JOURNAL OF APPLIED PHYSICS 114, 114503 (2013)

Analysis of diamond surface channel field-effect transistors with AlN passivation layers

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(Received 25 June 2013; accepted 13 August 2013; published online 17 September 2013)

Diamond surface channel field effect transistors were passivated with thin AlN layers grown by metal-organic chemical vapor deposition in order to improve the chemical stability of the surface-near p-type channel. Electrical characterization showed that the surface-near conductivity in the diamond is preserved during AlN overgrowth if the process temperature does not exceed 800 °C. However, the sheet carrier density is decreased by a factor of about 5 compared to the unpassivated hydrogen-terminated surface. A combination of TEM and XPS analysis showed that this effect is not induced by a partial modification of the surface termination or by a polarization of the AlN passivation. The preserved, but reduced surface-near conductivity in the diamond can however be explained by a hydrogen double bond between the diamond and the AlN film. Field-effect transistor structures fabricated on the passivated diamond substrates showed stable operation up drain-source voltages to −70 V and might therefore be promising candidates for future high-voltage applications.
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INTRODUCTION

Diamond surface channel field effect transistors (FETs) based on the hydrogen surface termination are considered as promising devices in particular for power applications. The hydrogen termination induces a surface-near p-type channel with a hole sheet charge density in the range of 10^{13} cm^{-2} and charge carrier mobilities of about 150 cm^2/Vs.[1] In this case, the two-dimensional hole gas (2DHG) is generated by the field effect through equilibrium with the surface, usually associated with the electrochemical potential of adsorbates on a humidity saturated surface.[2] Both the adsorbates and the hydrogen termination are required for the presence of the p-type channel. This implies that the channel is thermally and chemically instable and very sensitive to the ambient environment. Exposure to vacuum results in desorption of the adsorbates and therefore to a reversible loss of the channel, whereas surface oxidation leads to a modification of the surface termination and therefore to an irreversible degradation of the surface-near channel. The solution for this problem can be the development of a suitable passivation layer of the free diamond surface. The passivation layer either stabilizes or replaces the adsorbates in this case. In previous studies, it has been shown that aluminum oxide grown by atomic layer deposition (ALD)[3,4] and aluminum nitride grown by ALD or metal-organic chemical vapor deposition (MOCVD)[5–8] are promising materials for this purpose. One important aspect, which has, however, not been investigated in detail so far, is the bonding between the initially hydrogen-terminated surface and the passivation layer. Van der Waals bonds are expected to be mechanically instable and should therefore be avoided. On the other hand, it had been proposed by Speranza[9] and Kueck *et al.*[9] to improve the mechanical bonding strength between diamond and the passivation by the formation of a H-double bond by splitting up the hydrogen electron wave package into two packages of opposite directions at the interface to the passivation dielectric. Hence, a direct bond between the H-terminated diamond surface and the first atomic layer of the passivation can be provided. This might also explain the reduced current densities for diamond FETs with AlN passivation[8] compared to unpassivated devices, since a hydrogen double bond is expected to weaken the C-H dipole. However, similar effects could be expected for a partial oxygen termination, where the hydrogen-terminated areas induce the conductivity and the oxygen-terminated areas the adhesion of the AlN on the diamond surface. Another important aspect is that hexagonal AlN is a polar material, where the polarization can enhance or partially compensate the C-H dipole. In this work, the focus was on the deposition of AlN at by MOCVD on (111) orientated single-crystal diamond substrates temperatures above the desorption temperature of common adsorbates, avoiding any oxygen termination. The diamond/AlN interface and the AlN layer itself were analyzed by X-ray photoelectron spectroscopy (XPS) and high-resolution transmission electron microscopy (HR-TEM) measurements. The electrical characteristics were analyzed by transmission line model (TLM) and FET measurements. The aim of the study was to investigate how the surface-near conductivity can be preserved and stabilized by using an AlN passivation layer.

AlN GROWTH AND FET FABRICATION

Intrinsic diamond buffer layers were grown on (111)-orientated Ib single-crystal diamond substrates (Sumitomo) by microwave plasma CVD. In the next step, thin AlN layers

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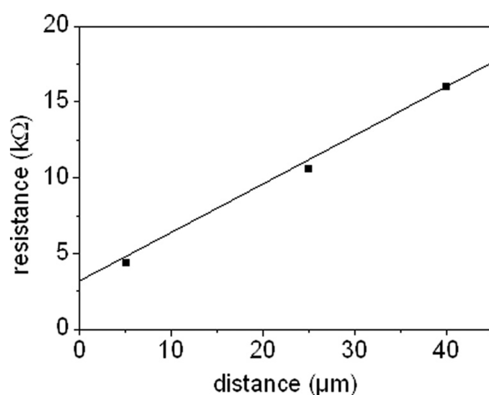

FIG. 1. TLM measurements of a diamond sample with AlN passivation layer grown by MOCVD at 800 °C. The width of the TLM structures was 100 μm.

of 30–40 nm in thickness were grown by MOCVD in an Aixtron 200 RF reactor, using trimethyl aluminum (TMA) and ammonia as precursors as well as hydrogen as carrier gas. The growth temperature was set to 800 °C, where significantly higher temperatures lead to the irreversible loss of the p-type channel in the diamond. The ramp up to the process temperature was also performed under hydrogen atmosphere, followed by an exposure to hydrogen/ammonia atmosphere for about 2 min prior to the onset of the AlN growth (opening of the TMA supply), similar to previous experiments.[8] This pre-treatment will be denoted as "nitridation" in the following. The pressure during AlN growth was 74 millibars, the gas fluxes for TMA and ammonia were 2.4 μmoles/min and 14.3 mmoles/min, respectively. The thickness of the AlN layer after 25 min growth was in the range of 35–40 nm.

The local structure of the AlN layer and interface AlN/diamond were investigated by TEM using an aberration corrected FEI Titan 80–300 microscope. Prior to the TEM investigations an electron transparent lamella was prepared using a focused Ga-beam within a FIB/SEM instrument (Zeiss NVision 040). For details about the FIB-milling, see Ref. 10.

The elemental composition of the sample surfaces was determined by XPS measurements using monochromatized Al K_α (1486.6 eV) radiation (PHI 5800 MultiTechnique ESCA System, Physical Electronics). The measurements were done with a detection angle of 45°, using pass energies at the analyzer of 93.9 and 29.35 eV for survey and detail spectra, respectively. In order to compensate for charging effects at the surface, the samples were neutralized with electrons from a flood gun (current of 3 μA). The binding energy of the XPS peaks was referenced to the C1s signal of bulk diamond at 284.0 eV.

The FET fabrication started with the deposition of Ti/Al/Ni/Au ohmic contacts directly on top of the AlN film with subsequent annealing, similar to comparable experiments.[6,7] Mesa etching was performed by sputtering in Ar/O_2 plasma, where the active FET region and the ohmic contacts were protected by a photoresist mask. Finally, Al gate contacts were deposited using electron beam lithography. The gate width was scaled between 25 μm and 75 μm, the gate length between 0.25 μm and 1.0 μm.

RESULTS AND DISCUSSION

Fig. 1 shows the results of TLM measurements of a passivated diamond structure which was fabricated as described above. The width of the TLM structures was 100 μm. From the slope, a sheet resistance of about 30 kΩ was extracted, which is in the same range as shown in a previous study[8] and therefore higher compared to unpassivated surface-near channel diamond devices (see also below). As stated above, the reduced conductivity can be explained by (a) a partial substitution of the hydrogen termination, e.g., with nitrogen or oxygen, (b) an AlN polarization, counter-acting the C-H surface dipole, or (c) a hydrogen double bond. Fig. 2(a) shows a bright-field TEM image of the AlN layer on top of the diamond substrate. The black layer above the AlN layer is a gold protection layer necessary to avoid surface damaging during FIB-milling. The 40 nm thick AlN layer has surface roughness in the order of 3 nm. The HR-TEM investigations (Fig. 2(b)) and local diffractograms (Fig. 2(c)) showed that the AlN films are polycrystalline with no preferential orientation between the c-axes of the diamond substrate and the AlN crystallites. The interface between substrate and AlN is very smooth with a roughness smaller than 1 nm. The average grain size of the crystallites was approximately 20 nm, as estimated from dark field analysis. The polycrystalline structure of the AlN can be explained by the high lattice mismatch between diamond and AlN and by

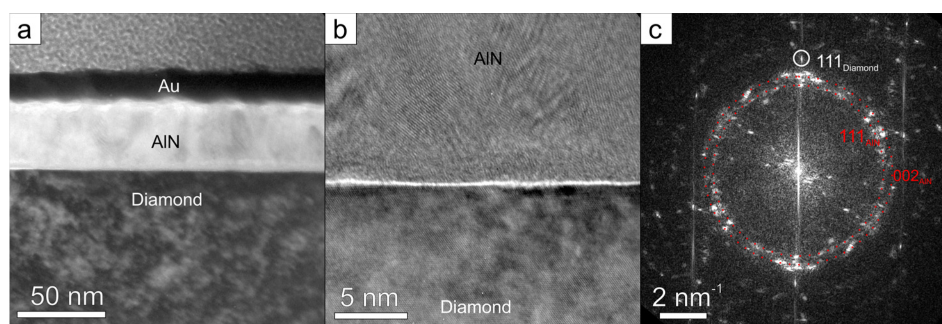

FIG. 2. Investigations of the AlN-diamond interface. (a) Bright-field image showing the 40 nm thin AlN layer on top of diamond (the black layer on top of AlN is a gold protection layer necessary for TEM-sample preparation). (b) HR-TEM image of the interface AlN/diamond showing the polycrystalline nature of AlN. (c) Diffractogram showing the crystal structure of the AlN/diamond interface, 111 and 002 reflections of AlN are marked by the red-dotted circle. There is no preferred relationship to the c-axis of diamond (marked by circle).

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the relatively low growth temperature of the AlN, which was about 200 °C below process temperatures typically used for hexagonal growth on sapphire substrates.[11] Therefore, it seems most likely that the decreased conductivity is not induced by a polarization of the AlN layer. This result is also supported by AlN etching experiments: The deposited AlN films could be wet-chemically etched in AZ 726 photoresist developer. On the one hand, single-crystalline Al-face AlN is expected to be chemically extremely stable, similar to other III-nitrides.[12,13] On the other hand, N-face AlN can be easily etched,[13] would however result in an increased conductivity, since the C-H and the AlN dipole would point in the same directions. Therefore, the relatively low stability of the AlN against wet-chemical etching is explained by the polycrystalline structure. To investigate whether the AlN growth replaces the surface hydrogen, two test samples were analyzed by TLM and XPS measurements: The first sample was exposed to the nitridation treatment at 800 °C under H_2/NH_3 atmosphere in the MOCVD reactor, as described above. One could expect that this treatment might induce a partial nitrogen termination, thus reducing the surface-near conductivity. On the second sample, an AlN layer of 30 nm thickness was subsequently etched in AZ 726 MIF developer. Such an etching treatment does not affect the conductivity of the free hydrogen-terminated surface, as verified by another test sample. The results of the TLM analysis are shown in Fig. 3(a): The two test samples described above showed sheet resistances of about 14 kΩ and 10 kΩ, respectively. As a reference, an identical hydrogen-terminated diamond sample without any additional surface treatment was measured, showing a sheet resistance of about 7 kΩ. The TLM measurements are shown in Fig. 3(b). Both, the sample after nitridation and the sample after AlN growth and etching show therefore sheet resistances which are slightly higher compared to the reference sample, but significantly lower compared to a diamond sample with AlN.

A similar investigation was performed in the works by Imura *et al.*,[6,7] where diamond surface-channel FETs had been passivated with AlN grown at 1240 °C. The authors found that this "nitridation" treatment even induces a surface conductivity for the case of initially oxygen-terminated diamond surfaces. Similar to our results, subsequent AlN overgrowth decreased the hole density and therefore the surface conductivity.[7] Therefore, these results are in agreement with the investigations shown here, although the process temperatures in both cases are quite different.

More information about the effect of the AlN growth on the diamond surface termination could be extracted from XPS measurements of the two test samples described above. For the sample exposed to the nitridation treatment, the XPS analysis showed no significant contributions (intensity far below 1%) of nitrogen (Fig. 4(a)). Nitrogen would be expected at about 400.0 eV, as in the case of diamond exposed to N_2 plasma.[14] However, oxygen and fluorine with atomic concentrations of 2.7% and 1.6%, respectively, were

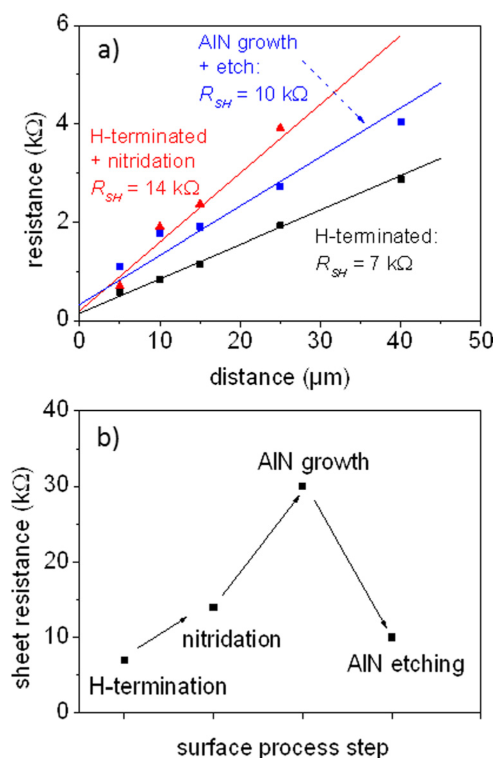

FIG. 3. (a) TLM measurements (contact width = 100 μm) of test samples: after hydrogen termination, after nitridation without AlN growth, after AlN growth and subsequent AlN etching. (b) Evolution of the sheet resistance with process steps.

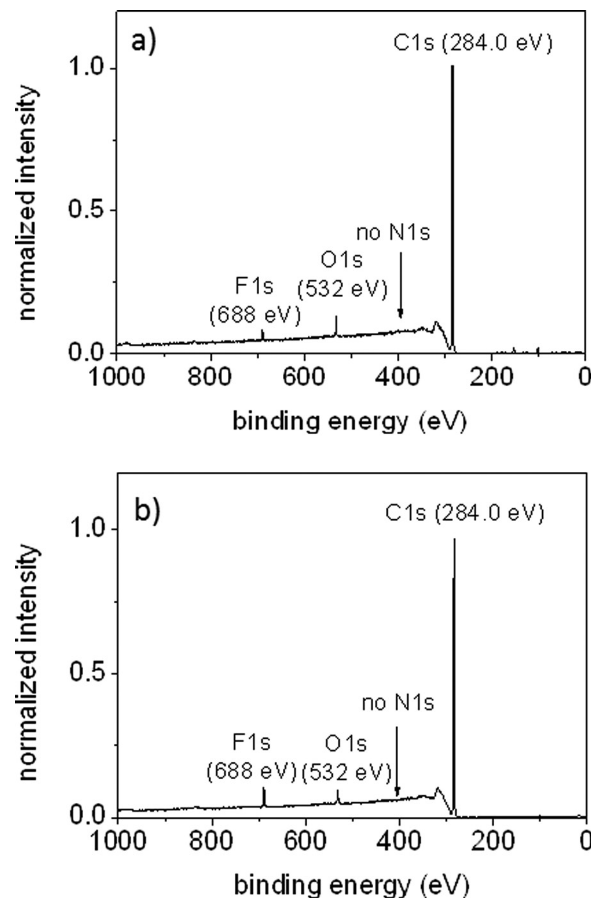

FIG. 4. Results of the XPS analysis for the two diamond test samples: (a) test sample exposed to the nitridation treatment, (b) test sample exposed to AlN growth and subsequent etching. Both XPS spectra show no contributions of nitrogen or aluminum, which indicates that the hydrogen termination is mainly preserved.

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detected, where the origin especially of the fluorine component are not fully clear. However, the main result is that the nitridation treatment does not induce a nitrogen termination, but mainly preserves the hydrogen termination and therefore the surface-near conductivity, as extracted from the TLM measurements of the test sample. To investigate, whether the AlN growth process itself affects the surface termination of diamond, the second test sample (where an AlN film of 40 nm thickness was grown and subsequently etched) was analyzed. Again, no nitrogen and also no aluminum (expected at about 75 eV) components were observed (see Fig. 4(b)). The results of both the TLM and the XPS analysis show that the hydrogen termination is not substituted by nitrogen or aluminum. This supports the idea of a hydrogen double bond at the diamond-AlN interface and in turn explains the reduced conductivity of the passivated compared to the unpassivated surface, since the double bond corresponds to a weaker C-H dipole and therefore to a lower upwards band bending. It can also explain the increased conductivity of the second test samples compared to the passivated diamond sample (Fig. 3(b)), since the removal of the AlN restores again the simple C-H bond on the diamond surface. The small amounts of oxygen and fluorine on the diamond surface might be responsible for the relatively small increase of the sheet resistances for the two test samples compared to the untreated hydrogen-terminated sample.

Since the diamond surface-near conductivity was preserved (although reduced compared to the unpassivated surface) after AlN growth, the diamond samples with AlN passivation could be used for FET fabrication. Figs. 5(a) and 5(b) show the output characteristics of the same FET structure with a gate length and gate width of 1 μm and 50 μm, respectively, measured under different conditions: In Fig. 5(a), the output characteristics were recorded for drain-source voltages up to -10 V. Similar to the results in Ref. 8, the maximum drain current at -3 V gate voltage is about -30 mA/mm. Further accumulation of charge carriers seems to be possible at more negative gate voltages, which would again agree with a reduced surface band bending due to the hydrogen double bond. However, the gate voltage range is limited by forward breakdown of the gate-source diode, since the AlN film still allows parasitic conductivity. Although the AlN layer was not yet fully optimized, FET operation was still possible for drain-source voltages up to -70 V, as shown in Fig. 5(b) for the same device. In this case, the FET showed however non-linear I–V characteristics for low drain-source voltages (except for a gate-source voltage of -3 V). This effect was reversible, which excludes degradation of the ohmic contacts of the FET. In spite of these still not yet solved challenges, these results show that diamond FETs with AlN passivation layer could indeed be a candidate for high-voltage applications.

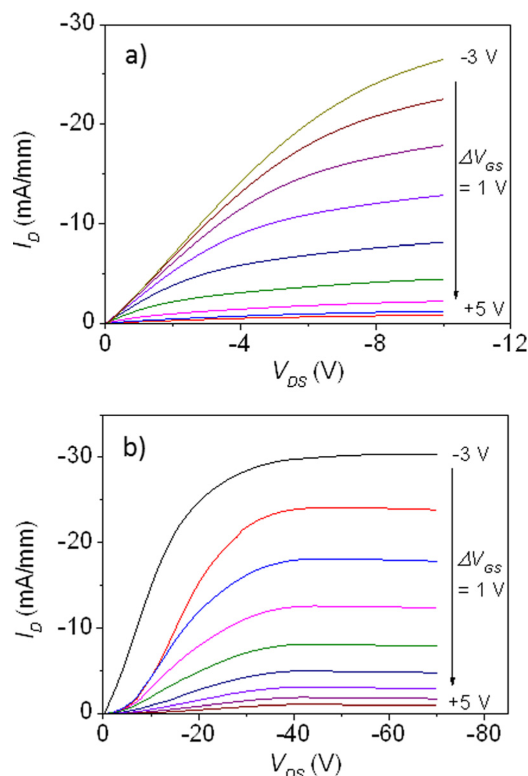

FIG. 5. FET output characteristics of the same FET device with gate length = 1 μm and gate width = 50 mm. Fig. 5(a): Measurement up to -10 V drain-source voltage, Fig. 5(b): Measurement up to -70 V drain-source voltage.

CONCLUSION

Hydrogen-terminated diamond substrates with intrinsic buffer layers have been passivated with 30–40 nm thin AlN films grown by MOCVD at 800 °C in order to stabilize the surface-near p-type channel in the diamond. Further experiments showed already that FET fabrication is possible, but the mechanism why the conductivity is provided after AlN growth (however reduced) was still unclear. A combination of electrical and physical (TEM and XPS measurements) analysis showed that the preserved, but reduced conductivity as compared to unpassivated devices can neither be explained by partial substitution of the hydrogen termination by nitrogen, oxygen, or aluminum, nor by a net polarization of the AlN film itself, since it was found to be polycrystalline with no preferential orientation of the crystallites. A possible and already previously discussed explanation is the formation of a hydrogen double bond between the diamond and the AlN, which explains the electrical characteristics of the passivated structures. The hydrogen double bond ensures a sufficient mechanical stability of the AlN passivation layer on the diamond surface.

FET structures processed on these passivated diamond layers showed full FET operation for drain-source voltages up to -70 V. This shows that such devices might indeed be promising candidates for high-voltage applications.

ACKNOWLEDGMENTS

This work was financially supported by the Deutsche Forschungsgemeinschaft (DFG).

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