The structure and methods of fabricating a high efficiency compact solid state neutron detector based on III-Nitride semiconductor structures deposited on a substrate. The operation of the device is based on absorption of neutrons, which results in generation of free carriers.

20 Claims, 10 Drawing Sheets
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Figure 1
Figure 5
Figure 7
Figure 10
SOLID-STATE NEUTRON DETECTOR DEVICE

This application claims priority to U.S. Provisional Patent Application Ser. No. 61/970,841, entitled “Compact Solid-State Neutron Detector,” filed Mar. 26, 2014, the entire content of which is hereby incorporated by reference. This invention was made with support, in parts, of the NASA Small Business Innovation Research Program. The government may have certain rights in the invention.

BACKGROUND

The present disclosure relates generally to solid-state neutron detectors, and more specifically to the structure and fabrication of high sensitivity compact solid-state neutron detectors.

Low energy neutron ($E_n<1\text{eV}$) detection plays an increasingly critical role in many applications including medicine, high energy physics (HEP), and homeland security. Currently, two inch helium-3 ($^3\text{He}$) tube neutron detectors can achieve detection efficiencies of up to 80%, while portable handheld one inch $^3\text{He}$ tube neutron detectors can achieve efficiencies of 10-15%. These efficiencies are reasonable for detection of thermal neutrons in high flux environments, such as in HEP applications, however they become impractical in low flux environments, such as in detecting presence of radioactive material for homeland security. Moreover, since the discovery of the scarcity of $^3\text{He}$, substitute neutron detectors have become a priority. $^3\text{He}$ is a rare isotope of helium primarily obtained from radioactive decay of tritium, and since the end of the cold war the United States have consistently reduced their nuclear stockpile making the helium isotope scarce. Also, the demand of helium-3 isotopes have increased primarily for use in neutron detectors, and current supplies are not able to meet the demand.

One alternative approach has been the use of scintillator, water-based, and semiconductor type detectors. Thus far the major challenge with semiconductor detectors is the ability to fabricate very thick layers of semiconducting materials and fabricate efficient devices from them. Thick layers are typically required due to the low neutron capture cross-sections of most available semiconductors. For example, the neutron capture cross-section of silicon-28 isotope is $\sim10^{-41}\text{ barns}$ as a bulk semiconductor. The material developed using the bridgeman method, has a bandgap of 2.85 eV and a high bulk resistivity of $3.17\times10^{11}\text{ Q}\cdot\text{cm}$. The material is claimed not suitable for growing thick films. However, these techniques are usually expensive and not suitable for growing thick films.

One material recently developed by Stowe et. al. is a $\text{LiInSe}_2$, that utilizes the $^6\text{Li}$ capture cross-section of 938 barns as a bulk semiconductor. The material developed using the bridge man method, has a bandgap of 2.85 eV and a high bulk resistivity of $3.17\times10^{11}\text{ Q}\cdot\text{cm}$. The material is claimed to have very high neutron detection efficiencies compared to $^3\text{He}$ tube detectors, primarily due to the close packed nature of the $^6\text{Li}$ atoms in the crystal compared to $^3\text{He}$ atoms in gaseous form. Thus utilizing materials with higher neutron capture cross-sections will only further improve the neutron detection efficiency and enable the development of handheld low cost and low power neutron detectors.

Recent developments on improvement of the growth quality of III-Nitride materials and the availability of GaN wafers, allow for the development of high efficiency harsh environment resistant devices. The group III-Nitride ternary compounds composed of $\text{Al}_x\text{Ga}_{1-x}\text{N}$ (bandgap 6.2 to 3.42 eV) and $\text{In}_x\text{Ga}_{1-x}\text{N}$ (bandgap 3.42 to 0.62 eV) exhibit inherent chemical and thermal ruggedness, which makes them suitable for several space and military applications. It has recently been determined that these Nitride materials can also offer exceptional radiation tolerance that is well beyond what can be achieved with conventional materials that are currently flown in space. For example silicon based detectors have shown up to 65% loss under irradiation of $10^{12}\text{protons/cm}^2$. III-Nitride materials present several advantages over other semiconducting materials currently used for optoelectronic devices. These include: 1) intrinsic radiation hardness; 2) ability to tune the bandgap from 6.2 eV to 0.62 eV; 3) layers of different bandgaps can be grown using well developed MBE or MOCVD methods; 4) large bandgap resulting in low thermal noise; 5) negative electron in wavelength shifting optical fibers and converted to 500 nm (green) photons still employ high sensitivity photon multipliers (PMTs) which are bulky, mechanically and thermally unstable, and expensive. Moreover, the combination of fiber optic cables and PMTs make it difficult to realize handheld portable neutron detectors.

In contrast, semiconductor based devices can have a low operating voltage, small device footprint, and excellent stability. The dilemma is how to best utilize semiconductors to design an optimized thermal neutron detector. Most semiconductor materials have very low neutron capture cross-sections, the most common approach to fabricate solid state neutron detectors is to coat the semiconductor devices with materials that have higher capture cross-sections. Thus the neutron absorbing layer generates charged ions and ionizing radiation that are detectable by the semiconductor. The ions are only detectable if they reach the semiconductor interface. The charged particle is sufficient enough to reach the interface of the semiconductor/moderator. Thus conventional neutron sensitive thin-film coated semiconductors face the challenge of balancing the neutron absorption efficiency in the thin film and charge transfer efficiency to the semiconductor junction. Thin film semiconductor neutron detectors have low neutron capture efficiency, for example MgB$_{14}$, thin films deposited on silicon diodes have an efficiency of only 1.3%.

In order to overcome this challenge, researchers have employed solid form semiconductor devices that are composed at least partially by a neutron sensitive material. Typically solid form semiconductors are grown by chemical vapor deposition (CVD) and have the neutron sensitive material embedded in the crystal of the semiconductor. However, these techniques are usually very expensive and not suitable for growing thick films.

The ions are only detectable if they reach the semiconductor interface. The charged particle is sufficient enough to reach the interface of the semiconductor/moderator. Thus conventional neutron sensitive thin-film coated semiconductors face the challenge of balancing the neutron absorption efficiency in the thin film and charge transfer efficiency to the semiconductor junction. Thin film semiconductor neutron detectors have low neutron capture efficiency, for example MgB$_{14}$, thin films deposited on silicon diodes have an efficiency of only 1.3%.

The present disclosure relates generally to solid-state neutron detectors, and more specifically to the structure and fabrication of high sensitivity compact solid-state neutron detectors.
affinity (NEA) enabling field emission capabilities; 6) high degrees of chemical, mechanical, and thermal stability, plus high resistance to sputtering.

Gadolinium isotopes 155 and 157 have higher capture cross sections compared to any other materials, with 155Gd having a cross section of 65,000 barns and 157Gd having a cross section of 255,000 barns. Most important is that the mean free path of neutrons in 157Gd is only 1.3 µm, and a thickness of 6 µm is sufficient to stop 99% of thermal neutrons (25 meV).

GdN is a semiconductor with a half metallic bandgap calculated to be 0.6 eV, however, recent experimental evidence based on the optical absorption spectrum of rf magnetron sputter deposited GdN have shown bandgaps ranging from 1.03 eV to 0.95 eV. The lattice constant of bulk GdN (111) is about 4.9 Å, however, GdN (111) plane has a hexagonal symmetry that matches with the 0001 plane of InN with a lattice mismatch close to 0%. High quality growth of GdN (111) has been attempted on GaN and AlN films, however due to the lack of readily available InN substrates, to the best of our knowledge, it has not been attempted on InN.

Naturally occurring Gd has a neutron capture cross section of 49,700 barns, which is much higher than those of most materials. Compared to 6Li, the capture cross-section is more than 50 times higher for naturally occurring Gd, and it is more than 500 times higher if 157Gd is used. Of high interest is that Gd easily forms GdN, which is a semiconductor with an experimental bandgap of 1 eV. GdN (111) plane has a hexagonal symmetry that matches with the 0001 plane of III-Nitride materials. Several groups have grown GdN on GaN and Mn, and have demonstrated polycrystalline quality GdN materials. Fig. 1 shows the alignment of the face centered cubic (fcc) structure of GdN with the hexagonal structure of GaN. The in-plane N—N interatomic distance is given by 4.9V2−3.52 A, where 4.9 Å is the lattice constant of GdN, this leads to a biaxial strain of -9.4%, that eventually leads to polycrystallinity. In contrast, the InN lattice constant is 3.54 Å, making InN a perfect substrate match for GdN. However, due to the lack of readily available InN substrates and/or "templates", there has been no work demonstrating the potential growth of GdN on InN.

Patent publication WO2013025259 describes a portable thermal neutron detector based on an array of Si CMOS transistors covered with a Gd containing film, such as gadolinium oxide, deposited by plasma enhanced atomic layer deposition.

Another patent publication, WO2011002906, describes at least thermal neutron detection with a capacitor type solid-state device based on gadolinium oxide deposited on low resistivity semiconductor substrate.

Patent publication WO2006085307 describes a solid-state device for detection of neutron and alpha particles detector that has an active region formed of a polycrystalline semiconductor compound containing 10Boron, 6Lithium, 111Cadmium, 157Gadolinium and 199Mercury. The semiconductor compound is sandwiched between an electrode assembly by using an organic or inorganic binder.

Patent publication WO2009117477 describes a silicon-on-insulator (SOI) neutron detector device with lateral carrier transport and collection detector structure within the active semiconductor layer of the silicon-on-insulator structure, and a neutron to high energy particle converter layer on the active semiconductor layer that includes cadmium, gadolinium, gadolinium phosphate, gadolinium oxide, and their combinations.
Solid-state neutron detectors can be used in a variety of techniques, ranging from medicinal practices to high energy physics (HEP) to space and military operations. The present disclosure relates to the structure and method for fabricating solid-state neutron detectors which are highly efficient, compact and producible at low cost. The present solid-state neutron detectors also alleviate decreased neutron detection efficiency in harsh environmental conditions.

In one embodiment, the neutron detector device comprises a first contact, a capping layer, a neutron absorption layer comprising a plurality of interdigitated layers of at least two distinct materials, a graded layer, a substrate further comprising a top substrate layer, and a second contact.

In another embodiment, the neutron detector device comprises a first contact, a capping layer, a neutron absorption layer comprising a plurality of micro- or nano-column structures of at least two distinct materials, a substrate, and a second contact.

The present disclosure also relates to methods for fabricating a neutron detection device comprising a first contact, a capping layer, a neutron absorption layer comprising a plurality of interdigitated layers of at least two distinct materials, a graded layer, a substrate further comprising a top substrate layer, and a second contact.

Additionally, the present disclosure relates to methods for fabricating a neutron detection device comprising a first contact, a capping layer, a neutron absorption layer comprising a plurality of micro- or nano-column structures of at least two distinct materials, a substrate, and a second contact.

Other systems, methods, features, and advantages of the present disclosure will be or become apparent to one with skill in the art upon examination of the following drawings and detailed description. It is intended that all such additional systems, methods, features, and advantages be included within this description, be within the scope of the present disclosure, and be protected by the accompanying claims.

BRIEF DESCRIPTION OF THE DRAWINGS

Aspects of the disclosure can be better understood with reference to the following drawings. The components in the drawings are not necessarily to scale, emphasis instead being placed upon clearly illustrating the principles of the present disclosure. Moreover, in the drawings, like reference numerals designate corresponding parts throughout the several views, and in which:

FIG. 1 shows a schematic representation of GdN lattice alignment with GaN structure.

FIG. 2 shows a side view in accordance with an exemplary embodiment of the present disclosure.

FIG. 3 shows a side view in accordance with an exemplary embodiment of the present disclosure.

FIG. 4 shows a side view SEM image of micro columns fabricated on a silicon wafer using pulsed laser ablation in accordance with an exemplary embodiment of the present disclosure.

FIG. 5 shows an x-ray diffraction pattern of sputtered GdN layers.

FIG. 6 shows high quality AlN and AlGaN films grown on a silicon substrate and InGaN film grown on a sapphire substrate.

FIG. 7 shows x-ray data for p-n junction InGaN devices with thickness 425-450 nm and various indium compositions, along with a 570 nm In_{0.84}Ga_{0.16}N single layer for reference.

FIG. 8 shows front illuminated photodiode structures.

FIG. 9 shows back illuminated photodiode structures.

FIG. 10 shows x-ray diffraction data for various sputtered layers (including ZrN) deposited on sapphire substrates.

DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

In the description that follows, like parts are marked throughout the specification and drawings with the same reference numerals. The drawing figures might not be to scale and certain components can be shown in generalized or schematic form and identified by commercial designations in the interest of clarity and conciseness.

Interdigitated Layer Structure

The device structure of a first exemplary embodiment is shown in FIG. 2. It consists of a high crystalline quality GaN layer (1), a graded GaN/InGaN/InN layer (2), interdigitated GdN (3) and InN (4) layers, a capping ZrN layer (5), and metal contacts to the top ZrN layer and the bottom GaN layer (6).

The operation of the device is based on absorption of neutrons by the Gd atoms in the GaN layers, which results in generation of free carriers. These carriers can be collected if a p-n junction is present in the structure. For example, such p-n junction is formed if the GaN/InGaN/InN interdigitated structure is of n-type, and the bottom GaN layer is of p-type.

If all the layers in the structure are electrically conductive, but do not form a p-n junction, generated carriers will add to the current resulted from a bias applied between the two contacts in the bottom and on the top of the device structure.

If any of the layers in the GaN/InGaN/InN graded layer or in the interdigitated GaN/InN exhibit dielectric properties (because, for example, of extremely low doping concentration or amorphous nature) the capacitance of the structure can be measured at a pulsed voltage applied to the contacts as a function of charge carriers generated during absorption of neutrons by Gd atoms.

While using Gd isotopes will greatly benefit the device efficiency, naturally occurring Gd can be used for a proof of concept in the first embodiment. Naturally occurring Gd has a neutron capture cross-section of 49,700 barns, which is still much higher than many other materials used for neutron detection.

The lattice constants of InN and GaN are 3.54 Å and 3.19 Å, respectively, which results in up to 10% lattice mismatch. In order to generate minimum defects associated with this mismatch, a graded GaN/InGaN/InN layer (2) is grown first by PAMBE on commercial templates of low defect density GaN pre-grown by MOCVD on sapphire substrates(1). Then >12 µm thick heterostructure comprising of interdigitated GdN (3) and InN (4) layers (>1.2 µm thick each) is grown using RF assisted DC magnetron co-sputtering, followed by the deposition of a capping ZrN layer (5). The structure is patterned, and etched by using reactive ion etching (RIE) to expose the bottom GaN layers (1) and separate pixels in the array. Metal contacts (6) are deposited then on the top of the capping layer (5) and onto the exposed GaN template layer (1) by using vacuum evaporation or sputtering.

In one embodiment of the present disclosure, the capping layer, interdigitated layer, and graded layer do not alter their
performance under harsh environments (unless exposed to Cl\textsuperscript{-} and/or F\textsuperscript{-} ion containing plasmas and/or temperatures above 250 °C.)

Method of Fabricating Interdigitated Layer Structure

First the GaN on sapphire template wafer is cleaned using a standard process followed by etching in concentrated sulfuric acid, DI water rinse, and nitrogen dry, before it is loaded into the PAMBE system and a graded GaN/InGaN/InN structure is grown by using conditions optimized in our previous experiments on the growth of InGaN with high (>80%) In content. The sample then is loaded into the sputtering chamber for deposition of interdigitated InN and GaN layers.

The sputtering chamber is equipped with 3 sputter guns that allow for co-sputtering. Each gun has a separate individually controlled N\textsubscript{2} gas input allowing for variation of the N\textsubscript{2} flow rate and optimization of the deposition conditions for each individual layer. The sputtering system is also equipped with a water cooled quartz crystal monitor that allows in situ monitoring of the deposition rate.

The substrate is degassed in the chamber at a temperature above 800 °C and then the substrate temperature is kept at 700 °C and 500 °C during the GaN and InN depositions, respectively. The individual sputter rates and deposition parameters for each of the materials has to be optimized by utilizing the growth conditions developed in the previous experiments.

The next step requires a capping layer to prevent the GaN layer from oxidation. A recommended option is to sputter ZrN as the capping layer, since it is highly conductive, almost transparent to neutrons, and can act as a contact layer while also being a very inert material.

The RIE etching is performed through a pre-patterned photore sist mask by using a mixture of chlorine and argon gases. The etch rates have to be accurately determined in order to etch mesas that will divide the active device structure on pixels and expose the bottom contact layer. Ohmic metal contacts are deposited on the exposed by RIE GaN bottom layer and the top ZrN layer by using standard vacuum evaporation or sputtering.

Nanor Micro Column Structure

The device structure of the second embodiment is shown in FIG. 3. It consists of a semiconductor silicon substrate (7), a nano or micro column structured Gd containing layer (8), a capping ZrN layer (5), and metal contacts to the top ZrN layer and the bottom of the silicon substrate (6).

The operation of the device is based on absorption of neutrons by the Gd atoms in the nano or micro column structured Gd containing layer. After passing through almost completely transparent ZrN layer (5) the neutrons interact with the Gd atoms generating free carriers. These carriers can be collected if a p-n junction is present in the structure. For example, such p-n junction is formed if the laser ablation structured Gd containing layer is of p-type, and the semiconductor silicon substrate is of n-type.

The Gd containing layer (8) is structured as an array of nano or micro columns with high height-to-width aspect ratio and density. Upon absorption of neutrons by the Gd atoms these nano or micro columns will emit scattered charged particles that can generate additional electron hole pairs resulting in the increase of the detection efficiency of the device.

If all the layers in the structure are electrically conductive, but do not form a p-n junction, generated carriers will add to the current resulted from a bias applied between the two contacts in the bottom and on the top of the device structure.
substrate. Similarly to the first embodiment, the device structure of the second embodiment can be fabricated as a multi-pixel device with individual pixels addressing in order to enable imaging capabilities.

It should be emphasized that the above-described embodiments are merely examples of possible implementations. Many variations and modifications may be made to the above-described embodiments without departing from the principles of the present disclosure. All such modifications and variations are intended to be included herein within the scope of this disclosure and protected by the following claims.

EXAMPLES

Polycrystalline GdN layers by sputter deposition of Gd targets in a nitrogen plasma environment onto sapphire wafers were demonstrated. FIG. 5 shows the x-ray diffraction pattern of the sputtered GdN layers. Gd is very reactive with oxygen and oxidizes rapidly in air, thus the GdN layer was capped with a ZrN layer. While being polycrystalline, the structure is predominantly of (111) or (100) orientation.

Optimization of III-Nitride growth conditions resulted in high quality films for applications in UV/IR photodetectors, solar cells, LEDs, cold cathode devices, enhancement of MCP performance, avalanche LEDs, and many other devices. FIG. 6 shows high quality AlN and AlGaN films grown on silicon, and InGaN film grown on sapphire. For the InGaN films the color variation is observed due to a different content of In in InGa x N.

High quality InGaN films grown using a custom built RF Plasma Assisted Molecular Beam Epitaxy (PAMBE) system have been confirmed by x-ray data shown in FIG. 7. Presence of (0002) InGaN peak is evidence of the high quality single crystalline InGaN layers with no phase separation.

FIG. 8 and FIG. 9 show front and back illuminated photodiode structures, respectively. The silicon substrate was also used as a visible light passive filter in the back illuminated structures to generate the narrow band IR response shown in FIG. 10.

High crystalline quality nitride layers deposited on sapphire substrates have been demonstrated by using RF assisted magnetron sputtering system equipped with high temperature substrate capability. FIG. 10 shows the X-Ray Diffraction (XRD) spectroscopy data for various (including ZrN) layers deposited by using the above method on sapphire (Al2O3) substrates.

Other systems, methods, features, and advantages of the present disclosure will be or become apparent to one with skill in the art upon examination of the following drawings and detailed description. It is intended that all such additional systems, methods, features, and advantages be included within this description, be within the scope of the present disclosure, and be protected by the accompanying claims.

What is claimed is:

1. A solid-state neutron detector device comprising:
   a layered structure, having the following layers interposed relative to one another as follows:
   a first contact; a capping layer; a neutron absorption layer comprising a plurality of interdigitated layers or grading layer exhibiting dielectric properties.

2. The device of claim 1, wherein all of the layers are electrically conductive.

3. The device of claim 1, wherein the graded layer comprises gallium nitride, indium gallium nitride, indium nitride, or a combination thereof.

4. The device of claim 1, wherein at least one of the plurality of interdigitated layers or grading layer exhibits dielectric properties.

What is claimed is:

1. A solid-state neutron detector device comprising:
   a layered structure, having the following layers interposed relative to one another as follows:
   a first contact; a capping layer; a neutron absorption layer comprising a plurality of interdigitated layers or grading layer exhibiting dielectric properties.

2. The device of claim 1, wherein the neutron absorption layer comprises gallium nitride, indium gallium nitride, indium nitride, or a combination thereof.

3. The device of claim 1, wherein the layer comprises gallium nitride, indium gallium nitride, indium nitride, or a combination thereof.

4. The device of claim 1, wherein the layer comprises gallium nitride, indium gallium nitride, indium nitride, or a combination thereof.

5. The device of claim 1, wherein the total thickness of the first material layer is between about 6 µm to about 12 µm.

6. The device of claim 1, wherein the capping layer comprises zirconium nitride, titanium nitride or other materials which prevent oxidation of the neutron absorption layer.

7. The device of claim 1, wherein the second contact is composed of a metal or combination of metals making it Ohmic to the top substrate layer.

8. The device of claim 1, wherein the neutron absorption layer and the top substrate layer form a p-n junction.

9. The device of claim 1, wherein at least one of the plurality of interdigitated layers or grading layer exhibits dielectric properties.

10. The device of claim 1, wherein all of the layers are electrically conductive.

11. A solid-state neutron detector device comprising:

12. The device of claim 11, wherein the substrate comprises a semiconductor silicon wafer.

13. The device of claim 11, wherein the semiconductor silicon wafer has an n-type doping concentration from about 10^{14} cm^{-3} to about 10^{16} cm^{-3}.

14. The device of claim 11, wherein the plurality of micro- or nano- column structures comprises gadolinium, boron, lithium, or a combination thereof; and wherein the micro- or nano- column structures have a height-to-base diameter aspect ratio of at least about 1.5.

15. The device of claim 11, wherein the plurality of micro- or nano- column structures comprise doped semiconductor silicon.

16. The device of claim 11, wherein the neutron absorption layer has a p-type doping.

17. The device of claim 11, wherein the capping layer comprises zirconium nitride, titanium nitride, or other materials which prevent oxidation of the neutron absorption layer and are transparent to neutrons.

18. The device of claim 11, wherein all of the layers are electrically conductive.

19. The device of claim 11, wherein the neutron absorption layer and the substrate form a p-n junction.

20. The device of claim 11, wherein at least one of the materials of the neutron absorption layer exhibits dielectric properties.