DIAMOND ELECTRONIC DEVICES AND METHODS FOR THEIR MANUFACTURE

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ABSTRACT
The present invention relates to a diamond electronic device comprising a functional interface between two solid materials, wherein the interface is formed by a planar first surface of a first layer of single crystal diamond and a second layer formed on the first surface of the first diamond layer, the second layer being solid, non-metallic and selected from diamond, a polar material and a dielectric material, and wherein the planar first surface of the first layer of single crystal diamond has an Rq of less than 10 nm and has at least one of the following characteristics: (a) the first surface is an etched surface; (b) a density of dislocations in the first diamond layer breaking the first surface is less than 400 cm\(^{-2}\) measured over an area greater than 0.014 cm\(^2\); (c) a density of dislocations in the second layer breaking a notional or real surface lying within the second layer parallel to the interface and within 50 \(\mu m\) of the interface is less than 400 cm\(^{-2}\) measured over an area greater than 0.014 cm\(^2\); and (d) the first surface has an Rq less than 1 nm.
DIAMOND ELECTRONIC DEVICES AND METHODS FOR THEIR MANUFACTURE

[0001] The present invention relates to electronic devices fabricated in diamond, and to methods of manufacture of these electronic devices in order to obtain high performance.

BACKGROUND OF THE INVENTION

[0002] The present generation of high frequency (HF) and microwave signals is mostly based on Si and GaAs devices. Due to physical limitations, these devices cannot achieve power levels higher than a few hundred watts (depending on the frequency to be amplified) in simple solid-state device configurations. Wide band gap materials (diamond, SiC, GaN, etc.), in principle, allow for higher power amplification per unit gate length at microwave frequencies. This is because a larger bias voltage, and hence a larger voltage amplitude on the microwave signal, can be supported across the transistor channel region over which the current is modulated. In effect, the higher breakdown electric field of a wide band gap semiconductor is exploited. In microwave transistors, the ability to support high voltage is particularly desirable since, generally, power has to be transferred to a relatively high impedance (for example 50Ω) load.


[0004] WO 2006/117621 A1 discloses a metal semiconductor field-effect transistor (MESFET). The MESFET is manufactured by providing a single crystal diamond material substrate having a growth surface on which further layers of diamond material can be deposited, depositing a plurality of further diamond layers on the substrate growth surface, and attaching appropriate contacts to the respective diamond layers, thereby defining a transistor structure. The further diamond layers deposited on the substrate include a boron doped interface layer (a “delta-doped” layer). Such a design presents several synthesis challenges. The main challenge is the requirement to produce nanometer-thin boron layers which transition very abruptly to an intrinsic layer (e.g. a change in B concentration from about 10^{13} B atoms per cm² to about 10^{20} B atoms per cm² in a few nm). Growing such boron layers (delta layers) is dependent upon a number of crucial steps including substrate surface preparation for flatness and smoothness and diamond growth conditions. In this type of device, the holes (acting as charge carriers) are essentially localised in a thin intrinsic diamond layer in the immediate vicinity of the boron acceptors in the delta layer.

[0005] An alternative design, described in co-pending application number GB0701186.9 provides a structure in which the charge carriers and ionised acceptors/donors are spatially separated leading to particular advantages in terms of device manufacture and performance. This is achieved by putting a polar layer in contact with the diamond surface in order to substantially confine the carriers in the diamond within a thin diamond surface layer adjacent to the polar layer.

[0006] Work has also taken place on diamond surface devices. These are not generally perceived as being practical devices in the long term, because they are in general intrinsically unstable, but they do offer a route to characterising the behaviour of diamond. A surface device utilises the fact that under certain circumstances a hydrogen terminated diamond surface has free carriers in a surface layer formed by band bending which can then be used in the fabrication of a device. The instability arises in these devices because further species need to be adsorbed to the hydrogen terminated surface in order to induce the band bending, and these species, and the hydrogen termination itself, can be lost, for example if the device is heated.

[0007] Preparation of diamond surfaces has historically focused on providing flat surfaces. Flat surfaces in diamond can generally only be prepared in the first instance by mechanical processing. Subsequently any further treatment tends to roughen or pit the surface because of anisotropic behaviour. WO 01/06633 reported that in homoepitaxial CVD diamond synthesis there is benefit in mechanically preparing a substrate surface which is flat and where the process is optimised to minimise sub-surface damage. Subsequently these surfaces are etched using an anisotropic etch such as a hydrogen etch or an oxygen etch prior to synthesis (preferably in-situ and immediately preceding growth), and this etch, being anisotropic reveals the sub-surface damage in the form of pits, so that synthesis takes place on a surface of reduced surface damage, but which is no longer completely flat, being roughened or pitted by the etch. This relatively damage free but etch roughened surface is then suitable for growth according to that disclosure.

[0008] WO 2006/117621 reveals that in fabrication of some electronic devices mechanical processes can be used to obtain parallel faces to the electronic material, and that this processing can be optimised to achieve both flatness or smoothness and the minimisation of subsurface damage, although the latter is not eliminated.

[0009] Electronic devices are manufactured in a number of materials. Typically fabrication of electronic devices comprises the preparation of a substrate and the synthesis of one or more ‘epi’ or epitaxial layers on this substrate. The epitaxial layers can differ from the substrate in a number of ways:

[0010] Higher purity and/or lower dislocation content, since these can be difficult to control in bulk grown substrate material

[0011] Dopant concentrations, for example the substrate can be insulating to provide isolation, and the epilayers doped to provide the active device regions.

[0012] In the case of heteroepitaxial layers, the basic material in the epilayer can be different.

[0013] The situation in diamond is different:

[0014] The highest purity material can be grown in thick layers, although the final surface of such thick layers is not flat.

[0015] Any interface, or new start of growth, in the diamond can be a source of generation of new dislocations, so the number of interfaces is in general minimised.

[0016] True single crystal diamond cannot be grown heteroepitaxially, so a diamond single crystal substrate is always used. Heteroepitaxial material can sometimes be described as single crystal from, for example, visual inspection of the growth surface, but still retains regions of crystal misoriented with respect to one another and separated by low angle boundaries.

[0017] One area of similarity between diamond and more conventional electronic materials is that diamond can be
doped, typically using boron. Doped layers are generally formed by CVD growth, generally in a separate growth stage to the intrinsic layer.

SUMMARY OF THE INVENTION

[0018] The present invention provides a diamond electronic device comprising a functional interface between two solid materials, wherein the interface is formed by a planar first surface of a first layer of single crystal diamond and a second layer formed on the first surface of the first diamond layer, the second layer being solid, non-metallic and selected from diamond, a polar material and a dielectric material, and wherein the planar first surface of the layer of single crystal diamond has an R_a of less than 10 nm and has at least one, preferably at least two, preferably at least three, preferably all four of the following characteristics:

[0019] (a) the first surface is an etched surface, preferably an isotropically etched surface;

[0020] (b) a density of dislocations in the first diamond layer breaking the surface is less than 400 cm^{-2} measured over an area greater than 0.014 cm^2;

[0021] (c) a density of dislocations in the second layer breaking a notional or real surface lying within the second layer parallel to the interface and within 50 μm of the interface is less than 400 cm^{-2} measured over an area greater than 0.014 cm^2; and

[0022] (d) the first surface has an R_a less than 1 nm.

[0023] Features (a)-(d) refer to the preparation of the diamond surface, and it is generally preferred that the diamond surface has at least one, more preferably 2 (two), more preferably 3 (three), more preferably all 4 (four) of the characteristics (a)-(d).

[0024] In addition to having at least one of the characteristics (a) to (d), preferably the functional interface of the diamond electronic device of the present invention has regions with a layer of charge carriers adjacent thereto such that the charge carriers form the active device current where, in use, the charge carriers either move substantially parallel to the interface or the charge carriers move substantially perpendicular to and through the interface.

[0025] An interface prepared according to the above method will be termed a ‘damage free planar interface’.

[0026] Preferably the interface formed, by a planar first surface of a first layer of single crystal diamond and a second layer formed on the first surface of the first diamond layer, is an internal interface.

[0027] In a further aspect, the present invention provides a diamond electronic device comprising a functional interface between two solid materials, wherein the interface is formed by a planar first surface of a first layer of single crystal diamond wherein the planar first surface has been mechanically processed and a second layer formed on the first surface of the first diamond layer, the second layer being solid, non-metallic and selected from diamond, a polar material and a dielectric material, and wherein the planar first surface of the first layer of single crystal diamond has an R_a of less than 10 nm and wherein the planar surface of the first layer of single crystal diamond is substantially free of residual damage due to mechanical processing.

[0028] Preferably the number density of defects revealed by a revealing etch in the functional planar surface is less than about 100 nm^2, preferably less than about 50 per mm^2, preferably less than about 20 per mm^2, preferably less than about 10 per mm^2, preferably less than about 5 per mm^2.

[0029] Preferably the planar surface of the first layer of single crystal diamond material is prepared from a processed surface, preferably a mechanically processed surface, preferably a mechanically prepared surface.

[0030] As used herein, the term “mechanically processed” means that the surface has been subjected to a step involving conventional polishing and lapping techniques. As used herein, the term “mechanically prepared” refers to a surface that has been mechanically processed such that it is suitable for a specific intended purpose. This might include processing by a route optimised to minimise the amount of subsurface damage as opposed to an arbitrary combination of lapping and polishing steps.

[0031] In a further aspect, the present invention provides a method for producing a diamond electronic device comprising providing a diamond layer having a thickness of greater than about 20 μm; preparing a first surface of the diamond layer by mechanical means to have a surface roughness R_a of less than about 10 nm; etching the first surface of the diamond layer to form a planar first surface having a surface roughness R_a of less than about 10 nm; and forming a second layer on the planar first surface of the diamond layer forming a functional interface between the diamond layer and the second layer, wherein the second layer is solid, non-metallic and selected from diamond, a polar material and a dielectric material.

[0032] In a further aspect, the present invention provides a method for producing a diamond electronic device comprising providing a diamond layer having a thickness of greater than about 20 μm; preparing a first surface of the diamond layer by mechanical means to have a surface roughness R_a of less than about 10 nm; growing a thin layer of diamond, preferably having a thickness of less than about 20 μm, on the first surface of the diamond layer to form a planar first surface having a surface roughness R_a of less than about 10 nm; and forming a second layer on the planar first surface of the diamond layer to form a functional interface between the diamond layer and the second layer, wherein the second layer is solid, non-metallic and selected from diamond, a polar material and a dielectric material.

[0033] Preferably the diamond layer is single crystal diamond.

[0034] In the context of this invention, a planar interface is an interface which is not necessarily flat over large dimensions, e.g. over dimensions larger than about 1 μm, more preferably larger than about 10 μm, more preferably larger than 100 μm, more preferably larger than about 1 μm, but on this scale may show a degree of curvature. However the interface is planar because it is free of sharp features which may degrade the performance of the device by causing scattering of the charge carriers. In particular, the first surface of the first layer, and preferably the interface formed on it, preferably has root-mean-square roughness R_a of less than about 10 nm, preferably an R_a of less than about 5 nm, preferably an R_a of less than about 3 nm, preferably an R_a of less than about 2 nm, preferably an R_a of less than about 1 nm, preferably an R_a of less than about 0.5 nm. Furthermore, the surface of the second layer facing the first layer preferably has an R_a of less than about 10 nm, preferably an R_a of less than about 5 nm, preferably an R_a of less than about 3 nm, preferably an R_a of less than about 2 nm, preferably an R_a of less than about 1 nm, preferably an R_a of less than about 0.5 nm,
preferably an $R_p$ of less than about 0.3 nm, preferably an $R_y$ of less than about 0.2 nm, preferably an $R_y$ of less than about 0.1 nm.

[0035] A functional interface is one which forms part of the operational design of the device, such that in the absence of the interface the design of the device would be different and/or its operation would be significantly changed. More specifically, the charge carriers which are, in use, the active current of the device, move in proximity to the functional interface, either substantially parallel thereto or substantially perpendicular therethrough.

[0036] The electronic device of this invention can comprise natural single crystal diamond, synthetic single crystal diamond made by high pressure-high temperature (HPHT) techniques, and synthetic single crystal diamond made by CVD techniques (‘single crystal CVD diamond’). Alternatively it may comprise a combination of these, for example a first layer comprising boron doped HPHT diamond providing a first surface, and single crystal CVD diamond providing a second layer.

[0037] Preferably the first layer of the electronic device of this invention comprises single crystal CVD diamond. Preferably where the second layer is diamond this comprises single crystal CVD diamond.

[0038] Preferably, either the first layer and/or the second layer, between which there is the interface, is high purity single crystal diamond, preferably high purity single crystal CVD diamond.

[0039] The high purity single crystal diamond preferably has a total impurity content, excluding hydrogen and its isotopes of about 5x10$^{16}$ atoms per cm$^3$ or less, preferably about 1x10$^{15}$ atoms per cm$^3$ or less, preferably about 5x10$^{15}$ atoms per cm$^3$ or less.

[0040] Alternatively or in addition, the high purity single crystal diamond has a nitrogen content of about 5x10$^{17}$ atoms per cm$^3$ or less, preferably about 1x10$^{17}$ atoms per cm$^3$ or less, preferably about 5x10$^{16}$ atoms per cm$^3$ or less, preferably about 1x10$^{16}$ atoms per cm$^3$ or less.

[0041] Alternatively or in addition, the high purity single crystal diamond has a boron content of about 1x10$^{17}$ atoms per cm$^3$ or less, preferably about 1x10$^{17}$ atoms per cm$^3$ or less, preferably about 5x10$^{15}$ atoms per cm$^3$ or less, preferably about 1x10$^{15}$ atoms per cm$^3$ or less.

[0042] The total impurity, nitrogen and boron concentrations can be measured by techniques including secondary ion mass spectrometry (SIMS). SIMS can be used to provide bulk impurity concentrations and to provide ‘depth profiles’ of the concentration of an impurity. The use of SIMS is well known in the art, for example the measurement of boron concentrations by SIMS is disclosed in WO 03/052174.

[0043] The interface may be formed by etching or regrowth. Preferably the interface is formed by etching, preferably by isotropic etching. Where the interface is formed by isotropic etching preferably it is prepared by ICP etching using a gas mixture containing a halogen and an inert gas. Preferably the halogen is chlorine and the inert gas is argon.

[0044] Advantageously, by use of the technique of isotropic etching, the surface(s) which form the interface are etched at approximately the same rate irrespective of crystal orientation. This is particularly advantageous as it means that the interface may be formed from single crystal or polycrystalline diamond. This also means that the surface(s) can be etched without preferentially removing the damaged regions, as would otherwise be the case were an anisotropic etch to be used. Thus, the isotropic etch removes damage from the surface without significantly roughening the surface.

Etching

[0045] An etched surface means the removal of a minimum thickness of material from the surface.

[0046] In one embodiment, an etched surface means the removal of a minimum thickness of material from an as mechanically processed surface, preferably a mechanically prepared surface, based on grit size of last mechanical process, to provide a surface which is free or substantially free of mechanical processing damage, and is also free or substantially free of damage etch features.

[0047] As indicated above, an isotropically etched surface means that the surface roughness of the surface is not substantially increased by the etch. Surface roughness measurements $R_p$ and $R_y$ are taken on the same area of the diamond. By “same area” is meant an equivalent area as close as reasonably practical, using multiple measurements and statistical analysis where necessary to verify the general validity of the measurements, as is known in the art. In particular the isotropically etched surface of the invention has a roughness $R_y$ (After the etch) and the original surface a roughness $R_y$ (Before the etch), such that $R_y$ is preferably less than about 1.5, more preferably less than about 1.4, more preferably less than about 1.2, more preferably less than about 1.1, and in addition, the isotropic etch preferably provides at least one, preferably at least two of the following features:

[0048] an etched surface which is smooth and preferably smoother than the initially prepared surface, and in particular where the $R_y$ of the etched surface ($R_y$) is preferably less than about 10 nm, preferably less than about 5 nm, preferably less than about 2 nm, preferably less than about 1 nm, preferably less than about 0.5 nm, preferably less than about 0.3 nm.

[0049] Removal of a thickness of material exceeding at least about 0.2 μm, preferably at least about 0.5 μm, preferably at least about 1.0 μm, preferably at least about 2 μm, preferably at least about 5 μm, preferably at least about 10 μm.

[0050] Removal, by etching, of a minimum thickness of material from the as mechanically processed surface based on grit size of last mechanical process, to provide a surface which is free or substantially free of mechanical processing damage, requires the removal of sufficient depth to significantly reduce the surface damage and thus needs removal by etching of the same order of thickness as the surface damage layer. Typically surface damage layers have thicknesses in the range of about 0.2 μm to about 20 μm (or thicker with very aggressive lapiadary techniques). Thus preferably the etch removes a thickness of material from the surface, where the thickness of material removed is at least about 0.2 μm, more preferably at least about 0.5 μm, more preferably at least about 1.0 μm, more preferably at least about 2 μm, more preferably at least about 5 μm, more preferably at least about 10 μm. The surface damage layer typically has a thickness that is about the same as the size of the largest diamond grit particle used for the last stage of lapiadary processing; for example a surface scale polished with 1-2 μm sized diamond grit will typically have a surface damage layer about 2 μm thick. Therefore, to minimise the amount of damage from lapiadary processing that remains after etching by the method of the invention, the amount of material removed by the method of the invention should preferably be at least about
0.2 times the size of the largest grit particles, more preferably at least about 0.5 times the size of the largest grit particles, more preferably at least about 0.8 times the size of the largest grit particles, more preferably at least about 1.0 times the size of the largest grit particles, more preferably at least about 1.5 times the size of the largest grit particles. After the etch, the surface of the single crystal diamond preferably has a surface roughness after the etch, \( R_s \), of less than about 10 nm, more preferably less than about 5 nm, more preferably less than about 2 nm, more preferably less than about 1 nm, more preferably less than about 0.5 nm, more preferably less than about 0.3 nm.

[0051] Where the interface is formed by etching it can extend across the whole of a surface of the first diamond layer, or across a proportion of the surface such as structural features etched into the surface, using known techniques such as photolithography, this portion of the surface then forming the first surface.

[0052] Where the interface is formed by etching, the interface is preferably a functional interface in the design of the electronic device, and is preferably one of the following interfaces deemed to be an internal surface or interface of the final device:

- [0053] a diamond to diamond interface, such as intrinsic diamond to boron doped diamond, or vice versa, or between two diamond layers of different doping concentration, where a dopant concentration changes across the interface by at least a factor of about 2, preferably by at least a factor of about 5, preferably by at least a factor of about 10, preferably by at least a factor of about 20;
- [0054] a diamond to diamond interface where the level of at least one impurity changes at the interface, such that:

- [0055] the impurity concentration in at least one layer is greater than \( 10^{19} \) atoms/cm\(^2\), preferably greater than \( 3 \times 10^{19} \) atoms/cm\(^2\), preferably greater than \( 10^{18} \) atoms/cm\(^2\), or
- [0056] where the change in impurity concentration at the interface is by at least a factor of about 5, preferably by at least a factor of about 10, preferably by at least a factor of about 30, preferably by at least a factor of about 100, and preferably where the impurity is other than hydrogen;
- [0057] a diamond to non-diamond polar material interface;
- [0058] a diamond to a non-diamond dielectric material interface.

[0059] Where the interface is formed by etching, more preferably the interface is functional interface in the design of the electronic device, and is preferably one of the following interfaces deemed to be an internal surface or interface of the final device:

- [0060] a diamond to diamond interface, such as intrinsic diamond to boron doped diamond, or vice versa, or between two diamond layers of different doping concentration, where a dopant concentration changes across the interface by at least a factor of about 2, preferably by at least a factor of about 5, preferably by at least a factor of about 10, preferably by at least a factor of about 20;
- [0061] a diamond to diamond interface where the level of at least one impurity changes at the interface, such that:

- [0062] the impurity concentration in at least one layer is greater than \( 10^{15} \) atoms/cm\(^2\), preferably greater than \( 3 \times 10^{15} \) atoms/cm\(^2\), preferably greater than \( 10^{14} \) atoms/cm\(^2\), preferably greater than \( 10^{13} \) atoms/cm\(^2\), or
- [0063] where the change in impurity concentration at the interface is by at least a factor of about 5, preferably by at least a factor of about 10, preferably by at least a factor of about 30, preferably by at least a factor of about 100, and preferably where the impurity is other than hydrogen;
- [0064] a diamond to non-diamond polar material interface.

[0065] Where the interface is formed by etching, more preferably the interface is functional interface in the design of the electronic device, and is preferably one of the following interfaces deemed to be an internal surface or interface of the final device:

- [0066] a diamond to diamond interface, such as intrinsic diamond to boron doped diamond, or vice versa, or between two diamond layers of different doping concentration, where a dopant concentration changes across the interface by at least a factor of about 2, preferably by at least a factor of about 5, preferably by at least a factor of about 10, preferably by at least a factor of about 20;
- [0067] a diamond to diamond interface where the level of at least one impurity changes at the interface, such that:

- [0068] the impurity concentration in at least one layer is greater than \( 10^{15} \) atoms/cm\(^2\), preferably greater than \( 3 \times 10^{15} \) atoms/cm\(^2\), preferably greater than \( 10^{14} \) atoms/cm\(^2\), preferably greater than \( 10^{13} \) atoms/cm\(^2\), preferably greater than \( 10^{12} \) atoms/cm\(^2\), or
- [0069] where the change in impurity concentration at the interface is by at least a factor of about 5, preferably by at least a factor of about 10, preferably by at least a factor of about 30, preferably by at least a factor of about 100, and preferably where the impurity is other than hydrogen.

[0070] Furthermore the etched diamond surface with low \( R_s \) preferably is substantially free of processing damage such that the number of defects revealed by the revealing etch test is less than about 100 per mm\(^2\).

[0071] In the context of this invention the term 'impurity' refers to atoms other than C\(^5\)-bonded carbon (that is carbon bonded as diamond) or hydrogen (and their isotopes) that are either intentionally or unintentionally present in the diamond of the invention. A dopant is such an impurity added to modify the electronic properties of the diamond, and the material containing the dopant described as 'doped diamond'. An example of an impurity which is intentionally present in the invention is boron, which is added so as to provide a source of carriers and is thus a dopant. An example of an impurity which may be unintentionally present in the invention is nitrogen, which may have been incorporated as a result of being present in the source gases used for synthesis or as a residual gas in the CVD synthesis system.

[0072] Impurity concentrations can be measured by techniques including secondary ion mass spectroscopy (SIMS). SIMS can be used to provide bulk impurity concentrations and to provide 'depth profiles' of the concentration of an impurity. The use of SIMS is well known in the art, for
example the measurement of boron concentrations by SIMS is disclosed in WO 03/052174.

Regrowth

[0073] Formation of the interface by regrowth is advantageous because it has the effect of distancing any damaged layer(s) from the surface(s) which forms the functional interface(s) of the device.

[0074] Where the interface is formed by growth it can be restricted to a portion of a surface of the first diamond layer by using masking techniques, this portion corresponding to the first surface, or, more preferably, it can extend across the whole of a surface of the first diamond layer, this whole surface forming the first surface.

[0075] As growth is a much slower process than etching, e.g., \(-1\) \(\mu\)m/hr as compared to \(-0.1\) \(\mu\)m/min, there is greater scope for the control of the thickness of the layer.

[0076] In some circumstances, the technique of regrowth may be more attractive than an etching technique, specifically where it is possible to reduce the effect of mechanical damage sufficiently by regrowth alone. An example of such a situation might be the deposition of a buffer layer on to a substrate where the charge carriers do not move in the buffer layer.

[0077] An interface formed by regrowth means growing a new thin diamond layer, where the surface of this thin layer is then used as the first surface in its as grown state.

[0078] The interface between the mechanically processed surface and the regrowth layer preferably does not itself serve as an inherent part of the device design (or as a functional surface) other than to provide a layer of material to displace or separate an interface which is designed to act as an interface in the electronic device design (a functional interface) away from an interface where there is mechanical processing damage.

[0079] Such a thin diamond layer is preferably grown by CVD synthesis, and is thin to limit the formation of macroscopic growth steps. The thickness of this layer, grown onto a previously mechanically prepared surface, is less than about 20 \(\mu\)m, preferably less than about 10 \(\mu\)m, preferably less than about 3 \(\mu\)m, preferably less than about 1 \(\mu\)m, preferably less than about 100 nm, preferably less than about 50 nm, preferably less than about 20 nm, preferably less than about 10 nm.

[0080] Such a thin layer may be prepared using a number of techniques including monolayer growth techniques and use of off-axis surfaces to control the propagation of surface steps, and thus retain a very flat and smooth surface.

[0081] Where the surface upon which the thin layer is grown has Miller indices close to those of a \{001\} surface, this being the surface upon which homoepitaxial CVD diamond growth is most easily accomplished, the normal to the surface is preferably between 0° and 5°, preferably between about 0.5° and about 1°, of the normal to a \{001\} or a \{111\} surface. Where the surface is close to a \{001\} surface, the normal to the surface is preferably within about 10° of the great circle passing through the pole of the \{001\} surface and the pole of an adjacent \{101\} surface.

[0082] Such a thin layer may comprise high purity intrinsic diamond, more preferably comprising high purity intrinsic diamond with material properties conforming to the disclosures in WO 01/96633.

[0083] Alternatively such a thin layer may comprise conductive doped diamond, for example B doped diamond.

[0084] The surface of this thin as-grown layer forms the first surface and preferably has an \(R_s\) of less than about 10 nm, preferably an \(R_s\) of less than about 5 nm, preferably an \(R_s\) of less than about 3 nm, preferably an \(R_s\) of less than about 2 nm, preferably an \(R_s\) of less than about 1 nm, preferably an \(R_s\) of less than about 0.5 nm, preferably an \(R_s\) of less than about 0.2 nm, preferably an \(R_s\) of less than about 0.1 nm. Thus, this surface has very low surface roughness and in addition is free of processing damage.

[0085] The prepared surface onto which this layer may be grown could be any form of diamond, but is preferably CVD synthetic diamond, preferably boron doped CVD diamond.

[0086] Furthermore, where the interface is formed by regrowth, preferably the interface is one of the following surfaces deemed to be an internal surface or interface of the final device:

[0087] A conductive doped diamond to conductive doped diamond interface, such as a boron doped diamond to boron doped diamond, where both layers contain a dopant at a concentration preferably greater than about \(10^{17}\) atoms/cm\(^2\), preferably greater than about \(10^{18}\) atoms/cm\(^2\), preferably greater than about \(10^{19}\) atoms/cm\(^2\), preferably greater than about \(10^{20}\) atoms/cm\(^2\), and preferably where any difference in boron doping between the layers is not relevant to device performance and the damaged layer is essentially encapsulated in a region of conducting diamond away from any active device interfaces. Preferably the dopant is boron.

[0088] A diamond to diamond interface, such as intrinsic diamond to intrinsic diamond, where the properties of the diamond either side of the layer are sufficiently similar for the interface not to be designed to act as an interface in the electronic device design. Preferably the intrinsic diamond comprises high purity intrinsic diamond with material properties conforming to the disclosures in WO 01/96633.

[0089] More preferably, where the interface is formed by regrowth, the interface is a conductive doped diamond to conductive doped diamond interface, where both layers contain a dopant at a concentration preferably greater than about \(10^{17}\) atoms/cm\(^2\), preferably greater than about \(10^{18}\) atoms/cm\(^2\), preferably greater than about \(10^{19}\) atoms/cm\(^2\), preferably greater than about \(10^{20}\) atoms/cm\(^2\), and preferably where any difference in boron doping between the layers is not relevant to device performance and the damaged layer is essentially encapsulated in a region of conducting diamond away from any active device interfaces. Preferably the dopant is boron.

Combined

[0090] The techniques of etching and regrowth may be combined, such that a surface is first etched and then a thin layer regrown to form the first surface of the first layer and subsequently the interface. This approach is generally advantageous only if the etch has not been completed to sufficient depth to remove all mechanical processing damage. However, by use of a combination of the two techniques, it is envisaged that it is possible to produce an interface which has minimal surface damage. This is because the damage has first been removed by etching and then any residual damage is distance from the functional interface by the growth of the thin diamond layer.

PREFERRED EMBODIMENTS OF THE INVENTION

[0091] It is desirable that the first layer has a low dislocation density in the region of the first surface. In particular, it is
desirable that the density of dislocations breaking the first surface of the first layer is less than about 400 cm$^{-2}$, preferably less than about 300 cm$^{-2}$, preferably less than about 200 cm$^{-2}$, preferably less than about 100 cm$^{-2}$, measured over an area of greater than about 0.014 cm$^2$, preferably greater than about 0.1 cm$^2$, preferably greater than about 0.25 cm$^2$, preferably greater than about 0.5 cm$^2$, preferably greater than about 1 cm$^2$, and preferably greater than about 2 cm$^2$.

This invention of producing a suitable diamond surface from the as-grown surface of a thick diamond layer, which processing steps are included in the method. In the context of this invention, a single crystal CVD layer is considered to be thick when it has a thickness exceeds about 20 μm.

Firstly, a first surface may be prepared on the thick diamond layer using mechanical lapping and polishing processes, which have been optimised for minimum surface damage by using feedback from, for example, a revealing etch. Such a technique is described in for example WO 01/96633. Whilst such a surface may have a low damage level, it is unlikely to be sufficiently free of damage to obtain more than adequate performance from the device.

The first surface may then be prepared from a processed surface, preferably from a mechanically processed surface, preferably a mechanically prepared surface itself optimised for minimum surface damage by using the method above, by using a further processing stage comprising chemical etch or other forms of etching, such as ion beam milling, plasma etching or laser ablation, and more preferably plasma etching. Preferably the etching stage removes at least about 10 nm, preferably at least about 100 nm, more preferably at least about 1 μm, more preferably at least about 2 μm, more preferably at least about 5 μm, more preferably at least about 10 μm. Preferably the etching stage removes less than about 100 μm, preferably less than about 50 μm, preferably less than about 20 μm.

This further processed surface preferably has an $R_q$ of less than about 10 nm, preferably an $R_q$ of less than about 5 nm, preferably an $R_q$ of less than about 3 nm, preferably an $R_q$ of less than about 2 nm, preferably an $R_q$ of less than about 1 mm, preferably an $R_q$ of less than about 0.5 mm, preferably an $R_q$ of less than about 0.3 nm, preferably an $R_q$ of less than about 0.2 nm, preferably an $R_q$ of less than about 0.1 nm.

Alternatively, the first surface may be prepared from a processed surface, preferably from a mechanically processed surface, preferably a mechanically prepared surface itself optimised for minimum surface damage by using the method above, or from an etched surface such as those described above, by growing a further thin layer of diamond on the surface, preferably using a CVD process. Prior to deposition of the further thin layer of diamond (termed regrowth), the processed surface has an $R_q$ of less than about 10 nm, preferably an $R_q$ of less than about 5 nm, preferably an $R_q$ of less than about 3 nm, preferably an $R_q$ of less than about 2 nm, preferably an $R_q$ of less than about 1 mm, preferably an $R_q$ of less than about 0.5 mm, preferably an $R_q$ of less than about 0.3 nm, preferably an $R_q$ of less than about 0.2 nm, preferably an $R_q$ of less than about 0.1 mm. After deposition of the further thin layer of diamond (termed regrowth), the new as grown regrowth surface has an $R_q$ of less than about 10 nm, preferably an $R_q$ of less than about 5 nm, preferably an $R_q$ of less than about 3 nm, preferably an $R_q$ of less than about 2 nm, preferably an $R_q$ of less than about 1 mm, preferably an $R_q$ of less than about 0.5 mm, preferably an $R_q$ of less than about 0.3 nm, preferably an $R_q$ of less than about 0.2 nm, preferably an $R_q$ of less than about 0.1 nm.

Where the first surface is prepared by plasma etching, preferably the etching is achieved by ICP etching, preferably using a gas mixture containing a halogen and an inert gas, preferably where the inert gas is argon, and preferably where the halogen is chlorine.

The electronic device may be a 2-terminal device, such as a diode.
The electronic device may have at least 3 terminals, such as a 3-terminal transistor.

The electronic device is preferably a transistor, preferably a field effect transistor.

In one embodiment of the present invention, the electronic device comprises a functional interface between two solid materials, wherein the interface is formed by a planar first surface of a first layer of single crystal diamond, wherein the planar first surface has preferably been mechanically processed and subsequently isotropically etched and a second layer formed on the first surface of the first diamond layer, the second layer being solid, non-metallic and selected from diamond, a polar material and a dielectric material, and wherein the planar first surface of the first layer of single crystal diamond has an \( R_{q} \) of less than about 10 nm, preferably an \( R_{q} \) of less than about 5 nm, preferably an \( R_{q} \) of less than about 3 nm, preferably an \( R_{q} \) of less than about 4 nm, preferably an \( R_{q} \) of less than about 2 nm, preferably an \( R_{q} \) of less than about 1 nm, preferably an \( R_{q} \) of less than about 0.5 nm, preferably an \( R_{q} \) of less than about 0.3 nm, preferably an \( R_{q} \) of less than about 0.2 nm, preferably an \( R_{q} \) of less than about 0.1 nm.

In another embodiment of the present invention, the electronic device comprises a functional interface between two solid materials, wherein the interface is formed by a planar first surface of a first layer of single crystal diamond and a second layer formed on the first surface of the first diamond layer, the second layer being solid, non-metallic and selected from diamond, a polar material and a dielectric material, and wherein the planar first surface of the first layer of single crystal diamond has an \( R_{q} \) of less than about 10 nm and wherein the first surface of the diamond layer is a surface of a diamond layer, preferably having a thickness of less than about 20 \( \mu m \), preferably less than about 10 \( \mu m \), preferably less than about 5 \( \mu m \), preferably less than about 3 \( \mu m \), preferably less than about 1 \( \mu m \), preferably less than about 0.5 \( \mu m \), preferably less than about 0.3 \( \mu m \), preferably less than about 0.2 \( \mu m \), preferably less than about 0.1 \( \mu m \), grown on a single crystal diamond layer.

In another embodiment of the present invention, the electronic device comprises a functional interface between two solid materials, wherein the interface is formed by a planar first surface of a first layer of single crystal diamond, wherein the planar first surface has preferably been mechanically processed and subsequently isotropically etched and a second layer formed on the first surface of the first diamond layer, the second layer being solid, non-metallic and selected from diamond, a polar material and a dielectric material, and wherein the planar first surface of the first layer of single crystal diamond has an \( R_{q} \) of less than about 10 nm, preferably an \( R_{q} \) of less than about 5 nm, preferably an \( R_{q} \) of less than about 3 nm, preferably an \( R_{q} \) of less than about 2 nm, preferably an \( R_{q} \) of less than about 1 nm, preferably an \( R_{q} \) of less than about 0.5 nm, preferably an \( R_{q} \) of less than about 0.3 nm, preferably an \( R_{q} \) of less than about 0.2 nm, preferably an \( R_{q} \) of less than about 0.1 nm, and wherein the first surface of the diamond layer is a surface of a diamond layer, preferably having a thickness of less than about 20 \( \mu m \), preferably less than about 10 \( \mu m \), preferably less than about 5 \( \mu m \), preferably less than about 3 \( \mu m \), preferably less than about 1 \( \mu m \), preferably less than about 0.5 \( \mu m \), preferably less than about 0.3 \( \mu m \), preferably less than about 0.2 \( \mu m \), preferably less than about 0.1 \( \mu m \), and wherein the first surface of the diamond layer is a surface of a diamond layer, preferably having a thickness of less than about 20 \( \mu m \), preferably less than about 10 \( \mu m \), preferably less than about 5 \( \mu m \), preferably less than about 3 \( \mu m \), preferably less than about 1 \( \mu m \), preferably less than about 0.5 \( \mu m \), preferably less than about 0.3 \( \mu m \), preferably less than about 0.2 \( \mu m \), preferably less than about 0.1 \( \mu m \), grown on a single crystal diamond layer.

For the purposes of this invention the roughness of a surface is described by its \( R_{q} \) value. \( R_{q} \) is also known as the ‘root mean square’ (or RMS) roughness. \( R_{q} \) is defined as the square root of the mean squared deviations from the centre-line or plane of the surface profile:

\[
R_{q} = \sqrt{(y_1^2 + y_2^2 + \ldots + y_n^2)/n}
\]

where \( y_i \) etc are the squared deviations from the centre-line or plane of the surface profile and \( n \) is the number of measurements.

A surface may also be quantified by its \( R_{q} \) value (also referred as ‘average roughness’ or ‘centre line average’):

\[
R_{q} = \sqrt{(y_1^2 + y_2^2 + \ldots + y_n^2)/n}
\]

where \( y_i \) etc are the moduli of the deviations from the centre-line or plane of the surface profile and \( n \) is the number of measurements.

For a surface with a Gaussian distribution of deviations from the centre-line or plane of the surface profile, the value of \( R_{q} \) is 1.25 \( R_{q} \).

\( R_{q} \) and \( R_{q} \) may be measured along lines (a one-dimensional measurement) or over areas (a two-dimensional measurement). An area measurement is essentially a series of parallel line measurements.

The extent of sub-surface damage can be revealed and quantified using a deliberately anisotropic thermal etching etch. The revealing etch preferentially oxidises regions of damaged diamond and therefore allows such regions to be identified and thereafter quantified. Regions containing sub-surface damage from mechanical processing are typically darkened or even blackened by the revealing etch.

The revealing etch consists of:

(i) examining the surface at a magnification of 50 times using reflected light with a typical metallurgical microscope to ensure that there are no surface features present,

(ii) exposing the surface to an air-butane flame thereby raising the diamond surface to a temperature of typically about 800°C to about 1000°C for a period of about 10 seconds,

(iii) examining the surface at a magnification of 50 times using reflected light with a typical metallurgical microscope and counting the damage features revealed by the revealing etch, in the manner described below, to determine their number density.

(iv) repeating steps (ii) and (iii) and comparing the measured density of defects with that of the previous cycle until the following condition is met: if the number density of defects counted is less than or equal to 150%, preferably less than or equal to 120%, of the number density determined in the previous cycle, then all the defects are deemed to be revealed and the measurement recorded is the average of the measurements of the last two cycles, if not the cycle is repeated again.

The number density of defects is measured by the following method:

(i) the defects to be counted are those defects visible at a magnification of 50 times with a typical
metallurgical microscope which fall totally or partially within a rectangular area 1 mm x 0.2 mm projected onto the surface being characterised,

(ii) the area is selected at random over the surface or portion of the surface to be characterised and randomly oriented,

(iii) the defects are counted in a minimum of 5 such areas,

(iv) the number density of defects is calculated by dividing the total number of defects counted by the total area examined to give a number density in defect per mm².

To measure the number density of defects in areas less than 1 mm² the above method is adapted by completing the defect count over the whole area as a single measurement.

For the surface to be considered to be substantially free of residual damage due to mechanical processing the number density of defects revealed in a surface of single crystal CVD diamond prepared by the method of the invention is less than 100 per mm², preferably less than about 50 per mm², preferably less than about 20 per mm², preferably less than about 5 per mm².

Methods of preparing and characterising diamond and diamond surfaces with low dislocation density are reported in the prior art of WO 01/96633, WO 01/96634, WO 2004/027123, and co-pending application PCT/IB2006/003531. The preferred methods of characterising the dislocation density are the use of a “revealing plasma etch” and the use X-ray topography.

As used herein, the term “about x” is intended to include the value x itself.

EXAMPLE

By way of example, in the case of a diode, preferably the damage free interface functional interface is formed between doped conducting diamond and intrinsic diamond, and is formed by one of the following methods:

By regrowth, wherein the boron doped layer is formed, and a planar surface formed on the doped diamond by lapidary or mechanical processing. A further thin B doped layer is then grown onto this layer, preferably using growth conditions selected to minimise roughening and preferably keeping the layer sufficiently thin and in the thickness range 10 nm-20 μm, more preferably in the thickness range 100 nm-10 μm to minimise roughening, and thus encapsulating the surface with mechanical damage between two regions of doped conducting diamond. Then a high purity intrinsic diamond layer is grown onto the regrown layer surface, this layer preferably comprising high purity intrinsic diamond with material properties conforming to the disclosures in WO01/96633 thus forming a damage free interface between the thin doped conducting diamond layer and a further layer of intrinsic diamond which is displaced from the damage layer encapsulated within the boron doped layer.

By etching, wherein the conducting doped diamond layer is formed, and a planar surface formed on the doped diamond by lapidary or mechanical processing. This surface is then etched, preferably using a plasma etch, more preferably an Argon/Chlorine plasma etch. Optionally, a further thin B doped layer may be regrown onto this layer, preferably using growth conditions selected to minimise roughening and preferably keeping the layer sufficiently thin and in the thickness range 10 nm-20 μm, more preferably in the thickness range 100 nm-310 μm, more preferably in the thickness range 1 μm-10 μm to minimise roughening. Preferably this optional layer is not used. Then a high purity intrinsic diamond layer is grown onto the etched surface, or optional regrown layer surface, this layer preferably comprising high purity intrinsic diamond with material properties conforming to the disclosures in WO 01/96633, thus forming a damage free interface between the conducting diamond layer and a further layer of intrinsic diamond.

Alternatively the diode structure above may be formed between a heavily boron doped layer providing a highly conductive layer, and a lightly boron doped layer providing the reverse voltage hold-off.

In the case of a transistor, preferably the damage free interface is formed by etching or regrowth so that the damage free surface is prepared in the intrinsic diamond. Preferably the damage free surface is parallel to the primary current flow in the device, with this current flow taking place primarily in the intrinsic diamond layer adjacent to the damage free surface, and that current flow is in close proximity to the interface, typically less than 1 μm and more typically less than about 100 nm. Thus, there is a 2-dimensional charge carrier gas, where the meaning of the term “2-dimensional charge carrier gas” is as is normally understood in the art, present in the intrinsic diamond adjacent to the damage free surface.

It will of course be understood that the present invention has been described above purely by way of example, and that modifications of detail can be made within the scope of the invention as defined by the claims.

1. A diamond electronic device comprising a functional interface between two solid materials, wherein the interface is formed by a planar first surface of a first layer of single crystal diamond and a second layer formed on the first surface of the first diamond layer, the second layer being solid, non-metallic and selected from diamond, a polar material and a dielectric material, and wherein the planar first surface of the first layer of single crystal diamond has a surface roughness Rₐ of less than 10 nm and has at least one of the following characteristics:

(a) the first surface is an etched surface;
(b) a density of dislocations in the first diamond layer breaking the first surface is less than 400 cm⁻² measured over an area greater than 0.014 cm²;
(c) a density of dislocations in the second layer breaking a notional or real surface lying within the second layer parallel to the interface and within 50 μm of the interface is less than 400 cm⁻² measured over an area greater than 0.014 cm²; and
(d) the first surface has an Rₐ less than 1 nm.

2. A diamond electronic device according to claim 1, wherein the planar first surface of the first layer of single crystal diamond is a mechanically processed surface.

3. A diamond electronic device comprising a functional interface between two solid materials, wherein the interface is formed by a planar first surface of a first layer of single crystal diamond wherein the planar first surface has been mechanically processed, and a second layer formed on the first surface of the first diamond layer, the second layer being solid, non-metallic and selected from diamond, a polar material and a
dielectric material, and wherein the planar first surface of the first layer of single crystal diamond has a surface roughness $R_s$ of less than 10 nm and wherein the planar surface of the first layer of single crystal diamond is substantially free of residual damage due to mechanical processing.

4. An electronic device according to claim 2, wherein the number density of defects revealed by a revealing etch in the functional planar surface is less than 100 per mm$^2$.

5. A diamond electronic device according to claim 1, wherein the first surface of the first diamond layer is an etched surface.

6. A diamond electronic device according to claim 5 where the etched first surface is an isotropically etched surface.

7. An electronic device according to claim 6, wherein the first surface has been isotopically etched using a gas mixture comprising a halogen and an inert gas.

8. An electronic device according to claim 6, wherein the etch removed at least 0.2 μm from the first surface of the first diamond layer.

9. An electronic device according to claim 8, wherein the etch removed at least 0.2x the largest grit particle size used in the last stage of lapidary processing.

10-16. (canceled)

17. A diamond electronic device according to claim 1 wherein the first surface of the diamond layer is a surface of a diamond layer grown on a single crystal diamond layer.

18. A diamond electronic device according to claim 17 wherein the grown diamond layer has a thickness of less than 20 microns.

19. A diamond electronic device according to claim 1 wherein the first surface of the first diamond layer is substantially free from damage introduced by post-growth mechanical processing of an as-grown surface to a depth of at least 1 nm.

20-24. (canceled)

25. A diamond electronic device according to claim 1 wherein diamond on one side of the interface is intrinsic diamond and the diamond on the other side of the interface is boron-doped diamond.

26-31. (canceled)

32. A method for producing a diamond electronic device comprising:

(i) providing a diamond layer having a thickness of greater than about 20 μm;
(ii) preparing a first surface of the diamond layer by mechanical means to have a surface roughness $R_s$ of less than about 10 nm;
(iii) etching the first surface of the diamond layer to form a planar first surface having a surface roughness $R_s$ of less than about 10 nm; and
(iv) forming a second layer on the planar first surface of the diamond layer to form a functional interface between the diamond layer and the second layer, wherein the second layer is solid, non-metallic and selected from diamond, a polar material and a dielectric material.

33. A method for producing a diamond electronic device comprising:

(i) providing a diamond layer having a thickness of greater than about 20 μm;
(ii) preparing a first surface of the diamond layer by mechanical means to have a surface roughness $R_s$ of less than about 10 nm;
(iii) growing a thin layer of diamond, preferably having a thickness of less than about 20 μm, on the first surface of the diamond layer to form a planar first surface having a surface roughness $R_s$ of less than about 10 nm; and
(iv) forming a second layer on the planar first surface of the diamond layer to form a functional interface between the diamond layer and the second layer, wherein the second layer is solid, non-metallic and selected from diamond, a polar material and a dielectric material.

34. The method according to claim 32, wherein the etch is an isotropic etch.

35. The method according to claim 34, wherein in step (iii), at least about 10 nm is removed.

36. The method according to claim 32, wherein the diamond layer is a boron-doped single crystal diamond.

37. (canceled)

38. The method according to claim 33, wherein the thin layer of diamond on the first surface has a thickness of less than 20 μm.

39. The method according to claim 34, wherein a gas mixture comprising a halogen and an inert gas is used in the isotropic etch.