New Surface Morphology for Low Stress Thin-Film-Coated Thermal Neutron Detectors

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***Abstract*—Experimental devices using patterns of holes etched into semiconductor surfaces are under evaluation for use as neutron detectors. The devices have miniature holes equidistantly spaced so as to completely cover the front surface of a planar semiconductor device. The devices have both electrical contacts and neutron-reactive coatings applied over the surface and within the holes. The tiny via holes assist in thin-film adhesion while offering a method to increase the thermal-neutron detection efficiency.**

***Index Terms*—Neutron detector, semiconductor detector.**

I. Introduction

THIN-FILM-COATED semiconductor devices have been investigated for thermal neutron detection by a variety of research groups [1]–[8], all of which have generally used ^{10}B, ^{6}Li, ^{6}LiF, and Gd coatings as the neutron reactive layer. Each of these neutron-reactive materials listed has advantages and disadvantages.

^{10}B has been used by the present research group for a variety of reasons, including its generally large microscopic thermal neutron absorption cross section and its relatively high-energy reaction products. The maximum thermal-neutron detection efficiency for single-coated planar devices is 3.95%, attained when using a *lower level discriminator* (LLD) setting of 300 keV [7], [8]. However, adhesion problems and delamination of the ^{10}B coatings have been limiting factors in realizing the maximum neutron detection efficiency. Thick films of ^{10}B become stressed and often peel off of the device during or after the evaporation process. Although the theoretical optimum thickness is 2.4 μm of pure ^{10}B material [7], [8], it is, in fact, difficult to achieve such a thickness and maintain high device yields. This work describes a method to improve film adhesion to the device surfaces while actually increasing the thermal neutron detection efficiency.

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II. Background and Theoretical Considerations

GaAs-based neutron detectors coated with 98% purified ^{10}B have been under investigation [7]–[11]. The devices are thin diodes, with either a p-i-n configuration or a Schottky diode configuration and operate by detecting neutron-induced reaction products. The $^{10}\mathrm{B}(n,\alpha)^7$Li reaction leads to the following reaction products and branching ratios [12]:

$$^{10}\mathrm{B}+n \rightarrow \begin{cases} 6\%: & ^7\mathrm{Li}(1.015\ \mathrm{MeV})+\alpha(1.777\ \mathrm{MeV}) \\ & Q=2.792\ \mathrm{MeV}(to\ ground\ state) \\ 94\%: & ^7\mathrm{Li}^*(840\ \mathrm{keV})+\alpha(1.470\ \mathrm{MeV}) \\ & Q=2.310\ \mathrm{MeV}(1st\ excited\ state) \end{cases}$$

The thermal neutrons absorbed by ^{10}B produce energetic particles emitted in opposite directions. After absorption, 94% of the reactions leave the ^{7}Li ion in its first excited state that rapidly de-excites to the ground state ($\sim 10^{-13}$ seconds) by releasing a 480-keV gamma ray. The remaining 6% of the reactions result in the ^{7}Li ion going directly to its ground state. The thermal neutron (0.0259 eV) microscopic absorption cross section is 3840 barns. The microscopic thermal neutron absorption cross section increases with decreasing neutron energy; thus, the cross-section dependence is proportional to the inverse of the neutron velocity ($1/v$) in much of the energy range [13], [14].

The ^{10}B-coated GaAs devices have proven to work well and are reliable. Studies have revealed that the detectors can withstand thermal neutron fluences up to 10^{12} n/cm^2 before noticeable degradation begins to appear [11]. Catastrophic damage occurs at thermal neutron fluences above 10^{13} n/cm^2 [11].

The ^{10}B film is applied onto the diode blocking contacts by an electron beam evaporator, a technique that helps to ensure accuracy in the film thickness. Furthermore, precise features for small single-pad detectors and pixel detectors are easily achieved with a lift-off process when using evaporation techniques [15]. Achieving the correct ^{10}B film thickness is critical for optimum performance and undershooting or overshooting the proper thickness will result in decreased neutron detection efficiency [10]. Unfortunately, achieving the optimum ^{10}B thickness of 2.4 μm through evaporation methods has been elusive until recently. For flat surface devices, the ^{10}B film becomes stressed in evaporated layers greater than approximately

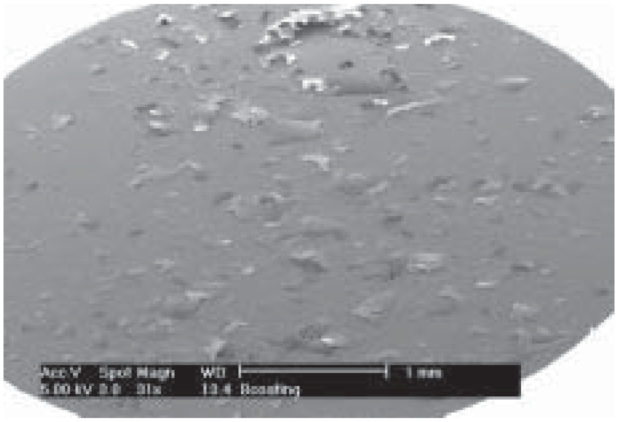

Fig. 1. An example of boron film cracking from thin-film stress. Samples generally began showing signs of boron film cracking at a thickness greater than 1.0 μm.

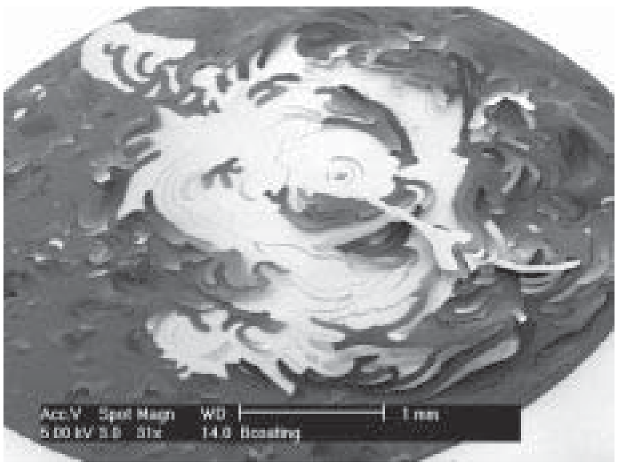

Fig. 2. An example of boron film peeling off of a detector contact after the completed process.

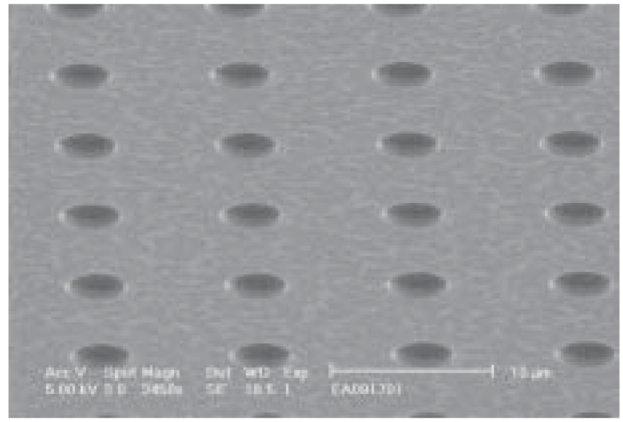

Fig. 3. SEM photogaph showing the via hole matrix used in the present work. The substrate is SI GaAs and the via holes are 3.5 μm in diameter, spaced at 9.5 μm center-to-center, and are 1.7 μm deep.

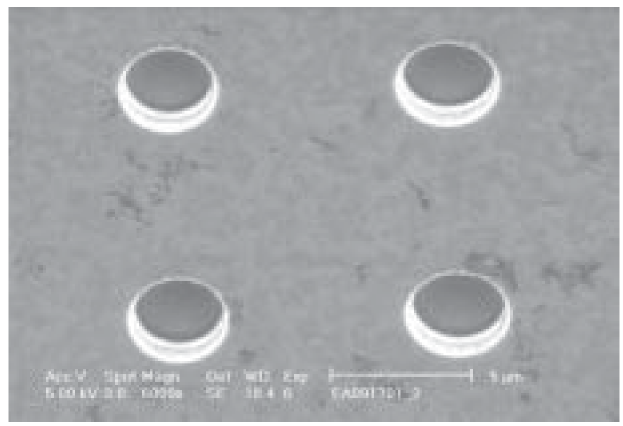

Fig. 4. SEM photograph showing the bottoms of the via holes used in the present work. The hole bottoms are smooth, an important result for reliable Schottky contacts.

1 μm, which causes cracking and peeling (see Figs. 1 and 2). A variety of multiple layering schemes have been attempted with little success. Films thicker than 1 μm that did adhere often peeled later, especially noticeable with temperature changes. To help prevent peeling, devices were coated immediately after the ^{10}B evaporation step with Humiseal 1A33 or 1B73. Yet, coating with Humiseal for ^{10}B films greater than 1.3 μm was seldom effective and peeling would still occur for over 90% of the devices while the coating cured. Consequently, ^{10}B coatings were generally held at 1 μm or less, thereby limiting the thermal neutron detection efficiency to only 2.9%. The new method of surface preparation described in this paper has allowed for the deposition of films up to 4 μm thick with repeatable and reliable results. The films no longer peel even without the Humiseal application.

III. Device Fabrication Steps

The devices were built from vertical gradient freeze-grown semi-insulating (SI) GaAs wafers 3 in in diameter. The SI GaAs wafers were lapped and polished to remove 100 μm of material as described in the literature [9], [16]. The wafers were then cleaned and etched to remove residual lapping and polish damage [9], [16]. A layered combination of Ni, Au, and Ge was evaporated onto the polished and etched GaAs surfaces. The evaporated contacts were subsequently sintered for 1 min at 400°C in argon to produce a low-resistivity ("ohmic") contact [16].

The front surfaces were then lapped and polished until the GaAs wafer thickness was only 200 μm. The front surface was then cleaned and etched. A matrix of open circles 3.5 μm in diameter and spaced 9.5 μm apart from center-to-center was patterned with photoresist over the entire wafer surface. A short oxygen plasma clean (2 min) was used to remove the residual photoresist film from the hole patterns.

Via holes were then etched into the GaAs wafers in a reactive ion etching (RIE) system with a plasma composed of Ar and BCl_3 gases (Figs. 3 and 4). Various via hole depths were fabricated; 1.7 μm, 4.0 μm, and 5.0 μm. For comparison, control samples were also fabricated without undergoing the RIE etch. Afterwards, the photoresist was removed in the RIE with an oxygen plasma.

The devices were then photoresist-patterned with 3.5-mm-diameter open circles covering the surface, spaced apart by 7 mm from center-to-center. A sputtering system was used to coat the wafer with 200 Å of Ti followed by 1000 Å of Au. DC sputtering was used to ensure the formation of a conformal metal coating

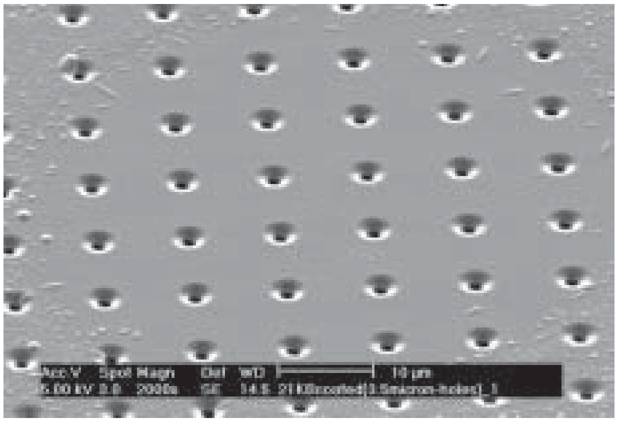

Fig. 5. SEM photograph showing a top view of a device with 4 μm of evaporated ^{10}B coating. There is no sign of peeling or cracking.

over and into the via holes. Lift-off in acetone was used to remove excess Ti/Au material between the devices. Smaller photoresist circular patterns 3 mm in diameter were centered over the 3.5-mm metal contacts to define the boron layers. Hence, the diameter of the boron pads were 0.5 mm smaller than the diameter of the metal Schottky contacts.

Evaporating boron radiates a tremendous amount of heat over the time period required to deposit several microns of material. Excessive substrate heating will cause the photoresist to soften and reflow, which can cause the pattern to smear and can also cause difficulty with the liftoff procedure. To prevent excessive heating, the photoresist patterned samples are routinely attached to a cooling plate inside the evaporator and cooled with chilled water during the ^{10}B evaporation. ^{10}B layers of different thicknesses were deposited, ranging from 1 μm up to 4 μm. Lift-off was used to remove the excess ^{10}B material from the surface.

The devices were cleaved into 7 mm $\times$ 7 mm squares and mounted with Ag epoxy upon custom Al_2O_3 substrate plates for testing. The substrate plates have Au patterns to which the detectors were bonded for electrical connection.

For reasons explained in the next section, ^{10}B powder with a particle size distribution ranging from 0.5 μm in diameter up to 1.8 μm in diameter was placed on top of the ^{10}B-coated contacts for several detector samples. The samples were placed into a beaker within an ultrasonic bath for 15 min so as to shake the tiny ^{10}B particles into the holes. All of the devices were then coated with Humiseal to prevent the powder from falling out.

IV. Results

A. RIE Etch and ^{10}B Film Results

Before the devices were coated with conductive contacts, the depth of the etched via holes were determined by profilometer measurements and confirmed with a scanning electron microscope (SEM). The etch rate was determined to be 85 Å/min. Figs. 3 and 4 show the resulting matrix of 1.7 μm deep via holes in which straight sidewalls and smooth bottoms are apparent. The smooth surface is important for the manufacture of reliable Schottky blocking contacts. Fig. 5 shows a top view of the same via holes after depositing 4 μm of ^{10}B. The SEM photograph clearly shows that the film did not peel, nor did the other devices in the batch. However, many of the planar devices processed as

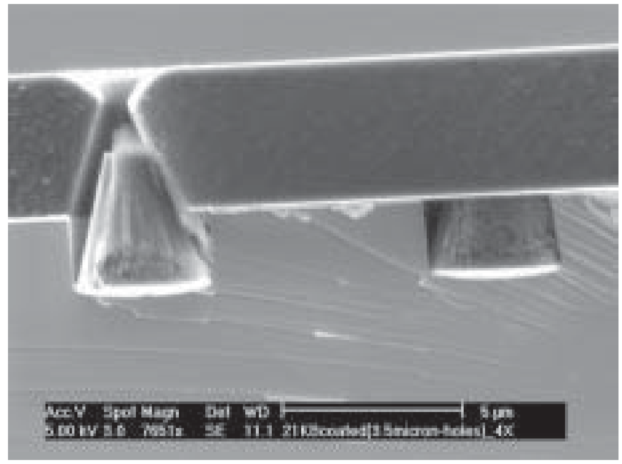

Fig. 6. SEM photograph showing a cross section view of a device with 4 μm of ^{10}B coating. The film is adhered well to the substrate with no sign of peeling. Both via holes are filled with ^{10}B.

control samples during the evaporations did suffer from peeling, much as shown in Figs. 1 and 2. Indentations are apparent from the top view and the via hole openings are narrower, which is also shown in the cross-section view of Fig. 6. The via holes in Fig. 6 are filled with conical-shaped boron plugs, which are a consequence of the slowly narrowing opening with increased boron deposition. The mechanical action of cleaving the sample most likely caused the upper portion of the conical plug to detach (on the left), yet it is still attached to the bottom of the hole. The cleaved slice was not centered through a row of holes, but instead at an angle. Hence, on the right is shown a conical plug still attached and encased within the surrounding boron. Also visible in Fig. 6 are the conductive contacts on the sides and bottoms of the via holes. This important result demonstrates that evaporation is a method by which the holes can be filled with neutron reactive material.

B. Neutron Detection Results

The finished devices were tested in a double-diffracted thermal neutron beam at the Ford Nuclear Reactor (FNR) [10], [11]. Each device was installed in a custom aluminum housing (or "chimney box") that allowed for repeatable indexing into the same location of the neutron beam [10], [11]. The double-diffracted beam had a calibrated flux of 3.3×10^4 n/cm$^2 \bullet$ s, the value used to determine the efficiency for each device. Fig. 7 shows a comparison between the via-hole-covered device with a 4-μm ^{10}B coating and a flat device with a 1-μm ^{10}B coating. Operated as counters, the thermal neutron detection efficiency of the via-hole-covered device is 3.9% compared to only 2.9% for the flat device, demonstrating the advantage of using the thicker film. However, as described earlier, the maximum achievable single film value for front irradiated ^{10}B-coated devices is only 3.95% [7], [8], which occurs at a film thickness of 2.4 μm.

Boron-10 films thicker than 2.4 μm cause neutron attenuation that decreases the detection efficiency and a device coated with 4 μm of ^{10}B should be able to achieve only 3.67% efficiency [7], [8]. Hence, the observed efficiency is higher than expected but easily explained as an additional benefit of the via-hole-covered

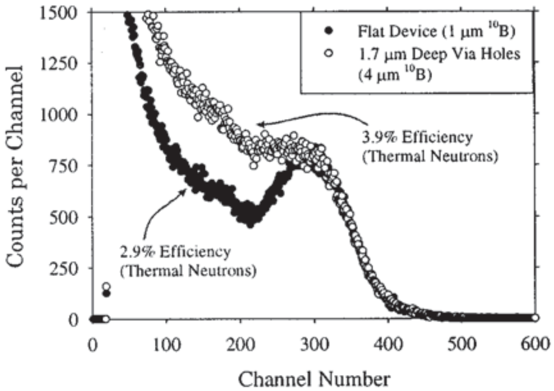

Fig. 7. Comparison $^{10}B(n,\alpha)^7Li$ reaction product spectra for a flat device with a 1 μm ^{10}B coating and a via hole covered device with a 4 μm ^{10}B coating. The via holes on the etched device are 1.7 μm deep, 3.5 μm in diameter and spaced 9.5 μm apart center-to-center.

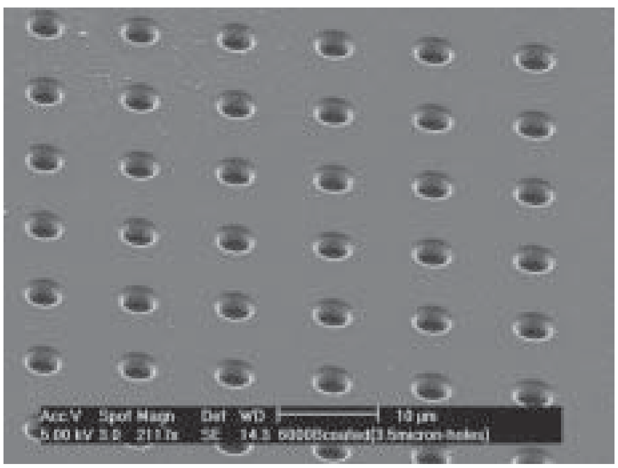

Fig. 8. SEM photograph showing a top view of a device with 1 μm of evaporated ^{10}B coating. The holes are still open enough to fill with ^{10}B powder.

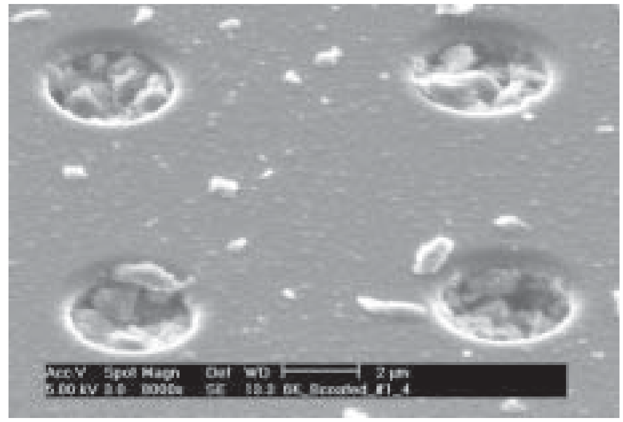

Fig. 9. SEM photograph showing a top view of a device with 1 μm of evaporated ^{10}B coating with the holes filled with ^{10}B powder.

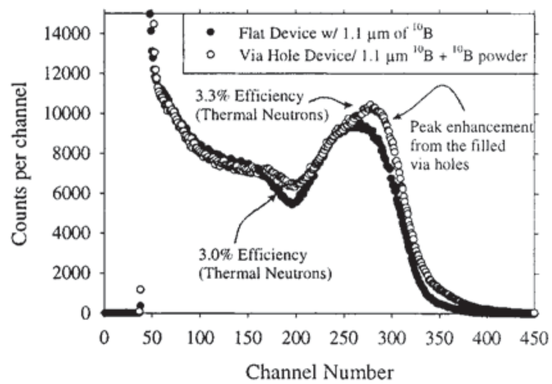

Fig. 10. Comparison spectra of $^{10}B(n,\alpha)^7Li$ reaction products from a flat device and from a device with 5.0-μm-deep via holes. The via holes, 3.5 μm in diameter and covering 10.6% of the surface area, were filled with ^{10}B powder. The evaporated ^{10}B coating was 1.1 μm thick for all devices.

detector surface. The ^{10}B material within the via holes allows for reaction products to enter the sides of the cavity, thereby increasing the detection probability above that of a flat device. From Fig. 6, it is apparent that a portion of the cavity is not filled with ^{10}B, indicating the neutron detection efficiency could actually be increased further. To explore more complete filling of the via-holes, another series of devices were fabricated using the same patterns, but this time comparing planar devices to devices with 5-μm-deep via holes. The boron overcoat was thinner at 1.1 μm, giving the appearance shown in Fig. 8 and thereby leaving the via hole openings large enough to insert the fine grain ^{10}B powder. Fig. 9 shows the holes filled with the boron powder. Fig. 10 shows comparison spectra between devices with no via holes and devices with 5.0 μm deep via holes filled with ^{10}B powder. The devices were operated with a reverse bias of 100 V and a gain of 300. A commercial Ortec 142A preamplifier was used to measure the induced charge.

Planar and via-hole sample devices processed in parallel from the same original wafer (cleaved in half) were tested within a time period of a few hours, thereby reducing deviations due to neutron flux changes from control rod motion in the FNR. The batch-processed devices had similar characteristics and all were operated with the same bias voltage, gain, and LLD settings. Furthermore, planar and via hole devices were alternately tested to remove systematic errors. The planar devices had measured thermal-neutron detection efficiencies of 3.05%, matching well with theory [7], [8]. The devices with 5.0-μm-deep via holes had measured efficiencies of 3.34%, an increase of 10% over the flat devices. The total counts (and count rate) are statistically well beyond $3\sigma(+2\sigma$ would increase the efficiency to only 3.06%). Neutron flux changes amount to no more than approximately 3% of the average flux during normal operations during a day, which does not explain the observed changes in detection efficiency. It could also be argued that the via holes have increased the detector surface area, thereby allowing for more boron to contact the surface. Yet, such a claim is not supported by Fig. 6, where it is clear that the boron along the walls is either absent or very thin. The observed efficiency increase is not adequately explained to result solely from increased surface area imposed by the via holes. In other words, the via holes cover 10.6% of the device area, which, if filled entirely, would give a flat device value of only 3.6% efficiency for those regions. Correcting the remaining areas such that the 3.05% efficiency is represented by 89.4% coverage, the result would be an increase up to only 3.11% efficiency, not 3.34%. The increase to 3.34% is due to an increased detection probability of neutron interactions within the via holes. Given the experimentally observed trend, the same devices with 5.0-μm-deep via holes and 40% coverage will yield 4.3% thermal neutron detection efficiency. Etching 10 μm deep holes and filling with ^{10}B should increase the efficiency above 6.0%.

V. Conclusion

Boron expands when it solidifies from the melted liquid. It is believed that the stress produced by the evaporated film as it cools on the substrate is enough to cause it to peel and crack. Peeling has been observed for films that appeared stable until interrupted by an abrupt decrease in temperature. For instance, many planar devices with what appear to be stable films are observed to peel within minutes after moving them from a room-temperature environment (20°C) to a freezing environment (< 0°C). Attempts to remedy the problem included substrate cooling during the evaporation process, but yielded only limited success. Morphologically altering the detector surface with via holes has thus far proven to solve the problem of peeling of evaporated ^{10}B films, even with abrupt temperature changes. The tiny holes etched over the device surface are believed to assist in adhesion of the boron films by two methods. First, the pattern of holes relieves the stress produced by the film, thereby allowing the film to remain on the surface. Second, the conical boron plugs act as miniature anchors in the via holes that actually assist in holding the film in place.

Boron films up to 4-μm thick have not shown signs of peeling after several weeks, even without Humiseal coatings. The process is straightforward and can be easily transferred to other substrate materials such as Si and SiC. As a result, the optimum film thickness of 2.4 μm can be routinely manufactured for ^{10}B-coated neutron detectors.

The observed increase in efficiency can be explained as a fortuitous added effect from the filled via holes. Reaction products from neutrons interacting in the ^{10}B film covering the flat surface have similar probabilities of entering the detector as described in previous work [7], [8]. For a flat device, the probability of a reaction product entering the detector decreases as reactions occur further away from the detector-film/contact interface. However, the same restrictions do not apply for interactions occurring within the via holes and the probability of a reaction product entering the device is increased for neutron interactions within the via holes.

The reaction products can be emitted in any direction over 4π steradians and the probability that a reaction product will enter the detector is determined by the solid angle that intersects the detector [7]. Fig. 11 depicts a sphere with radius equal to a reaction product particle effective range, showing that the solid angle can intersect the bottom and side of the via hole. Increased sensitivity is best if the via hole diameters are less than the summed total effective ranges of both reaction products. For the alpha particle and ^{7}Li emissions in boron, the summed effective ranges [7], [8] equals 3.14 μm, a length very close to the via hole diameters in the present work.

VI. Future Directions

New via hole patterns are under development that will prevent film cracking and peeling while also increasing the overall

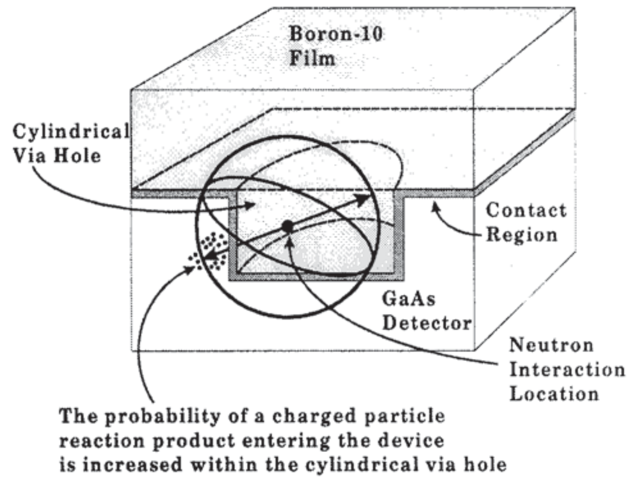

Fig. 11. Diagram of a single via hole with dimensions similar to the added ranges of the reaction products. The geometry increases the reaction product detection probability above that of flat surface designs since the products can enter the device from many directions.

observed thermal neutron detection efficiency. One such configuration is a system of narrow straight trenches. Straight trenches can be designed to run the entire length of a device and the straight trench design also allows for an increase in detection sensitivity. Unfortunately, particles with trajectories generally directed parallel to the trench length will not enter the semiconductor detector and will not be detected, a case not found with the via hole design.

However, the filled trench area density, the fraction of area covered by the depressions, can be quite high. Using the criteria that the trenches are 3 μm wide, a 50% area density is achieved with the trenches spaced 6 μm apart center-to-center and a 60% area density is achieved if the trenches are spaced 5 μm apart center-to-center. Using the same criteria for 3 μm diameter holes, an area density of only 22.67% is achieved with the via holes spaced 6 μm apart center-to-center (hexagonal pattern) and only 32.64% area density is achieved if the via holes are spaced 5 μm apart center-to-center. As a result, the total area of etched regions filled with reactive material is much smaller for the circular via hole design than the long-trench design.

The detector mass available between holes to absorb the particle energy diminishes as the pattern area density increases. Using the $^{10}\mathrm{B}(n,\alpha)^{7}\mathrm{Li}$ reaction as an example and assuming orthogonal trajectories, 3 μm of GaAs material can absorb all of the ^{7}Li ion energy, but only about 1.1 MeV of the alpha particle energy [17]. Furthermore, 2 μm of GaAs material can still absorb almost all of the ^{7}Li ion energy, but only about 700 keV of the alpha particle energy [17]. Different substrates and neutron-reactive materials are now under investigation. Si and SiC are being investigated as alternative semiconductor substrates for the devices and ^{6}LiF and 98% pure ^{6}Li are being studied as alternative neutron-reactive films.

Research for the optimum design that will yield the highest possible detection efficiency is ongoing, including patterns that mix via holes of circular, square, rectangular, and ellipse shapes. Some via hole patterns intermixing holes and trenches can exceed 40% area coverage. Calculations using a simple geometrical model project that thermal-neutron detection efficiency can

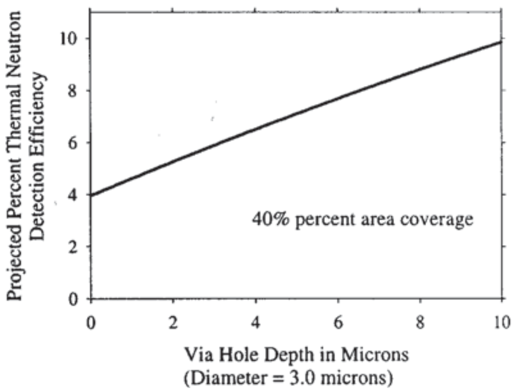

Fig. 12. Modeled efficiency for a device structure using a matrix of trenches and via holes covering 40% of the area. The trenches are 2 μm wide and the via hole diameters are 3.0 μm.

reach 10% with such changes (see Fig. 12), a 2.5 times increase in efficiency over the calculated 3.95% efficiency limit for flat devices [18].

The via hole pattern used in the present work allowed only 10.6% area coverage. The hole diameters (3.5 μm) are also not optimized for boron reactions. Next generation devices will use a hexagonal pattern and an area density of 22% with a matrix of 3.5-μm-diameter holes 10 μm deep. The powder-fill method used to fill the holes, although effective, leaves much of the space unused. For instance, a system of perfect spheres 0.5 μm in diameter will fill less than 73% of the via hole cylinder. Since the granules actually ranged from 0.5 μm to 1.8 μm, it becomes obvious that the packing fraction will be less than 73%, which most likely accounts for the lower observed efficiency than the models predict. A method to completely fill the holes while increasing the packing fraction must be developed if optimum performance is to be realized.

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