column (Fig.

5). Quanta 3D FEG's high-current FIB enables fast material removal. Automated FIB sectioning recipes enable accurate cross-sectioning. Automated FIB sectioning recipes enable accurate cross-sectioning and low damage sample cleaning.

Table 1 shows the elemental analyze in cross section figure contain Ni 42.43% at. and Ga 6.50% at., in the area where it was made the cut using ion beam. Ni comes from the substrate on which diamond is deposited on and Ga can appear because the cross-section is made by using high-current ion column with Ga liquid-metal ion source.

In order to investigate the morphology of the surface in sample #1, the EDAX Genesis device attached on the FEI scanning electron microscope to observe content mostly carbon according and a small amount of oxygen (Fig. 6). The mentioned mark in SEM photo of Fig. 6 shows the EDAX result which described in detail in Table 2.

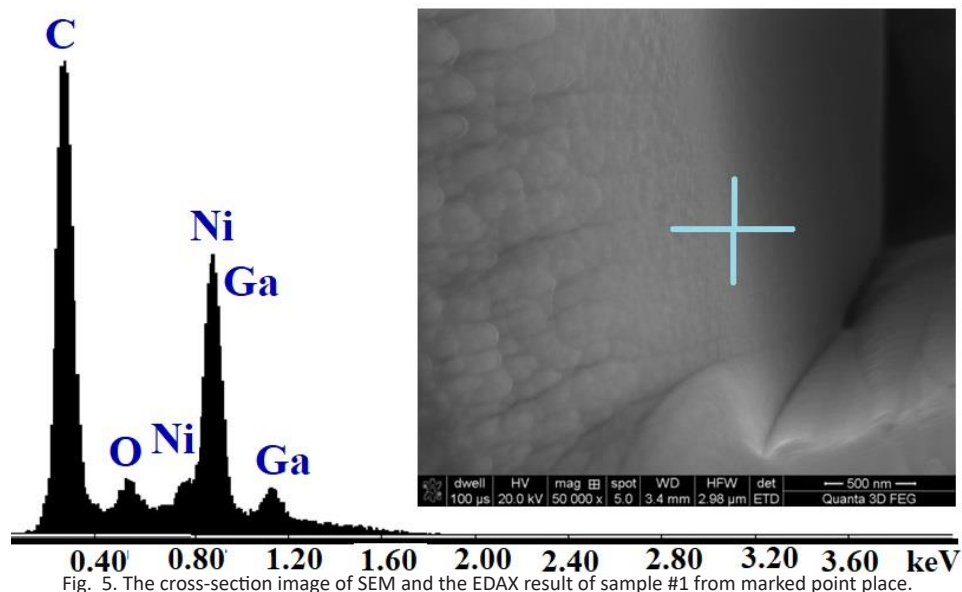


Fig. 5. The cross-section image of SEM and the EDAX result of sample #1 from marked point place.

Table 1. The result of the EDAX analysis from inside of the of sample #1 cutting with the ions beam

Element	Wt%	At%	K-Ratio	Z	A	F
C K	15.43	45.97	0.1950	1.4623	0.8640	1.0001
O K	2.28	5.10	0.0295	1.3597	0.9500	1.0007
Ni L	69.61	42.43	0.6357	0.9161	0.9967	1.0002
GaL	12.67	6.50	0.1000	0.8218	0.9599	1.000
Total	100.00	100.00				

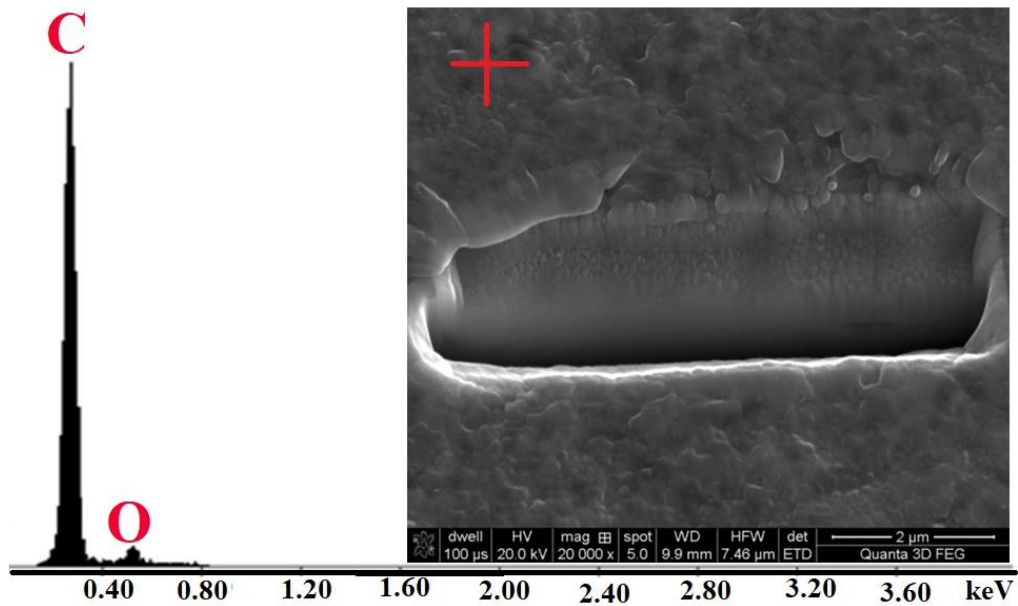


Fig. 6. The cross-section image of SEM and the EDAX result of the marked point place on the surface in sample #1.

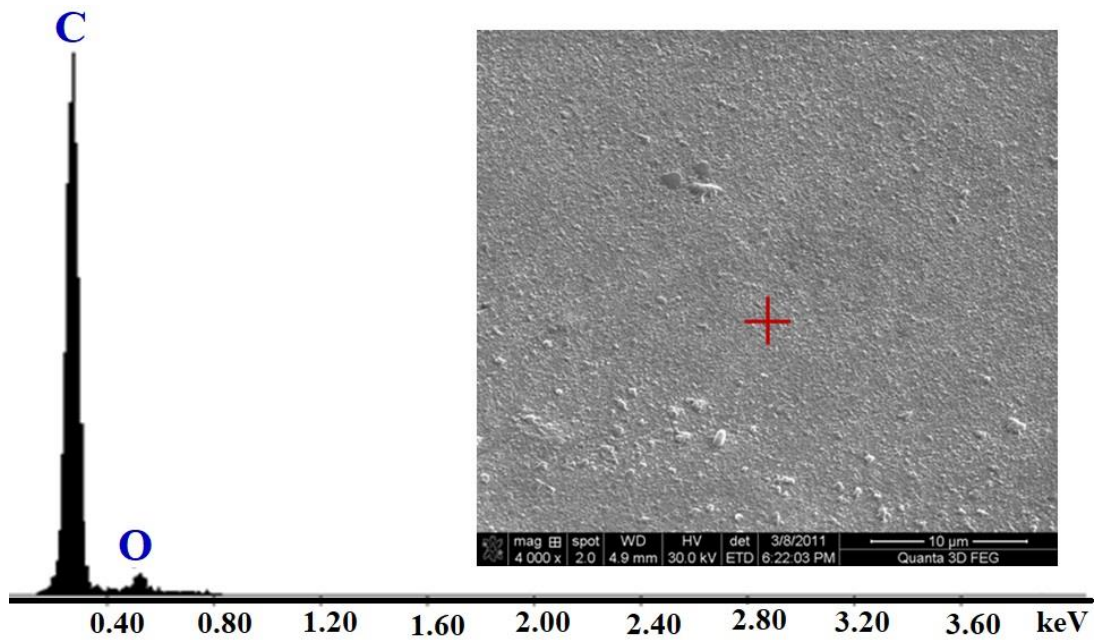


Fig. 7. The SEM image and the EDAX result of the marked point place on the surface in sample #2.

Table 2. The result of the EDAX analysis from the surface of the sample #1

Element	Wt%	At%	K-Ratio	Z	A	F
C K	0.00	90.41	0.8822	1.0082	0.9986	1.0000
O K	0.00	9.59	0.1146	0.9452	0.9802	1.0000
Total	100.00	100.00				

Table 3. The result of the EDAX analysis from the surface of the sample #2

Element	Wt%	At%	K-Ratio	Z	A	F
C K	0.00	92.44	0.9066	1.0065	0.9989	1.0000
O K	0.00	7.56	0.0909	0.9437	0.9797	1.0000
Total	100.00	100.00				

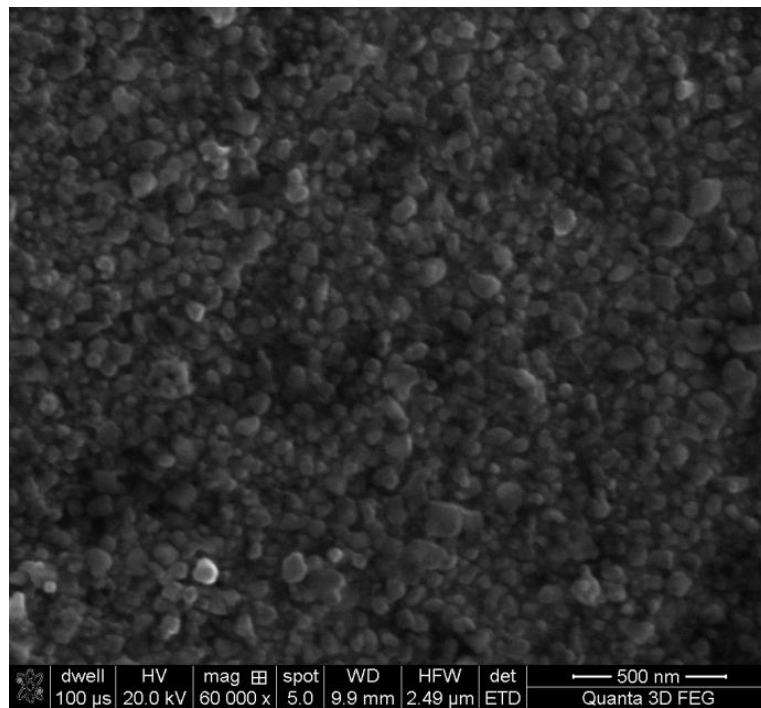


Fig. 8. High quality faceted thin diamond film synthesized on Ni substrate using MF and etching pre-treatment.

Fig. 7 shows the SEM image of diamond film covered the Ni substrate homogeneously which is completely due to the good pre-treatment with magnetic field that has a strong effect on producing faceted diamond on substrate. The EDAX were taken from the marked point (Table 3). The results show the highly amount of Carbon 92.44 at% and a few oxygens.

It can be seen in Fig. 8 that the average grain size of diamond deposited with MF pre-treatment is about 40 nm. The results indicated MF pre-treatment has a significant effect on growing high quality diamond film with (103) crystalline structure. Therefore, the results show that, for producing perfect quality of diamond and successfully coaching carbon atoms to find a suitable algorithm for growth in substrate, a combination of magnetic field before loading to

the chamber and etching gas before main reaction is a perfect choice [40-50]. This combination was a novel method for diamond production. The obtained results show a high quality of diamond film production on substrate surface [43-52].

Fig. 8. High quality faceted thin diamond film synthesized on Ni substrate using MF and etching pre-treatment.

## CONCLUSION

In the present work, nanocrystalline diamond have been deposited on Ni substrates by utilizing DC-PECVD deposition technique and the influence of MF pre-treatment on the structure and surface morphology of the deposited films was investigated. The experimental results show that the MF pre-treatment plays undeniable role on the characteristics of the deposited samples. It



has been found that the diamond films deposited with MF pre-treatment condition have good crystallinity and high quality.

#### CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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