



Hot wire chemical vapour deposition (HWCVD) of boron carbide thin films from ortho-carborane for neutron detection application

Pradip Chaudhari^a, Nagsen Meshram^a, Arvind Singh^b, Anita Topkar^b, Rajiv Dusane^{a,*}

^a Department of Metallurgical Engineering and Materials Science, Indian Institute of Technology Bombay, Powai, Mumbai, 400 076, India

^b Electronics Division, Bhabha Atomic Research Centre, Trombay, Mumbai, 400 085, India

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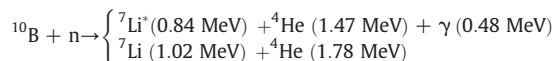
ABSTRACT

Detection of neutrons is possible if suitable converters such as Li, LiF or ¹⁰B in the form of thin films are used along with the semiconductor device. The use of boron (¹⁰B) in some host matrix as a neutron detector is attractive due to its large neutron capture cross-section. Boron carbide (BC) films are deposited on silicon substrates by HWCVD technique using solid ortho-carborane (o-C₂B₁₀H₁₂) precursor with argon as carrier gas. The films contain ¹⁰B required for neutron detection as confirmed by the Secondary Ion Mass Spectroscopy. Variations in its structure as well as the chemical bonding configurations using Fourier Transform Infra-Red, Raman and X-ray diffraction spectroscopy have been studied.

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1. Introduction

Boron carbide is attractive as a non-metallic material with useful physical and chemical properties. It is an extremely hard and wear-resistant material with a high melting point, low density and good thermal and electrical properties. The large resistance to chemical agents and very high neutron absorption/capture cross-section makes boron carbide (BC) a strong candidate for high technology industries, fast-breeders and light weight armours application [1]. When used for thermal neutron detection the common practice is to deposit the BC layer on a Si p-i-n device which is responsible for generating the electrical signal corresponding to the passage of the α-particle which results due to the reaction of the neutron with the Boron of the BC layer. The reaction is based on the ¹⁰B(n, α)⁷Li neutron capture reactions [2] and written as:



It is very important that the process of depositing the BC layer does not in any way affect the electrical characteristics of the underlying device.

Of the different types of CVD processes, Plasma Enhanced Chemical Vapour Deposition (PECVD) has been considered as the powerhouse of semiconductor industry up till now [3]. PECVD, however, suffers from a serious drawback of low energy electron/ion bombardment on the sample surface and charge induced field

related damage to the underlying devices. The Hot-Wire Chemical Vapour Deposition (HWCVD) technique, an emerging technique overcomes these problems faced in PECVD. The first report of the HWCVD technique was made in 1979 [4] and later in the year 1986 [5] and 1988 [6] after which it gained importance in the deposition of silicon and carbon based alloy films. The exhaustive work carried out over the last two decades has shown that the HWCVD has all the potential for deposition of amorphous, polycrystalline, microcrystalline silicon, silicon carbon and silicon germanium alloys and silicon nitride with similar properties as those made by PECVD. In some cases, it is even better than the PECVD process [7–10]. It is an amazing technique due to its design simplicity, high deposition rate, lower ownership cost and low temperature thin film deposition.

Till today, PECVD has been used to deposit BC thin films using the solid precursor ortho-carborane (o-C₂B₁₀H₁₂) [3,11]. There are some reports of using the HWCVD to deposit the BC layers but with other gaseous precursors like BCl₃, CH₄, C₇H₈ and H₂ [12,13]. The HWCVD boron carbide film using the solid precursor o-C₂B₁₀H₁₂ has been reported only once [14] in the year 1992. In the last 18–20 years the HWCVD BC using ortho-carborane was not widely adopted or studied. Here, in the present work we have used HWCVD for deposition of boron carbide thin films using o-C₂B₁₀H₁₂ with argon as the carrier gas.

2. Experimental details

The Hot-Wire Chemical Vapor Deposition (HWCVD) is a deposition technique that employs thermal decomposition of a precursor gas at a resistively heated filament (usually tungsten or tantalum) to obtain the required material to be deposited on a substrate. Fig. 1 shows the general setup of the HWCVD tool in our laboratory tuned

* Corresponding author.

E-mail address: rodusane@iitb.ac.in (R. Dusane).

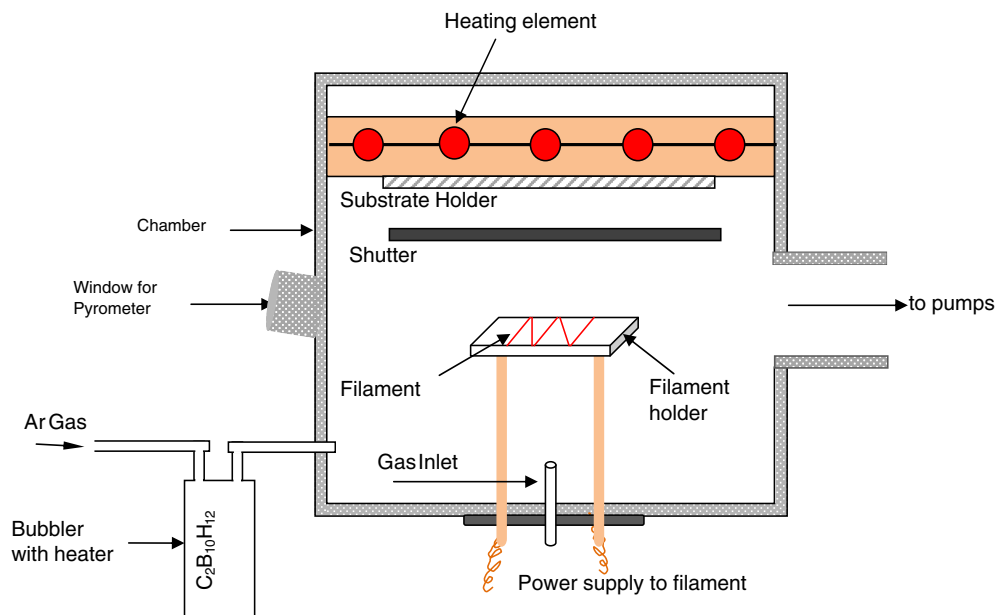


Fig. 1. Schematic of hot-wire chemical vapour deposition chamber used for the boron carbide film deposition.

for the deposition of the BC layers using ortho-carborane. The substrate holder is heated with an external heater with a PID controller. The filament array is located at about 7 cm from the substrate. The gas flow is perpendicular to the plane of the filaments. As the precursor gas comes in contact with the filaments it undergoes thermal dissociation leading to the generation of depositing radicals leading to film formation on the substrates. The precursor is a large molecule and should have a complex dissociation and recombination chemistry within the gas phase. This implies that the filament to substrate distance should play a significant role in the chemical and structural characteristics of the film. Also the vapour pressure of the precursor is highly sensitive to the temperature of the vapour delivery system and hence the flow of the carrier gas should become an important parameter.

Boron carbide films were deposited on n-type silicon and glass Corning (eagle) substrates by HWCVD using ortho-carborane ($\text{o-C}_2\text{B}_{10}\text{H}_{12}$) precursor. The n-type silicon wafer was cleaned by DI water and methanol with ultrasonic cleaning. The solid ortho-carborane precursor is sublimed in the bubbler at 70–80 °C. The vapors of the precursor are then introduced into the chamber using argon as carrier gas. The films were deposited at pressures in the range 100–200 mTorr at substrate temperatures between 200 and 300 °C and the filament temperature was varied from 1500 to 2000 °C. BC films were deposited and characterized by Fourier-transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD), Raman spectroscopy and Secondary ion mass spectroscopy (SIMS).

3. Results and discussion

The transmission spectra of the boron carbide films deposited at argon flows 5 and 10 sccm by HWCVD using ortho-carborane were measured with a FTIR spectrometer (JASCO FT/IR-6100) in the spectral range of 4000 to 400 cm^{-1} with a resolution of 4 cm^{-1} is shown in Fig. 2. As observed in the literature [15], three elemental bonds B–H, C–H_n and B–C are identified.

The two bands at 3060 cm^{-1} and 2580 cm^{-1} are attributed to the stretching vibrations of the C–H and B–H bonds respectively revealing the presence of hydrogen bonding in the film. The corresponding bending mode of C–H_n appears at 1360 cm^{-1} . The other prominent peak which appears at 1100 cm^{-1} is attributed to vibrations associated with the B–C bond. The strength and narrow linewidth of the peaks is sufficient to make us believe that the band is a local mode

[15]. Werheit et al. [16] have studied the IR phonon spectra of ^{10}B and ^{11}B enriched boron carbide and the peaks are observed at 427, 510, 537, 668, 715, 789, 859, 866, 992, 1105 and 1624 cm^{-1} corresponding to $^{10}\text{B}_{4.3}\text{C}$ and $^{10}\text{B}_{6.5}\text{C}$ [16]. The presence of bands at 431, 513, 670, 786, 994, 1105 and 1625 cm^{-1} in our FTIR spectra indicates the presence of ^{10}B abundance in the deposited BC films. Surprisingly, the film deposited with 5 sccm of Ar and at a pressure of 140 mTorr has a higher intensity of the BC mode as compared to the one deposited with 10 sccm of Ar and at a pressure of 215 mTorr. This could be due to the difference in the residence time and subsequent gas phase reaction under these two different deposition conditions. Secondary gas phase reactions could get severely affected under different pressure conditions leading to such an observation. Thus it seems that a lower Argon flow rate would be beneficial for the BC films.

Fig. 3 shows the Raman spectra of the HWCVD BC recorded on JOBIN YVON-HORIBO-HR 800 UV spectrophotometer. A prominent peak at 1095 cm^{-1} is observed in the spectrum which relates to the phase of boron carbide. Earlier work on Raman scattering studies of BC films have shown that the peaks at 270, 320, 430, 481, 489, 534, 551, 560, 606, 656, 710, 815, 990, 1060, 1100 and 1108 cm^{-1} indicate the

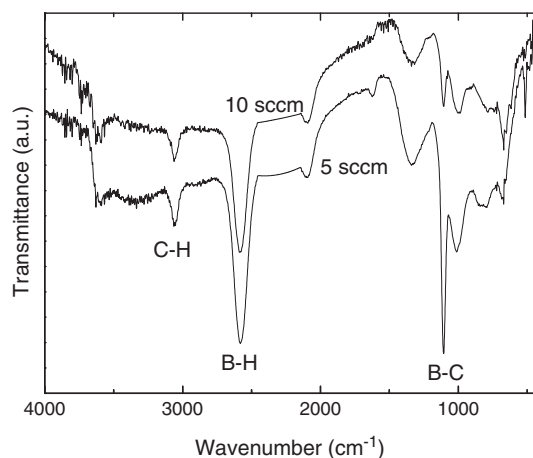


Fig. 2. Infrared spectra of HWCVD boron carbide films deposited using ortho-carborane with argon as carrier gas having flow rates of 5 and 10 sccm.

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