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INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 10 **Issue:** V **Month of publication:** May 2022

DOI: https://doi.org/10.22214/ijraset.2022.42825

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International Journal for Research in Applied Science & Engineering Technology (IJRASET)
ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 7.538
Volume 10 Issue V May 2022- Available at www.ijraset.com

Synthesis of SiC films by Atmospheric Pressure Chemical Vapor Deposition using Dimethylisopropylsilane Polymeric Precursor

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Abstract: ***Silicon carbide (SiC) thin films were deposited on Quartz, SiC, and GaAs substrates under a hydrogen (H_2) ambient at 850°C using dimethylisopropylsilane [$CH(CH_3)_2SiH(CH_3)_2$] as a single molecular polymeric precursor by the atmospheric pressure chemical vapor deposition (APCVD) technique. Effects on the SiC films from variation of precursor to H_2 flow ratio in the reactive chamber as well as those which resulted when vacuum or positive pressures are utilized during temperature ramping have been investigated. Detailed analysis of Fourier Transform Infrared Spectroscopy (FTIR) results indicate the formation of SiC at 850°C in accordance with residual Si-CH_2, Si-CH_3 and Si-O bonds. Confirmed by either X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) or Energy Dispersive Spectroscopy (EDS), results show that the produced SiC films are dense, rough surfaced, void-free, and consist of a crystalline core showing the presence of beta-SiC and small amounts of alpha-SiC. Pre-experimental thermodynamic modeling provided the valuable insight that an increase of the precursor to direct H_2 flow ratio would provide a decrease of unreacted carbon in the SiC matrix yielding higher quality SiC films. These simulations also have shown reasonable agreement with our experimental observations.***

Keywords: SiC thin Film, Polymeric Precursor, Chemical Vapor Deposition, Thermodynamic Modelling

I. INTRODUCTION

Due to its wide energy band gap, extreme hardness, high thermal conductivity[10,11], and other promising semiconducing properties, SiC is one of the potential candidates for aerospace high temperature applications, and is currently being utilized in micro-electronic devices such as in SiC/Si heterojunction bipolar transistors, solar cells, photodiodes, and phototransistors. Several groups have successfully synthesized SiC films by a variety of techniques[15–25]. Conventionally, silicon carbide has been deposited by chemical vapor deposition using silane and various hydrocarbons. The main disadvantage of utilizing multiple precursors is the instability of the system's stoichiometric properties during deposition. It has been shown that a single precursor, such as what has been used for this work, allows for a more stable system and lower deposition temperatures which decrease tensile stresses as well as lattice defects in the film[1]. The focus for this work was not to find the lowest temperature capable of producing SiC, but to understand how different flow ratios utilized during deposition in conjunction with a set system pressure throughout the furnace ramping phases would reflect on the deposit. As a result, 850°C was chosen for the main furnace set point since SiC has previously been achieved at this particular temperature using $CH(CH_3)_2SiH(CH_3)_2$ as the single molecule precursor[12].

Understanding the influence of these experimental variables provides important information for future work involving temperature and pressure variations during deposition, gas dilution, and thick versus thin film comparisons. Holding constant the most promising flow ratio as well as furnace ramping procedures established by this work, new variables, stated previously, can then be adjusted for optimization.

II. EXPERIMENT

SiC films were deposited in a hydrogen atmosphere APCVD system at 850°C over times ranging from 10 to 90 minutes. Polished GaAs was utilized as the main substrate for deposition due to its ability provide good quality XRD, SEM, and EDS results as well as its capability to transmit light in the infrared region where a typical SiC FTIR wavenumber peak appears (800 cm^{-1})[1,4,5]. SiC samples were also used as substrates during certain experiments and were only tested under SEM. Fused quartz was considered as the main substrate, however, due to its absorption of larger wavelengths above 5μm (this information was provided by the Momentive Performance Materials Company), this material would remove any possibility of an FTIR result for the SiC deposit. Prior to deposition, the GaAs substrates were run through a Xylene, Acetone, and Methanol (XAM) cleaning process to remove any surface contamination.

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This sample along with a piece of SiC substrate would be loaded into the CVD system, shown in Figure 1, and each end of the reactor tube would be sealed. Using the output bubbler as a flow rate estimation tool, ratios of either 1:10, 1:40, or 1:100 would be established simply by counting bubbles over time. Three cycles of evacuation (using a mechanical vacuum pump) and filling of the chamber with H_2 would follow to remove any residual material left inside the system. Two methods were then employed during the furnace ramping phases which took up to three hours each. Either the chamber would be evacuated and held under vacuum, or a constant flow of H_2 would be passed through the system creating a net positive pressure inside the chamber. An FTIR result was immediately obtained on each sample once removed and its percent transmittance spectra would be analyzed for confirmation that the desired wavenumber had in fact been achieved. SEM, XRD, and EDS were utilized on most promising samples or when further investigation was necessary.

III. RESULTS AND DISCUSSION

For the sake of clarity, this section has been divided into four parts. The first deals with simulation results obtained from a chemical reaction simulator called FactSage prior to any experimentation taking place, the second section will show results from a 1:10 flow ratio of hydrogen through the precursor to direct hydrogen into the system, the third section will discuss results from a 1:40 flow ratio as well as a two 1:100 tests, and the final section will provide a brief comparison of EDS results to the initial simulations.

A. Pre-Experimental FactSage Simulation Results

When utilizing the FactSage simulator, specific attention was placed with the Equilib module associated with the tool. This module calculates the concentrations of various product species from specified reactants when they reach a state of equilibrium. Since the main focus was to determine how different flow ratios affect the deposited SiC films, the Equilib module provided the opportunity to examine what theoretically would be produced from one mol of precursor as the mol number of H_2 was varied (this represents a change in flow ratio). The result taken at 1100K (827°C), shown in Figure 2, represents how many moles of SiC and carbon would deposit with an increase in H_2. It can be seen that for all H_2 quantities used, one mole of precursor will yield one mol of SiC, however, at low flow rates the amount of undesired carbon deposit swells to greater values. Due to these results, ratios of 1:10, 1:40, and 1:100 were chosen for the direct purpose of comparing experimentation outcomes to those found under simulation.

B. 1:10 Flow Rate

Experimental results were first obtained after ramping the furnace under evacuation conditions. Figure 3 represents FTIR measurements from two separate deposition runs of 25, and 10 minutes in length. In each, it is evident that the desired SiC peak has been achieved at 800 cm^{-1}, however, a 1050 cm^{-1} peak has resulted during the 25 minute experiment whereas peak locations of 1050 and 1200 cm^{-1} are present in the 10 minute outcome. It has been shown that formations at 1050 cm^{-1} represent either wagging Si-CH_2[1,4,5] or Si-O[11] bonds, while a peak at 1200 cm^{-1} represents bending Si-CH_3[1,4,5]. Figure 4 displays an average XRD measurement using samples from those experiments which produced the FTIR images of Figure 3. Figure 4 confirms that SiC has been deposited, and also shows the material's crystalline structure holds both alpha and beta phases on the GaAs substrates[2,3]. SEM, Figure 5, determined these films contain a rough surface quality during initial deposit stages.

Figure 3 introduced the possibility that residual oxygen still remained inside the chamber once the flush sequence was complete. To understand if this was the case, a 30 minute deposition run was completed with a constant flow of H_2 during the temperature ramping phases. Allowing for a constant positive pressure to exist inside the system resulted in a continuous flushing of any undesired material prior to deposition as well as after its conclusion. Figure 6 represents the FTIR measurement of this run which shows a drastic decrease in the undesired peaks at both 1050 and 1200cm^{-1}. A close examination does demonstrate that there may still be some undesired Si-CH_3 bonds due to the small peak position occurring close to 1200 cm^{-1}. These results are consistent with the FactSage simulations which stated that an undesired amount of carbon should still remain in the film.

C. 1:40 Flow Rate

Similar in procedure, evacuation conditions were first applied during temperature ramping. Figure 7 represents FTIR results from two separate experiments of 25 and 10 minutes. In each measurement it is once again evident that the desired SiC peak of 800 cm^{-1} had been achieved. However, in each case, peaks at 1050 cm^{-1} as well as 1200 cm^{-1} have formed once again. Due to possible residual oxygen inside the chamber, a 20 minute run was completed under a constant flow of H_2 during the temperature ramping phases. Figure 8 represents the FTIR result of this experiment which shows the undesired peaks have been completely eliminated. Having achieved such a result, a 100 µm thick film was grown on both GaAs and SiC substrates for comparison purposes. FTIR for these films, shown in figure 9, has also confirmed the 1050 and 1200 cm^{-1} undesired peaks are not present.

SEM images obtained on the SiC substrate, Figure 10, indicate the new deposit is still rough in nature. This proves, in accordance with pre-experimental simulations, that an increase in flow ratio does in fact decrease the amount of excess carbon remaining in the film. Unfortunately, results from two later experiments, which were done at a ratio of 1:100 utilizing the same H_2 flow during temperature ramping, FTIR shown in Figure 11, resulted in a re-occurrence of the Si-CH_2 bond. Simply stated, this means the initial simulations are not accurate at high gas flow ratios.

D. EDS Comparison of 1:10 and 1:40 Flow Ratio

Figure 12 represents the average EDS measurements for the 1:10 and 1:40 ratio test results. It must be stated that the SiC samples chosen for EDS were obtained utilizing the evacuation temperature ramping technique as opposed to positive pressure. It is believed, due to FTIR comparisons, that the 1050 cm^{-1} peak shown for depositions done under this ramping procedure is in fact the Si-O bond due to residual oxygen in the chamber, which was again concluded from EDS. What is important to note about these results is the drastic decrease in carbon deposit with the increased flow ratio. This confirms that the pre-experimental simulations are in fact accurate for low gas flow rates and can be utilized to determine starting points for testing.

IV. CONCLUSION

This work reports the growth of SiC films utilizing dimethylisopropylsilane as the source by APCVD technique. Films deposited at 850°C where temperature ramping was completed under an evacuated chamber resulted in undesired Si-CH_3, Si-CH_2, and Si-O bonds. With an increase in the precursor to H_2 ratio as well as a change to constant H_2 flow during temperature ramping, these undesired bonds were completely removed from the SiC films. FTIR results as well as EDS measurements confirmed that FactSage experimental simulations have the ability to provide accurate estimations for SiC and carbon deposit. Utilizing this tool, future simulations can be run on new, untested precursor materials in order to determine if they are worth creating for full scale experimentation.

V. ACKNOWLEDGMENTS

The author would like to thank Partha Pratim Ray for XRD equipment access and would also like to acknowledge the financial support provided by DST,GoI.

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FIGURES

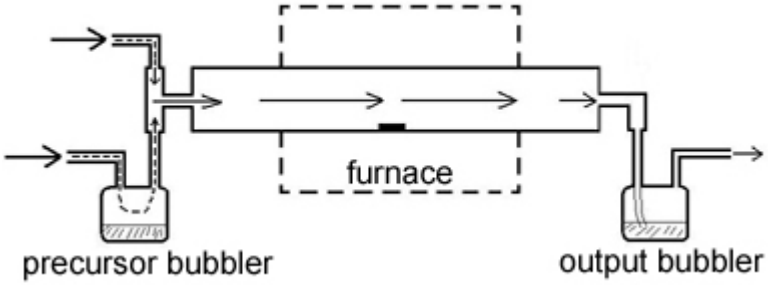


Figure 1

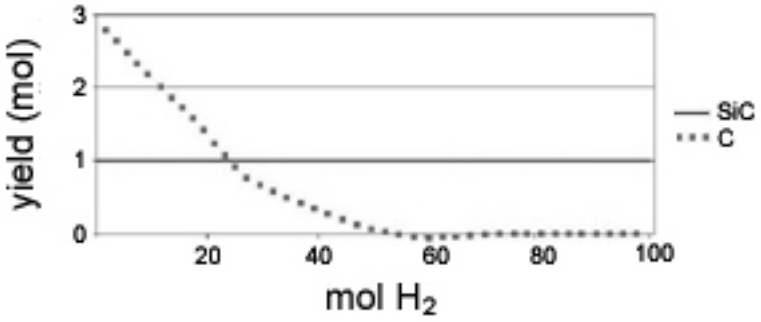


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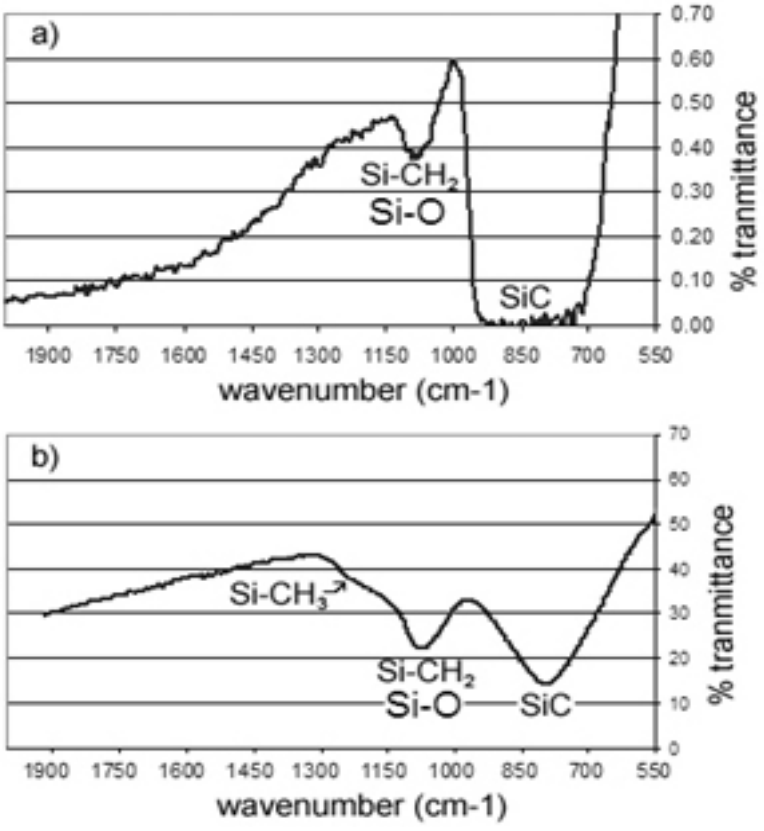


Figure 3

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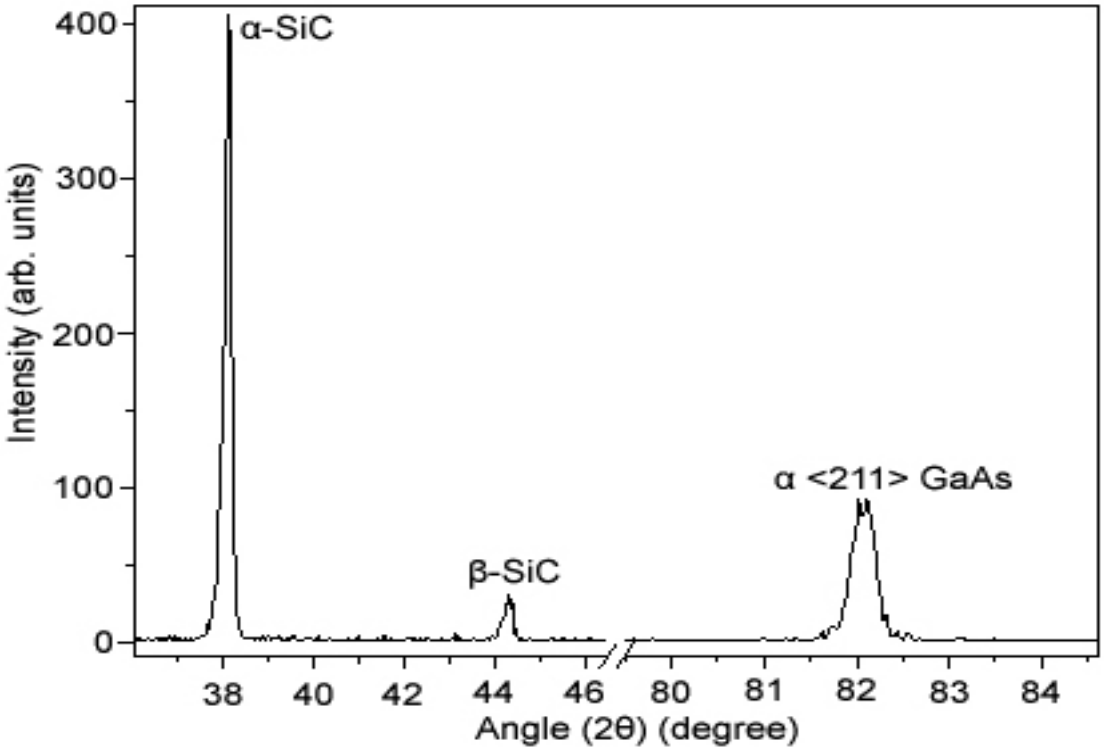


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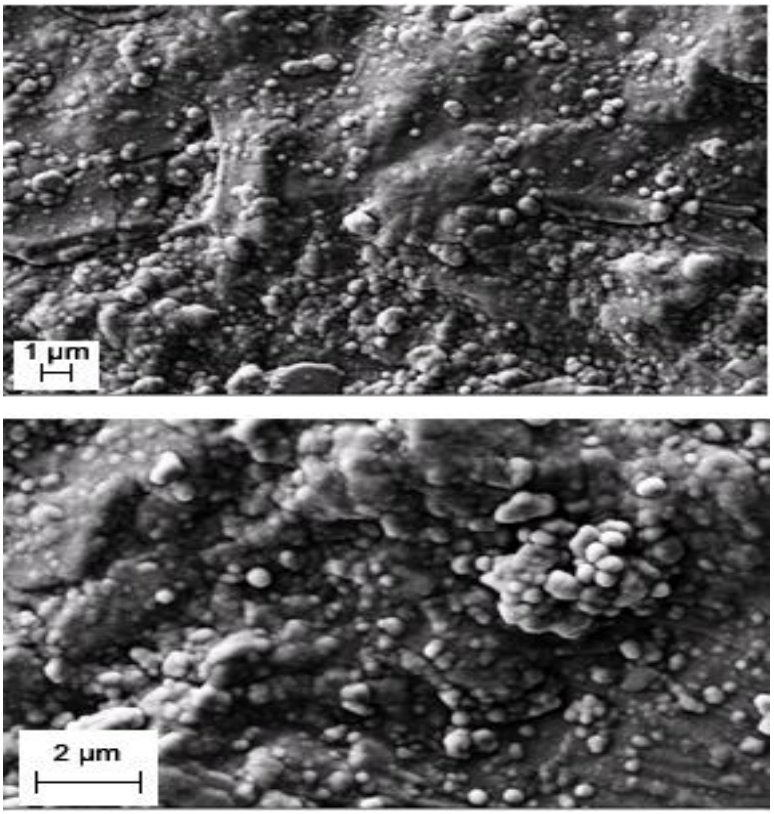


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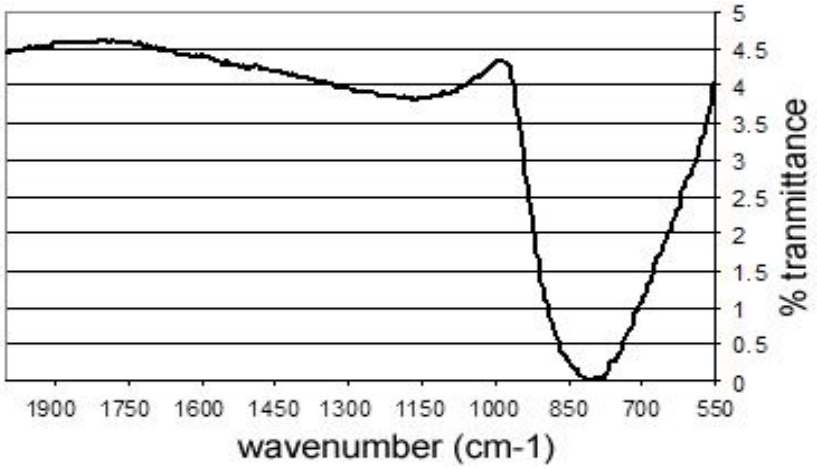
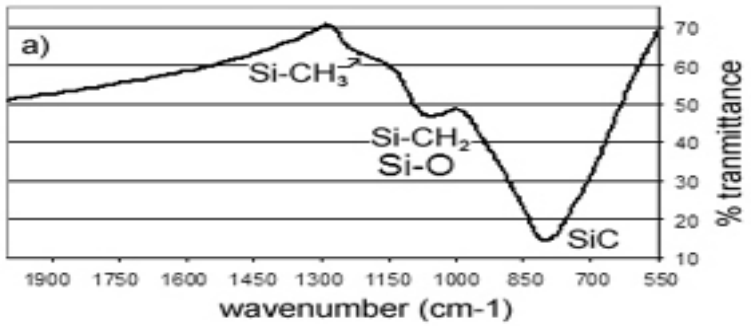


Figure 6



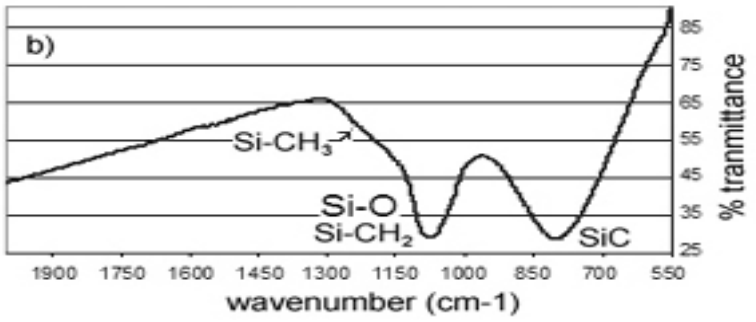


Figure 7

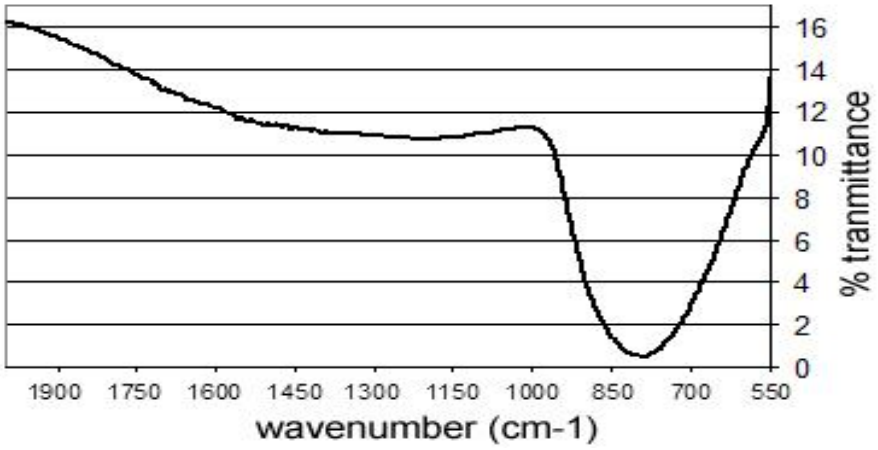


Figure 8

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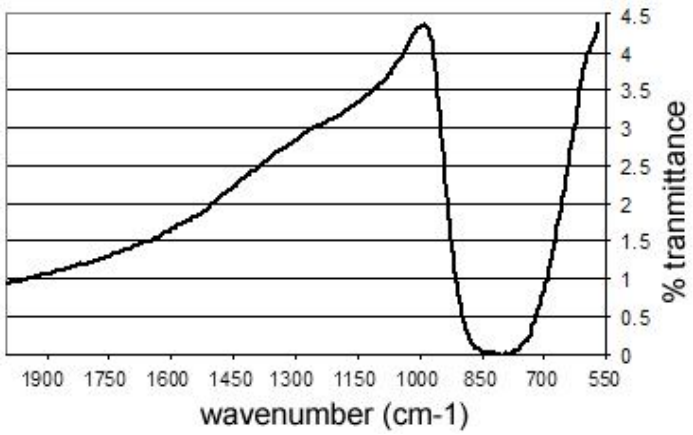


Figure 9

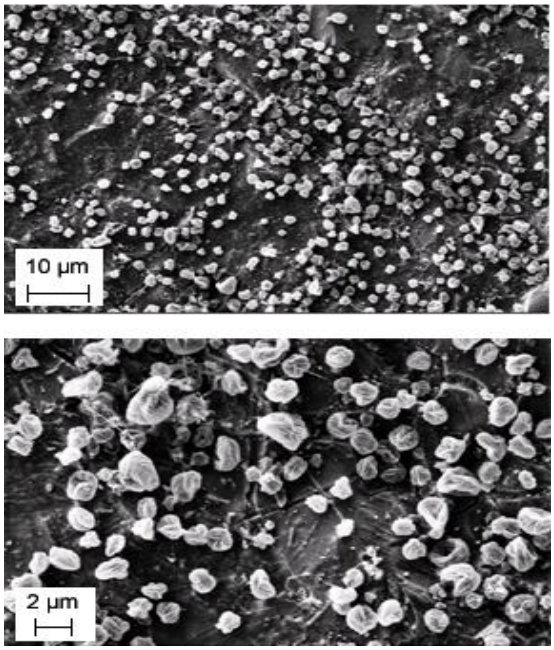


Figure 10

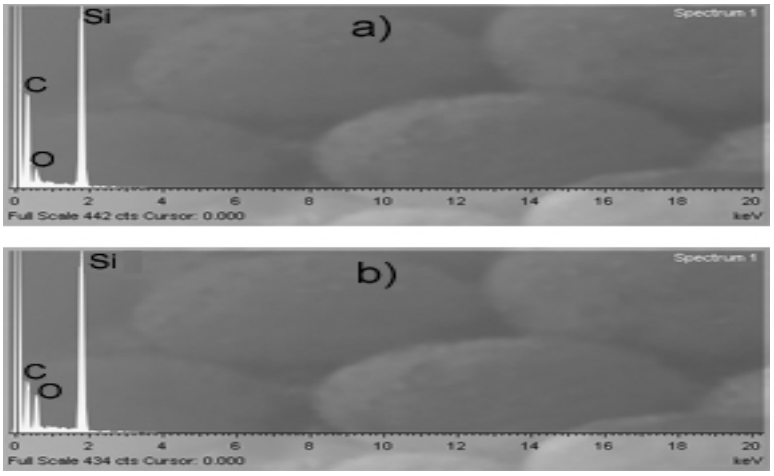


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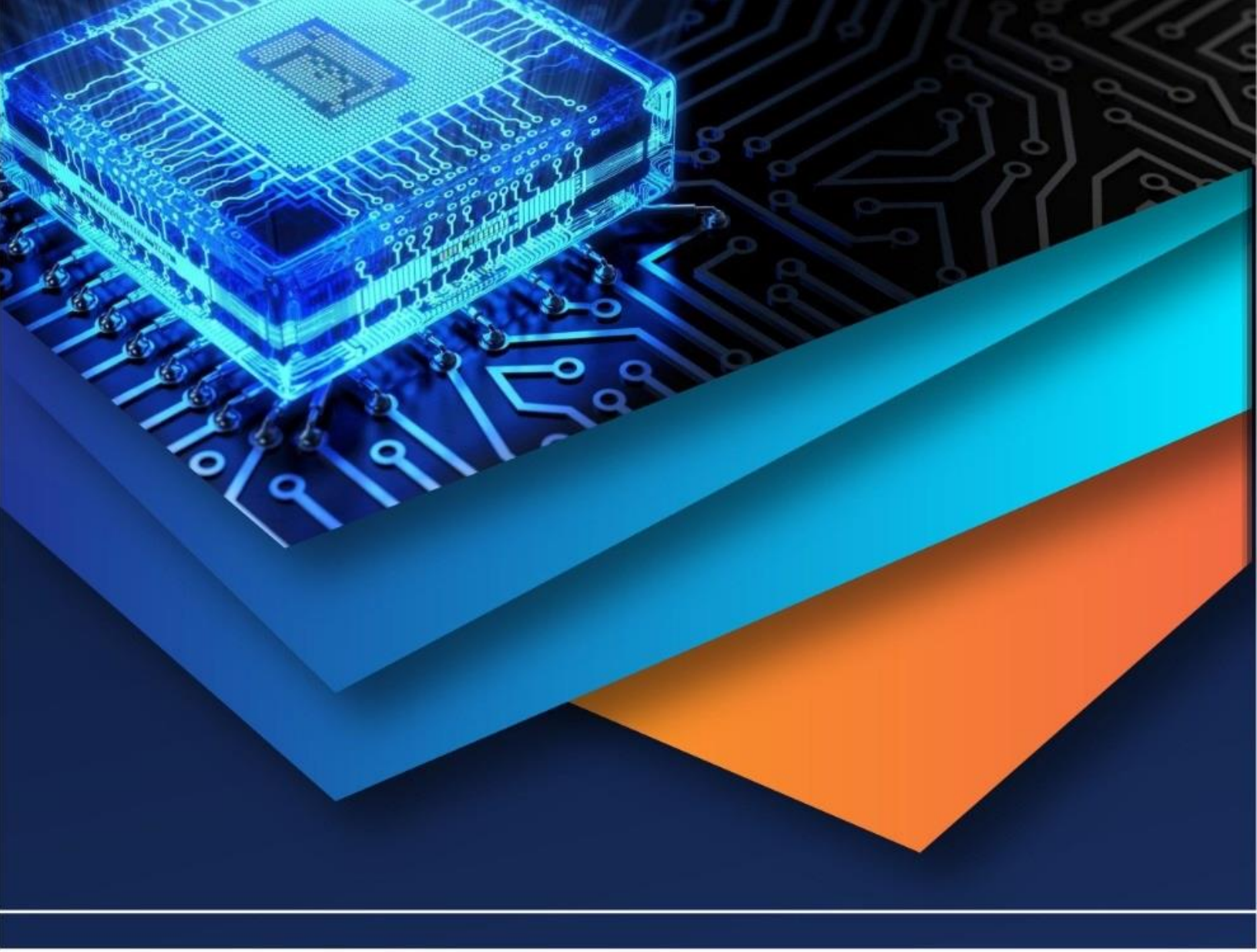
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FIGURE CAPTIONS

1) Schematic drawing of CVD reactor setup utilized for this work.
2) FactSage simulator results showing mol yield of SiC and carbon (C) with an increase in hydrogen mol content during reaction.
3) FTIR results obtained from SiC deposition runs of a) 25 minutes and b) 10 minutes in length utilizing a 1:10 flow ratio and GaAs substrate. Each experiment employed the evacuation method during temperature ramping.
4) Average XRD result obtained from SiC deposited with a 1:10 ratio at 25 and 10 minutes in length on a GaAs substrate.
5) SEM results at two magnifications of SiC deposit at the 1:10 ratio on a SiC substrate.
6) FTIR result obtained from a 1:10 ratio SiC deposition run of 30 minutes on a GaAs substrate with a constant flow of H_2 during temperature ramping.
7) FTIR results obtained from SiC deposition runs of a) 25 minutes and b) 10 minutes in length utilizing a 1:40 flow ratio and GaAs substrate. Each experiment employed the evacuation method during temperature ramping
8) FTIR result obtained from a 1:40 ratio SiC deposition run of 20 minutes on a GaAs substrate with a constant flow of H_2 during temperature ramping.
9) FTIR result obtained from a 1:40 ratio SiC deposition of 100 μm thickness on a GaAs substrate
10) SEM results at two magnifications of a 100 μm SiC deposit at the 1:40 ratio on a SiC substrate.
11) Average EDS measurements for the 1:10 and 1:40 ratio test results.

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