

# $^{nat}$ Boron Carbide ( $^{nat}$ B<sub>x</sub>C)/n-Si Portable Solid State Thermal Neutron Detector

- A. Bute & N. Maiti

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## 29.1 Introduction to Thermal Neutron Detectors

### 29.1.1 General Outline

Thermal neutrons are neutral particles and have very low velocity, they are unable to produce ionization directly. They can only be detected subsequent to a nuclear reaction with target atoms which produce ionizing radiation or ionizing particles. The target atoms, also called as neutron converter materials, should have large thermal neutron cross section  $\Sigma_t$  which increases their interaction probability with the incoming thermal neutrons. Thermal neutron detectors can be divided in three main categories:

- i. gas-based detectors,
- ii. scintillation detectors and
- iii. solid state (semiconductor) detectors.

The gas-based detectors utilize  $^3\text{He}$  and  $\text{BF}_3$  gases and they provide highest neutron detection efficiency but  $^3\text{He}$  is scarce, therefore extremely costly, and  $\text{BF}_3$  is toxic. They require high voltage for operation and on top of that, they are bulky and therefore lack mobility. Thus, researchers across the globe are currently working on developing suitable compact, cost effective semiconductor neutron detectors which will require low operating voltage and will provide portability with reasonable neutron sensitivity.

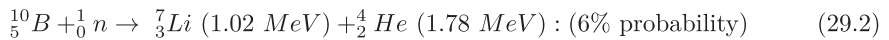
### 29.1.2 Semiconductor based detectors

There are only four potential neutron converter atoms suitable for fabrication of semiconductor based neutron detectors:  $^6\text{Li}$ ,  $^{10}\text{B}$ ,  $^{113}\text{Cd}$  and  $^{157}\text{Gd}$ ; having thermal neutron cross

sections 970 b, 3840 b, 20,000 b, and 1,50,000 b (b, barn & 1 barn =  $1.0 \times 10^{-24} \text{ cm}^2$ ) respectively. Li based semiconductor materials such as LiInSe, and LiZnP exist but these materials are very difficult to reliably fabricate them into devices. Gadolinium based neutron detectors are not particularly stable and produces low energy  $\gamma$ -rays and  $\beta$  particles which are difficult to distinguish from the  $\gamma$ -ray background of the neutron source. Cadmium zinc telluride has been shown to have good thermal neutron detection but it also has a problem of the  $\gamma$ -ray discrimination.  $^{10}\text{B}$  containing thin-films can be used for new generation neutron detection and is free from all the aforementioned constraints. Essentially four types of semiconductor diode structures are available: Planar, Pillar or trench type diodes, Si-diode with  $^{10}\text{B}$  as dopant, nanotubes loaded inside with neutron converters. In planar diodes, a thin neutron converter layer is used on top or bottom of a usual semiconductor diode. Converter materials such as  $^{10}\text{B}$  are also deposited in nano-scale structure, having high aspect ratio via reactive ion etching (RIE) technique. These diodes are reported to be efficient than planar converter diodes but pillar or trench masked diodes are found to be better in terms of dopant concentration. Gas filled neutron proportional counters are celebrated for their high detection efficiency. Semiconductor based thermal neutron detectors are gaining popularity for their low power consumption, compactness, low production cost and portability in spite of their low thermal neutron detection efficiency as compared to the gas filled detectors.

### 29.1.3 Boron carbide based neutron detectors

A  $^{10}\text{B}$  atom enables detection of thermal neutrons via production of  $\alpha$  particles through neutron capture reaction. Following are the neutron capture reactions:



$\text{B}_4\text{C}$  thin films have been reported to be investigated as neutron conversion layer in traditional proportional counters, scintillators and diode-based detectors, or it can be used as the p-type or n-type material itself in the semiconducting diode neutron detectors. In the lab work is being done on fabrication and characterization of  $^{nat}\text{Boron}$  carbide thin films ( $^{nat}\text{B}_x\text{C}$ ) deposited on high resistivity n-Si (100) substrates for neutron detection application in a suitable diode configuration. The  $^{nat}\text{B}_x\text{C}$  thin films have the natural abundance of boron that is 19.8% of  $^{10}\text{B}$  atoms and 80.2% of  $^{11}\text{B}$  atoms. Plasma Enhanced Chemical Vapour Deposition (PECVD) technique have been utilized for the deposition of the films. The stoichiometry of  $^{nat}\text{B}_x\text{C}$  thin films varies notably with various deposition techniques, deposition process parameters, and the precursors used for deposition [280]. The detection efficiency of a  $^{nat}\text{Boron}$  carbide/ n-Si hetero-junction based detector mainly depends on the percentage of  $^{10}\text{B}$  atoms, depletion depth, junction capacitance, and reverse saturation current. A good semiconductor neutron detector should be able to provide a reasonable detection efficiency,  $\gamma$ -ray discrimination and longevity. In the experiment varied the substrate “self-bias” (discussed in section 29.2.2) and studied its effect on the composition of the  $^{nat}\text{Boron}$  carbide thin films and neutron absorption cross section. In addition, all the properties like band-gap, adhesion, crystallinity, hardness etc. have been studied to assess the functionality of the films.

## 29.2 Deposition Process of $^{nat}\text{B}_x\text{C}$ Thin Films

### 29.2.1 Plasma enhanced chemical vapor deposition

The capacitively coupled RF-PECVD deposition chamber is made of a double walled cylindrical stainless steel (316) plasma CVD reactor, connected to a vacuum pumping station,

having a rotary vane pump and an oil-diffusion pump. With this pumping assembly, the chamber base pressure is first brought down to  $5 \times 10^{-5}$  mbar to get rid of the residual gases and moisture. Actual deposition experiments are carried out at rotary vacuum  $\sim 10^{-2}$  mbar. Inside the chamber there is a stainless-steel substrate holder. A RF power generator of frequency 13.56 MHz is connected to the substrate holder through an automated impedance matching network, with the help of an RG 213 cable. In this configuration, the substrate holder itself acts as the powered electrode. During deposition, depending upon the plasma conditions, impedance of the substrate holder-plasma system can change. The role of the au-

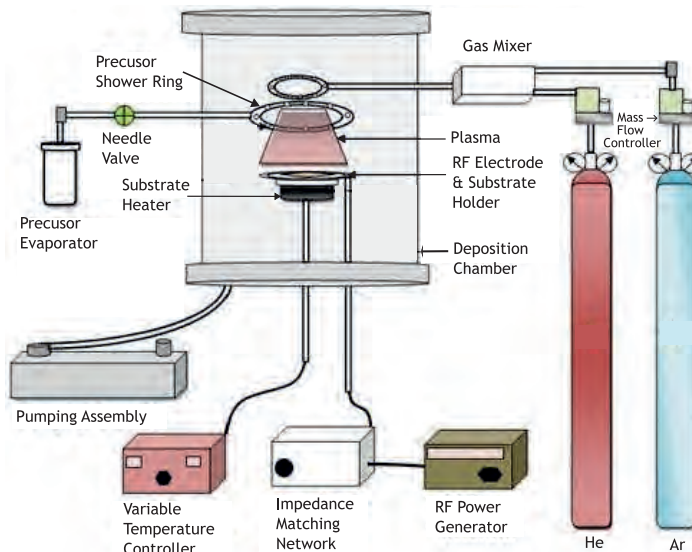


Figure 29.1: Simplified schematic of the RF-PECVD experimental arrangement.

tomated impedance matching network then is to act instantaneously to match the impedance of the RF power supply with plasma system so that maximum power transfer can take place, ensuring stable plasma generation and energy efficient deposition. During the experiments the dc “self-bias” developed on the powered electrodes were varied to find its effect on the thin film properties.

### 29.2.2 Development of dc self-bias in capacitively coupled RF-PECVD system

In a capacitively coupled RF discharge, the negative dc “self-bias” is generated in the RF-powered electrode as result of different mobility of electrons and ions in the plasma. For ease of discussion, the schematic of a typical capacitively coupled RF-PECVD system, given in Fig. 29.2 will be considered: Let us consider one full cycle of the applied RF signal at any instant. In the positive half cycle, the powered electrode is positive, therefore electron current reach the powered electrode, eventually the capacitor C, charging it negatively. In the next half cycle, the powered electrode becomes negative, the capacitor then gets charged up by ion current and the amount of negative charge stored in the capacitor is reduced as the ions neutralize some of the available electrons. Since electrons are more mobile than ions, number of electrons reaching the capacitor will be more than the ions after a complete cycle of the applied RF signal. As a net effect, some amount of negative charge will remain stored in the capacitor after completion of one full RF cycle. Over few more cycles, a time averaged

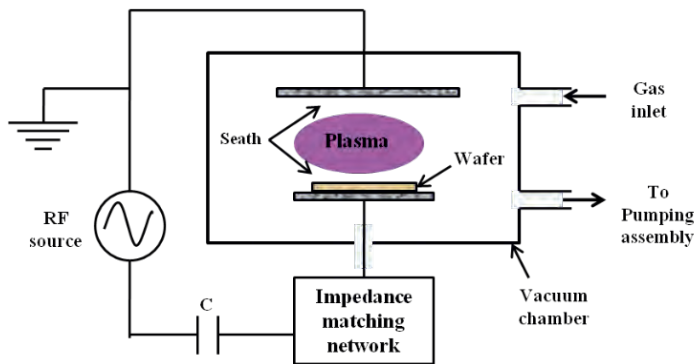


Figure 29.2: Schematic of a typical capacitively coupled RF-PECVD system.

negative DC potential will develop in the powered electrode called “self-bias”. All these phenomena take place within a very small-time scale ( $\sim$ ns). Development of DC “self-bias” requires two conditions to be satisfied:

- (i) the electrodes must have different surface area and
- (ii) a blocking capacitor should be present in the circuit. Role of this “self-bias” is to prevent further loss of electrons from the plasma and help maintaining “quasi-neutrality”.

“Self-bias” is an important feature of the RF-CVD plasma and plays a pivotal role in material processing via etching and deposition. Self-bias controls both the incoming ion flux, and ion energy impinging on the powered electrode.

## 29.3 Important Characterization Techniques for Performance

<sup>nat</sup>Boron carbide thin films deposited by PECVD technique were characterized for measurement of composition, structure, thickness, adhesion, hardness, and stress. The film composition was found to vary with substrate “self-bias” during deposition. This negative “self-bias” controls both the ion flux and ion impinging energy on the surface of the growing film during deposition and in turn controls the film structure, composition, density, hardness and several associated film properties. The boron to carbon atomic ratio in this <sup>nat</sup>Boron carbide ( $^{nat}B_xC$ ) thin films decreases with substrate self-bias as shown in the Fig. 29.3). Total macroscopic cross section ( $\Sigma_t$  in  $\text{cm}^{-1}$  of the <sup>nat</sup>Boron carbide thin films was calculated from neutron transmission measurement results. The variation in total macroscopic cross section was evaluated as a function of thickness of the films and substrate self-bias. The films prepared at separate substrate self-bias values produces films of different composition. Variation of  $\Sigma_t$  with substrate self-bias is given in table 29.1.  $\Sigma_t$  was estimated from the following formula, Eq. (29.4):

$$I(x) = I_0 e^{-\Sigma_t x} \quad (29.3)$$

$$\Sigma_t = \frac{\ln\left[\frac{I_0}{I(x)}\right]}{x} \quad (29.4)$$

where  $I_0$  = Incident neutron beam intensity;  $I(x)$  = Measured neutron beam intensity measured after passing through the sample, and  $x$  = Film thickness. This is a method for measurement of total macroscopic cross section,  $\Sigma_t$  directly. As seen in table 29.1, the film

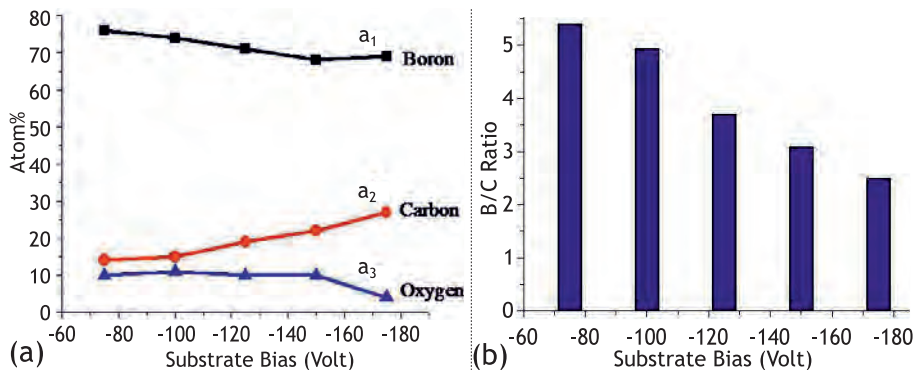


Figure 29.3: (a) Variation of elemental atomic percentages in  $^{nat}\text{B}_x\text{C}$  thin films with substrate self-bias, and (b) change of boron to carbon atomic ratio in the films.

Table 29.1: Variation of  $\Sigma_t$  with substrate self-bias.

Substrate Self-bias (in Volt)	Thickness ( $\mu\text{m}$ )	(in % Neutron absorption)	Total cross section ( $\text{cm}^{-1}$ )	Macroscopic $\Sigma_t$
-75	4.46	7.32	170.47	
-100	4.19	10.06	253.36	
-125	4.10	10.4	267.68	
-150	4.90	11.55	249.41	
-175	4.08	10.6	273.38	

deposited at -75 V, which is the lowest substrate “self-bias” considered in this study, exhibits minimum value of  $\Sigma_t$  ( $170.47 \text{ cm}^{-1}$ ). The film deposited at -175 V, exhibits the highest  $\Sigma_t$  ( $273.38 \text{ cm}^{-1}$ ). The increase in the value of  $\Sigma_t$  can be attributed to the structural densification, which can be confirmed by the enhancement in areal density of atoms with increase in substrate “self-bias” (table 29.1). Once it is found that the films are providing reasonable neutron absorption, the next realization was a p-n junction by depositing the  $^{nat}\text{B}$  boron carbide thin films on high resistivity n-silicon wafer. The thin film of  $^{nat}\text{B}$  boron carbide has properties of a p-type semiconductor. When deposited on a n-type silicon wafer via PECVD technique, the films form a diffusion bonded interface with the substrate and essentially forms the p-n junction. To investigate whether the junction exhibits p-n characteristics or ohmic characteristics, the current (I)-voltage (V) response of a typical sample were measured. For this measurement, metallization (aluminium) was done on opposite sides of the junction and I-V characteristics were measured with the help of an electrometer. The I-V characteristics of the sample are shown in Fig. 29.4. The sample was found to be non-ohmic, with low magnitude of the reverse saturation current (nA), exhibiting diode characteristics, suitable for fabrication of sensors. The breakdown voltage was found to be around -8 V. However, the increase of the reverse saturation current is not sharp at the breakdown voltage. This behavior may arise due to the defects (planer or otherwise) present in the sample. Also, improving the quality of the junction via post deposition annealing can be done. Thus, in future, more precaution will be taken during sample preparation, mainly during silicon wafer dicing to minimize the planer defects. Efforts will be given to improve the characteristics of the junction.

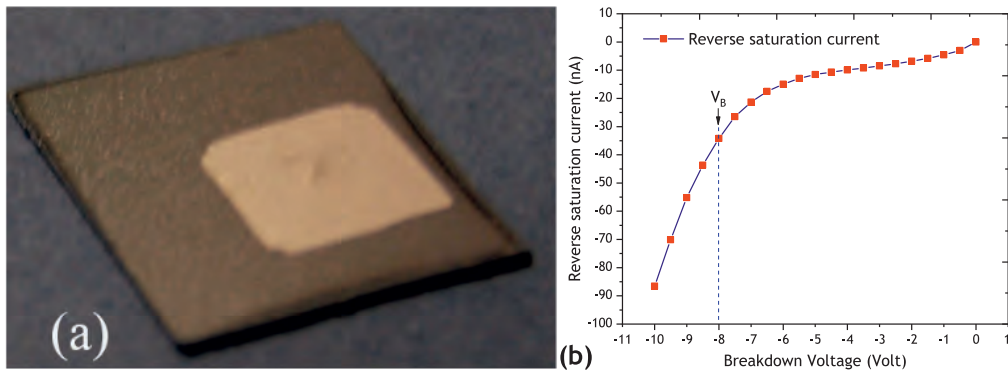


Figure 29.4: (a) Metallized Sample and (b) I-V characteristics of the sample.

## Frequently Asked Questions

- Q1. Out of the four possible configuration of the semiconductor neutron detector stated in the note, which one is preferable and why?
- Q2. What is the most significant parameter in capacitively coupled RF-PECVD system for thin film deposition and why?
- Q3. What is the advantage of boron carbide thermal neutron detector over conventional gas filled detectors?
- Q4. Which parameters of a semiconductor junction determine it's quality?
- Q5. How can one improve the efficiency of the p-n junction neutron detector?