Esraa A. Al-Oubidy
Tawfiq S. Mahdi

Department of Physics, College of Science, University of Baghdad, Baghdad, IRAQ

Synthesis of Silicon Carbide Nanostructures from Carbon Sooted-Silicon Substrates Using Magnetron Sputtering Technique

In this work, a simple, inexpensive, and fast method to prepared silicon carbide from formation of carbon soot layer on silicon substrate by air laminar diffusion flames. This layer was 143 nm in thickness and formed at different exposure times. The carbon soot-silicon substrate was used to synthesize silicon carbide nanostructures by magnetron sputtering technique at optimum conditions. The structural characteristics of the prepared samples were investigated by using x-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive x-ray (EDX) spectroscopy. The spectroscopic characteristics were determined by UV-visible and Fourier-transform infrared (FTIR) spectroscopy. As the formation of silicon carbide nanostructures was confirmed, the particle size of the final sample is the minimum found to be around 47.44 nm, with an absorption peak around 400 nm and energy band gap of 2.4 eV.

Keywords: Silicon carbide; Carbon Soot; Magnetron sputtering; Nanostructures
Received: 28 December 2023; **Revised:** 20 February; **Accepted:** 27 February 2024

1. Introduction

Silicon carbide (SiC) is a significant non-oxide ceramic with distinct properties such as high temperature, strength, oxidation resistance, high thermal shock resistance, high hardness and wear resistance, strong chemical resistance, low thermal expansion and high thermal conductivity, among other ceramic types [1-3]. These characteristics make silicon carbide an ideal contender for the production of fast, high temperature, and high voltage electrical device for abrasion and cutting applications. Furthermore, silicon carbide with present advanced technologies (e.g., MOSFETS, MEMS, sensors) can suit many practical applications; such as light-emitting diodes (LEDs) [4-6]. Silicon carbide crystallizes into a covalently linked structure that is tightly packed [7] in which the silicon and carbon atoms are linked by covalent bonds and produce polar constructions [6]. Several alternative production methods for high purity silicon (carbide, oxide, nitride) have emerged in recent years, including the carbo-thermal reduction method using binary systems, chemical vapor deposition (CVD), Acheson process, sol-gel, and physical vapor deposition (PVD) techniques such as the dc magnetron sputtering [7-11]. The latter technique has several benefits over other methods. First, any material may be deposited by magnetron sputtering with high adhesion of deposited films; second, coatings of alloys and compounds can be sputtering while retaining a composition identical to the parent material. In addition; pure and homogenous thin films can be obtained comfortably [12,13].

In the current work, soot layers were coated by allowing a flame to go over the silicon surface and use them as targets in the magnetron sputtering technique.

2. Experimental Part

The present work includes two parts: the production of carbon soot and the synthesis of silicon carbide nanostructures. The active management of soot required its production with particular features. This in turn necessitates a knowledge of physical and chemical methods; usually adopted to produce soot from fuel [14]. In most cases, soot is produced by partial rather than complete combustion. The fuel ought to be burnt at a lower temperature with a slight decrease in the oxygen supply [15]. This step is materialized to form soot in a wide and highly ventilated laminar diffusion flame by transmitting fuel through an oxidizer flowing in a cylindrical glass tube. The reaction area is formed between two opposing streams of fuel and oxidizer in order to ensure the entry of small oxygen amounts from the bottom of the glass tube to realized partial combustion and soot formation. When the fuel burns, it disintegrates into fine soot particles which settle as black powder deposits; shown in Fig. (1a).

High purity silicon wafer (99.99%) was heated on a burner flame for 2 minutes until a soot layer was deposited, as illustrated in Fig. (1b). To synthesize silicon carbide nanostructures, a dc magnetron sputtering system was utilized; with its stainless steel electrodes connected to a dc power supply that provides 150W power (50mA current and 3kV discharge voltage). The two electrodes were separated by 4cm, and the carbon soot-coated silicon wafer; representing the sputtering target, was mounted on the cathode. Plasma was generated by discharging an electric current through 0.07 mbar argon gas; Fig. (2a) shows the prepared sample after three hours of deposition time. The final samples were extracted as a powder from the thin film samples deposited on glass substrates using a conjunctional freezing-assisted ultrasonic extraction technique [16].

The final sample was a dark grey powder that is insoluble in water, alcohol, and acid, as seen in Fig. (2b).

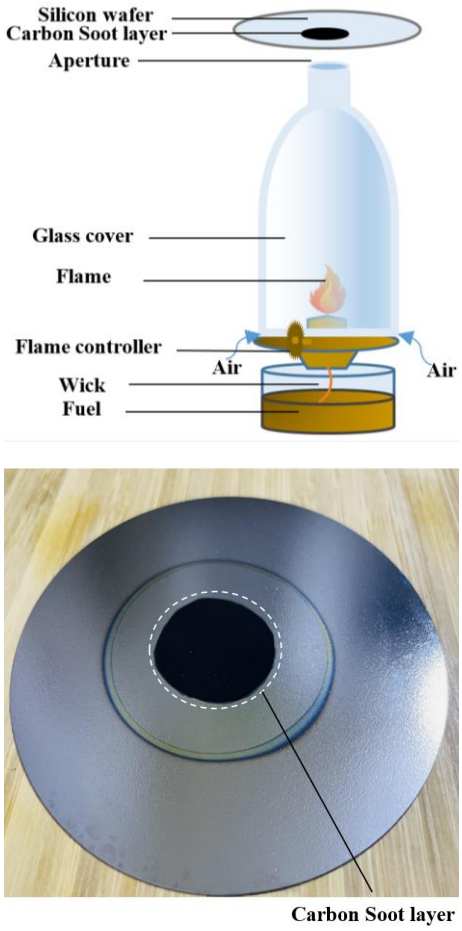


Fig. (1) Production of carbon soot layer on a silicon wafer (a) a silicon wafer is held in the flame for 2 min; (b) a carbon soot layer deposited on silicon wafer

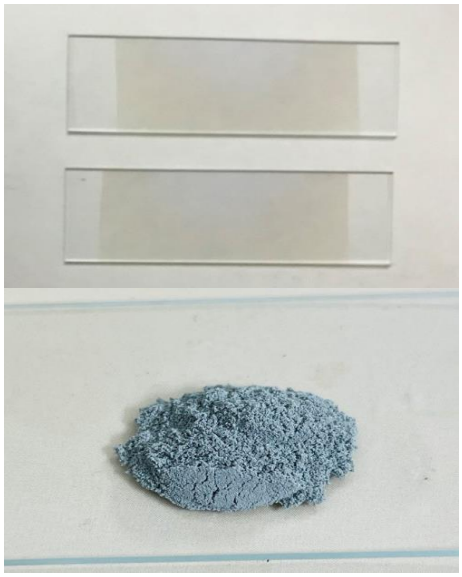


Fig. (2) The prepared samples (upper) SiC thin films, and (lower) extracted SiC nanopowder

3. Results and Discussion

Figure (3) shows the XRD pattern of final sample prepared after deposition time of 3 hours. Five most intense peaks are presumably observed on this pattern at 35.56°, 41.06°, 60.06°, 71.84° and 75.64° corresponding to lattice planes of (111), (200), (220), (311) and (222), respectively, according to JCPDS card no. 29-1129 [17].

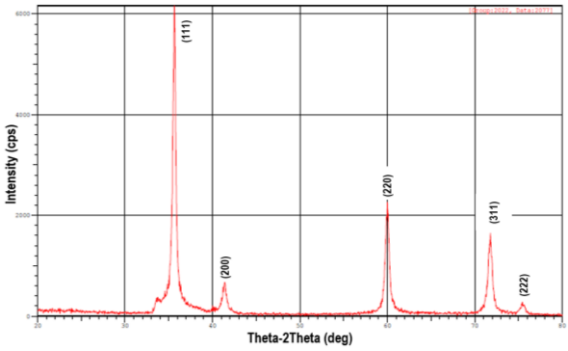


Fig. (3) The XRD pattern of SiC sample synthesized in this work

The diffraction pattern in Fig. (3) primarily confirms the production of SiC. Whereas each SiC molecule is a single-crystal grain that forms in a preferred crystallographic orientation, specifically, [111], since the (111) plane has the lowest surface energy of the SiC surface planes [18-21].

In general, the FTIR spectrum in Fig. (4) shows two peaks of Si-C bonds seen at 619 and 923 cm^{-1}, while the Si-O bond characteristic peaks are seen at 649 and 1045 cm^{-1}. Furthermore, the peaks observed at 1012, 1423 and 2400 cm^{-1} for the C-O bond adsorbed from the surrounding environment. Also, the distinctive peaks seen at 1573 and 3500 cm^{-1} are attributed to the stretching vibration bond of the O-H bonds. Depending on its surroundings, the prepared sample may differently adsorb water vapor or some gases [22-25].

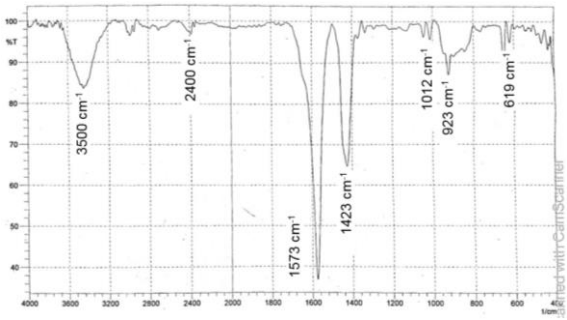


Fig. (4) The FTIR spectrum of the SiC sample synthesized in this work

Scanning electron microscopy (SEM) was used to introduce the surface morphology and determine the particle size of the deposited thin films, as shown in Fig. (5), which clearly shows that the particles are evenly dispersed, crack-free, homogenous in size,

and relatively tiny. The minimum particle size is about 47.44 nm. The EDX result and elemental analysis displayed in Fig. (6), validate the structural purity and stoichiometry of the synthesized SiC. Only peaks related to Si and C elements were observed. The origin of the oxygen peak is attributed to the fact that the prepared sample was exposed to the environment prior to the EDX test, and oxygen atoms adsorbed on SiC. As a result, the presence of such a trace quantity of oxygen in the final sample is unavoidable but ineffectual. The atomic ratio of [Si/C] obtained from EDX analysis, as shown in table (1), is around 1, indicating the formation of a stoichiometric SiC phase.

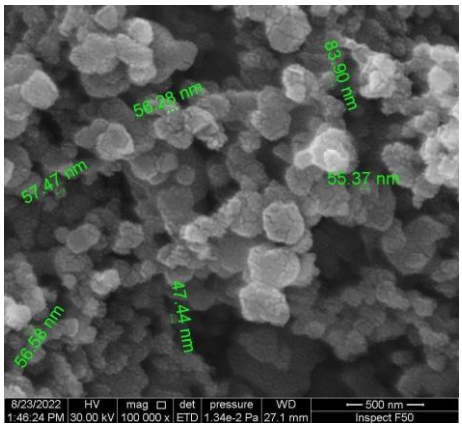
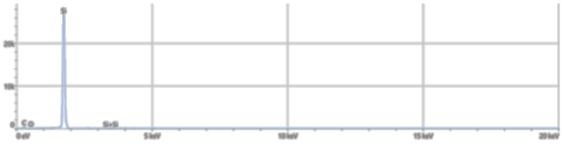


Fig. (5) SEM micrograph of the SiC layer grown in this work



Element	Weight%	Atomic %
C	27.8	46.8
O	2.2	2.8
Si	69.9	50.3

Fig. (6) EDX result and elemental constitution analysis of the SiC layer grown in this work

The UV-visible spectrophotometry was used to record the absorption spectra of carbon soot when deposited as thin film on silicon substrate with different exposure times as shown in Fig. (7) and notice that an increase in the absorption peaks as a function of the deposition time. This is due to the increase in the number of atoms sputter from the target as the deposition time increases, thus increasing the quantum yield and increasing the absorption peaks. The absorption is increased with increasing exposure time of laminar diffusion flames. Also; the absorption spectrum of SiC, as a final product (Fig. 8), shows a significant absorption around the wavelength of 400 nm, while the energy band gap was determined by using Tauc's formula [26]:

$$\alpha\, h\nu = A\left(h\nu - E_g\right)^{1/2} \quad (1)$$

where α is the absorption coefficient, h is Planck's constant, A is constant, and E_g is the energy band gap

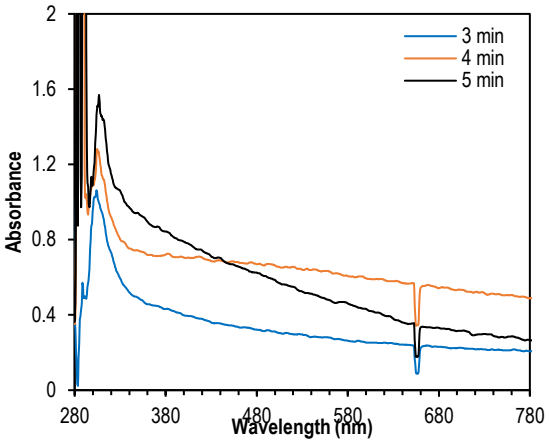


Fig. (7) Absorption spectra of the carbon soot layers prepared after different exposure times

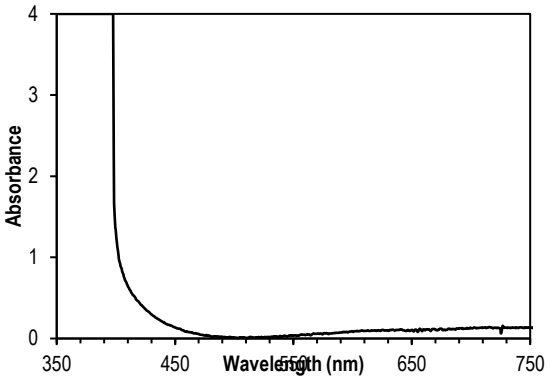


Fig. (8) Absorption spectrum of the SiC thin film sample prepared in this work

In Fig. (9), the optical energy band gap of SiC was determined to be 2.4 eV from the intercept of linear behavior of drawing the relationship between $(\alpha h\nu)^{1/2}$ and photon energy incident ($h\nu$) and its corresponds to the structural phase of SiC as a semi-conductor material.

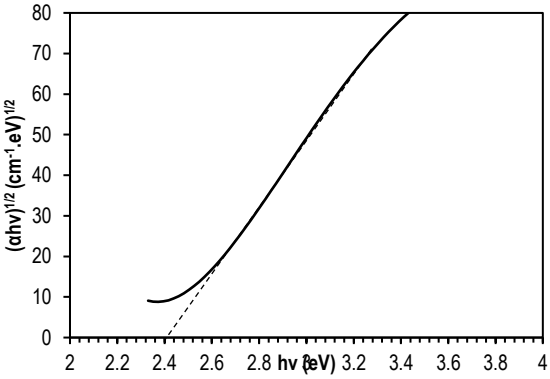


Fig. (9) Determination of optical energy band gap of SiC sample prepared in this work

4. Conclusion

In summary, the primary goal of this research is to provide a low-cost synthesis process to produce SiC nanostructures in two steps; first, production of carbon soot and deposit it on a silicon wafer, and second, using the carbon soot-coated silicon wafer is used a target in sputtering system to synthesize highly-homogenous nanostructured thin film samples of SiC, those finally were extracted as a black grey nano-powders.

References

[1] G. L. Harris, **"Properties of Silicon Carbide"**, INSPEC Publication (London, 1995).

[2] M. Mukherjee (ed.), **"Silicon Carbide - Materials, Processing and Applications in Electronic Devices"**, Intech Publications (Croatia, 2011).

[3] O.A. Hammadi, "Magnetically-supported electrically-induced formation of silicon carbide nanostructures on silicon substrate for optoelectronics applications", *Opt. Quant. Electron.*, 54 (2022) 427.

[4] G.L. Harris, **"Properties of Silicon Carbide"**, INSPEC (London, 1995), vol. 19, pp. 170-180.

[5] S. Somiya and Y. Inomata, **"Silicon Carbide Ceramics"**, Fundamental and Solid Reaction, Elsevier Science (Essex, 2012).

[6] O.A. Hammadi, Characterization of SiC/Si Heterojunction Fabricated by Plasma-Induced Growth of Nanostructured Silicon Carbide Layer on Silicon Surface, Iraqi J. Appl. Phys., 12(2) (2016) 9-13.

[7] D.A. Taher and M.A. Hameed, "Structural and Hardness Characteristics of Silicon Nitride Thin Films Deposited on Metallic Substrates by DC Reactive Sputtering Technique", *Silicon*, 15 (2023) 7855-7864.

[8] Y.R. Hathal et al., "Influence of DC Magnetron Sputtering Power on Structural, Topography, and Gas Sensor Properties of Nb_2O_5/Si Thin Films", *Iraqi J. Phys.*, 21(3) (2023) 41-54.

[9] K.A. Al-Hamdani, "Current–voltage and capacitance-voltage characteristics of Se/Si heterojunction prepared by DC planar magnetron sputtering technique", *Iraqi J. Phys.*, 8(13) (2010) 28-32.

[10] F.J. Kadhim and A.A. Anber, "Microhardness of Nanostructured Si_xN_{1-x} Thin Films Prepared by Reactive Magnetron Sputtering", *Iraqi J. Appl. Phys.*, 12(2) (2016) 15-19.

[11] O.A. Hammadi, "Plasma-Induced Growth of Silicon Carbide on Silicon Substrate for Heterojunction Fabrication", Iraqi J. Mater., 3(1) (2024) 19-24.

[12] K. Vaideki, S. Jayakumar and R. Rajendran, "Investigation on the effect of RF air plasma and neemleaf extract treatment on the surface modification and antimicrobial activity of cotton fabric", *Appl. Surf. Sci.*, 254 (2008) 2472-2478.

[13] C.H. Xue, R.L. Wang and J. Zhang, "Growth of ZnO nanorod forests and characterization of ZnO coated nylonfibers", *Mater. Lett.*, 64 (2010) 327-330.

[14] J. Du et al., "The effect of flame structure on soot particle inception in diffusion flames", *Combus. Flame*, 100(3) (1995) 367-375.

[15] Y. Wang and S.H. Chung, "Progress in Energy and Combustion Science Soot formation in laminar counter flow flames", *Prog. Ener. Combus. Sci.*, 74 (2019) 152-238.

[16] O.A. Hammadi, "Production of Nanopowders from Physical Vapor Deposited Films on Nonmetallic Substrates by Conjunctional Freezing-Assisted Ultrasonic Extraction Method", *Proc. IMechE, Part N, J. Nanomater. Nanoeng. Nanosys.*, 232(4) (2018) 135-140.

[17] L. Li et al., "Synthesis and characterization of 3C and 2H-SiC nanocrystals starting from SiO_2, C_2H_5OH and metallic Mg", *J. Alloys Comp.*, 484(1-2) (2009) 341-346.

[18] G.Y. Yang et al., "Direct observation of the growth process of silicon carbide nanowhiskers by vapor–solid process", *Physica E*, 39 (2007) 171-174.

[19] R.B. Wu et al., "Prism-shaped SiC nanowhiskers", *J. Alloys Comp.*, 453 (2008) 241-246.

[20] S. Dhage et al., "Formation of SiC nanowhiskers by carbothermic reduction of silica with activated carbon", *Mater. Lett.*, 63 (2009) 174-176.

[21] R. Dhiman, E. Johnson and P. Morgen, "Growth of SiC nanowhiskers from wooden precursors, separation, and characterization", *Ceram. Int.*, 37 (2011) 3759-3764.

[22] A. Popov, P. Savchyn and I. Karbovnyk, "Infrared Characterization of Silicon Carbide Nanowires", *Nanomater. Appl. Proper.*, 2 (2011) 134-136.

[23] A.B.D. Nandiyanto, R. Oktiani and R. Ragadhita, "How to Read and Interpret FTIR Spectroscope of Organic Material", *Indonesian J. Sci. Technol.*, 4(1) (2019) 97-118.

[24] E. Huseynov and A. Garibov, "Fourier transform infrared spectroscopic study of gamma irradiated SiO_2 nanoparticles", *Int. J. Mod. Phys. B*, 32(7) (2018) 1850074.

[25] Z.G. Sun et al., "Synthesis and microwave absorbing properties of SiC nanowires", *Appl. Phys. A*, 124 (2018) 802.

[26] Ł. Haryński et al., "A facile method for Tauc exponent and corresponding electronic transitions determination in semiconductors directly from UV–Vis spectroscopy data", *Opt. Mater.*, 127 (2022) 112205.

