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ION BEAM PROCESSING OF DIAMOND

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Ion Beam Processing of Diamond

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ABSTRACT

The objective of this work was to make an ohmic contact to diamond by the graded band gap concept whereby a smaller band semiconductor is intermixed epitaxially onto a larger band gap material. The project is a joint effort between Cornell University (J.W. Mayer) and the University of California, San Diego (Prof. S.S. Lau) under subcontract to Cornell. The project was successful in that low-resistance contacts to diamond were made by ion beam mixing.

All three aspects of the program were successful:

1. The Cornell group carried out or provided facilities for ion beam deposited at other laboratories.
2. The UCSD group formed ohmic contacts to diamond by depositing Si on Diamond and ion-mixing.
3. The Cornell group demonstrated compound formation between Ni and BP (boron phosphide) which was chosen as a prototype of a high melting point refractory semiconductor with a wide band gap for evaluation of ion beam mixing.

The ohmic contacts to natural type-IIb diamond were formed by depositing Si on the diamond and then ion-mixing the Si with diamond at 700°C with a dose of $2 \times 10^{15}$ Kr ions/cm². A Si/SiC/diamond structure was formed as evidenced by IR absorption measurements of SiC bands. Contact resistance measurements were made after annealing the ion-mixed sample at 1200°C for 1 hour in flowing forming gas. The specific values determined from
made after annealing the ion-mixed sample at 1200°C for 1 hour in flowing forming gas. The specific values determined from transmission line measurements were contact resistivity of $6 \times 10^{-4} \pm 3 \times 10^{-4} \Omega \cdot \text{cm}^2$. These values are a factor of two below that of standard, alloyed Au/Ta/diamond contacts.

I. INTRODUCTION

Diamond films are now being formed by a number of research groups. In anticipation of the growth of electronic quality diamond films, J.W. Mayer (Cornell) and S.S. Lau (UCSD) proposed to Max Yoder (ONR) that the Cornell/UCSD groups form ohmic contacts to diamond. The conventional contacts to diamond involve alloying a metal-film, for example Au-Ta, which leads to penetration of alloy spikes into the diamond while the alloyed contacts are adequate for bulk materials, non-uniform, penetrating alloy contacts are unacceptable for diamond-films. Mayer and Lau proposed ion-mixing to form a graded band-gap contact.

The concept of graded-band gaps for ohmic contacts has been used in GaAs structures. The barrier height of metal-Schottky barriers on n-type GaAs is about 0.8 eV and carrier concentrations rarely exceed $5 \times 10^{18} / \text{cm}^3$. Hence tunneling contacts will not be appropriate. However you can grade the band-gap from InAs (easy to contact) on the surface to InGaAs to GaAs. The lattice mismatch of InAs on GaAs is large and grading by way of In$_x$Ga$_{1-x}$As is required to prevent internal barriers.

Our proposition for diamond was to make a Si/SiC/Diamond structure. We can make ohmic contacts to silicon. If we could
tailor the structure from Si to $\text{Si}_x \text{C}_{1-x}$ to diamond, we should be able to form a low resistance contact. We chose to use ion-mixing. In this case a layer of Si is deposited on diamond and ion implantation at $700^\circ\text{C}$ (to prevent amorphization) is carried out to intermix the Si and diamond. The concept works.

II. RESULTS

1. The two key papers acknowledging ONR support are included as appendices:


2. Ohmic Contacts to Diamond.

   The diamond samples were colorless type IIb p-type diamond with a resistivity of 1200 ohm-cm. A 500 Si layer was deposited on the samples, implanted with $2 \times 10^{16}$ K ions/cm$^2$ at 240 keV and annealed at $1200^\circ\text{C}$ for 1 hour in forming gas. Th Kr implantation formed an n-type layer on the p-type substrate so that transmission line measurements could be made to evaluate the contacts. The specific contact resistivity is $6 \times 10^{-4}$ to $3 \times 10^{-4}$ $\Omega$-cm$^2$. The uniformity and contact resistivity are better than that of conventional Au/Ta diamond contacts.
Ohmic Contacts Formed by Ion Mixing in the Si-Diamond System

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Ohmic Contacts Formed by Ion Mixing in the Si-Diamond System

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Abstract—Graded-bandgap contacts to natural type-IIb diamond were formed by ion-beam mixing of Si(500 A)/diamond samples. Ion mixing carried out using 240-keV Kr⁺ ions at a dose of 2 x 10¹⁷/cm² at a temperature of 700°C. Si-C bonds were observed by IR absorption measurements, indicating the formation of a Si/SiC/diamond graded structure. Transmission line model (TLM) measurements show that well-behaved ohmic contacts can be formed on n-type channels by ion mixing with a subsequent thermal anneal. Samples without ion mixing showed higher specific contact resistivity than ion mixed samples (≈ 5 x 10⁻⁹ Ω-cm² versus ≈ 1 x 10⁻⁹ Ω-cm²). For comparison purposes, Au-Ta-based contacts were also prepared.

I. INTRODUCTION

Among the group IV semiconductors, diamond is attracting a great deal of interest because of its electrical, physical, and chemical properties. These properties make diamond a leading candidate for high-voltage optoelectronic switching [1], space optics devices [2], and high-temperature devices [3]. In order to fulfill its promise, however, it is necessary to develop an easily fabricated reliable ohmic contact.

Low-resistance ohmic contacts to diamond are difficult to form due to the very wide bandgap of diamond ($E_g \approx 5.5$ eV). A metal deposited on polished n-type diamond will not form a good ohmic contact because of the ~4-eV barrier height [4]. Some limited success has been achieved by mechanically damaging the diamond surface prior to metal deposition, a technique that produces a high surface recombination velocity and inhibits minority-carrier injection. Refractory metals such as tantalum, tungsten, or titanium have also been used on p-type diamond with some success [5], [6]. At high temperatures these metals will chemically react with diamond to form their respective carbides, allowing the alloy to wet the diamond and form a reasonable ohmic contact.

A different approach to forming ohmic contacts was used in this work. We applied the graded-bandgap concept utilized in III-V semiconductor technology to form ohmic contacts. This concept involves the deposition of a smaller bandgap semiconductor onto a larger bandgap material. A contact is more readily formed on the smaller bandgap material, and current transport across the junction is facilitated by the graded bandgap. This technique was first utilized in the Ge ($E_g = 0.66$ eV)/GaAs ($E_g = 1.42$ eV) system [7]. We propose the use of two smaller bandgap materials in series to form a graded bandgap to diamond. First, SiC with a bandgap of 2.9 eV (for α-SiC) is utilized to "reduce" the bandgap of diamond from 5.5 eV. Then Si is used to "reduce" the bandgap of SiC to 1.1 eV. Forming an ohmic contact to Si is relatively easy. The graded-bandgap scheme has the further advantage of allowing us to form ohmic contacts to n-type diamond (barrier height ~4 eV). Previous reports have all involved contacts to p-type diamond, with a barrier height of ~1.5 eV. In this work, we form the Si/SiC/diamond structure by first depositing Si on diamond. High-temperature ion mixing, plus subsequent thermal annealing, is then used to promote the formation of a uniform SiC layer at the Si-diamond interface. The resulting ohmic contacts are analyzed using a transmission line model (TLM) to determine the contact resistivity. A TLM measurement is the conventionally accepted technique for accurate determination of contact resistivity. The TLM is well documented in the semiconductor technology literature [8]. Briefly, the technique consists of a linear array of equally sized contact pads, with varying contact spacing. Current is passed between two pads, and the voltage drop is measured. The resulting resistance ($R$) is plotted as a function of contact spacing. Extrapolation of the data to zero contact spacing yields the resistance due to the contact itself. In order to correctly apply the TLM technique, one requirement is that current flow between contact pads be two dimensional, i.e., no vertical flow of current in the diamond. Since type-IIb diamond is p-doped, it is necessary to further dope the surface region in order to form a conducting channel to the best of our knowledge there have been only limited attempts to apply the TLM measurement to diamond, and none of these has utilized a conducting channel. Our n-type channel was formed by high-temperature implantation (concurrent with the formation of the SiC layer).

II. EXPERIMENTAL

Three sample types were prepared. All were natural colorless type-IIb p-type diamonds with a resistivity of 1200 Ω-cm. The sample size was $4 \times 4 \times 0.25$ mm. The

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Doping of diamonds has been investigated for a number of years with limited success. In-situ doping during growth of both n- and p-type epitaxial layers on synthetic diamond has recently been successful at introducing impurities in an activated state [10]. Ion implantation has also been investigated as a doping technique, primarily by workers in the Soviet Union [11]. Li⁺, B⁺, and P⁺ ions have been used with varying degrees of success: however, C⁺ and Xe⁺ ions may also be used for doping, suggesting that radiation damage may be responsible for the observed change in conductivity [10], [12]. Experiments have shown that the conductivity is dependent on the ion dose, dose rate, and the temperature during implantation [12]. For this study we are interested in ion mixing of Si on diamond, necessitating the use of a "heavy" ion. Kr⁺ ions were chosen as having the most suitable mass-ion range combination. Four-point probe measurements were used to determine sheet resistance after implantation. Additionally, hot probe measurements were carried out to determine carrier type. The results are summarized in Table I and Fig. 1. Room-temperature implantation resulted in p-type conductivity, possibly indicating the formation of a graphitic layer. Higher temperature during implantation gives n-type conductivity up to much higher doses. Subsequently post-implant annealing of these n-type layers at 1200°C resulted in a slight increase in sheet resistance, but the layer remained n-type. Because ion mixing is more efficient at high temperature, the remainder of this investigation was carried out with implantations at 700°C.

B. SiC Formation

Infrared absorption analysis for wavelengths between 2 and 25 μm was used to monitor the formation of SiC and subsequent structural changes after ion mixing and thermal annealing. SiC has an intense absorption band at 797 cm⁻¹ due to photon interaction with transverse optical phonons (Si-C bonds) [13]. Fig. 2 shows the absorption spectra obtained in the as-deposited sample (dotted line), after 1-h anneal in flowing forming gas at 1200°C (dashed line), and after ion mixing with 2.1 × 10¹⁶ Kr⁺/cm² at an energy of 240 keV and substrate temperature of 700°C plus a 1200°C anneal in flowing forming gas (solid line). After annealing, an absorption band near 797 cm⁻¹ appears. The peak height increases after Kr⁺ ion mixing and a high-temperature anneal. This is taken as an indication that ion mixing leads to the enhancement of the SiC structure. Diamond is relatively inert even at temperatures as high as 1200°C; the ion mixing may thus help to break carbon-carbon bonds. (Diamond has a vacancy formation energy of 3.7 eV versus 2.1 eV for Si and 1.9 eV for Ge.)

### Table I

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Dose (cm⁻²)</th>
<th>Carrier Type for Different Implant Conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1×10¹⁵</td>
<td>5×10¹⁵</td>
</tr>
<tr>
<td>R.T.</td>
<td>p</td>
<td>p</td>
</tr>
<tr>
<td>375</td>
<td>p</td>
<td>n</td>
</tr>
<tr>
<td>450</td>
<td>n</td>
<td>n</td>
</tr>
<tr>
<td>600</td>
<td>n</td>
<td>n</td>
</tr>
<tr>
<td>700</td>
<td>n</td>
<td>n</td>
</tr>
</tbody>
</table>
measurements. To the best of our knowledge this is the first time TLM measurements have been carried out using a covalent semiconductor such as diamond for two reasons. First, diamond has a barrier height essentially independent of the metal work function [4]. Second, it is difficult to produce a heavily doped surface layer in diamond structure. In this work, the graded-bandgap contacts to diamond involve the Si/SiC structure.

C. Formation of Ohmic Contacts

A metal-semiconductor ohmic contact is formed by lowering the effective barrier height or by doping the semiconductor very heavily in the vicinity of the contact. In the high doping case, although the barrier height remains the same, the barrier thickness is greatly reduced, and charge carriers in the semiconductor can easily tunnel through the barrier.

It is difficult to form ohmic contacts on wide-bandgap materials such as diamond for two reasons. First, diamond has a barrier height essentially independent of the metal work function [4]. Second, it is difficult to produce a heavily doped surface layer in diamond structure. In this work, the graded-bandgap contacts to diamond involve the Si/SiC/structure.

Specific contact resistivity was determined using TLM measurements. To the best of our knowledge this is the first time TLM measurements have been carried out using an n-channel region in diamond. Since the bulk diamond is p-type, this avoids bulk conduction and gives an accurate TLM measurement. Contact pads (150 μm square) with varying intercontact spacing (2 to 32 μm) were formed on diamond (500 Å, n-type, 2 × 10^{16}/cm² Kr⁺ implanted) substrates. Test structures were isolated by CF₄ plasma etching at a power of 100 W to form mesa structures. The injected current for the TLM measurement is 1 mA. The best measured contact resistances of the Si/diamond contacts with and without ion mixing as a function of contact spacing are shown in Fig. 3(a) and (b), respectively. The linear correlation coefficient is 0.98 in the case of the ion mixed contacts, and 0.92 in the case of the non-mixed contacts. The actual specific contact resistivities are 6 × 10⁻⁴ ± 3 × 10⁻⁴ Ω-cm² and 4.78 × 10⁻³ ± 2.15 × 10⁻³ Ω-cm² for the ion mixed and non-mixed contacts, respectively. It is clear, then, that the ion mixed sample has a lower contact resistivity and better linear correlation. This may be taken as an indication of the usefulness of ion mixing in the formation of more Si-C bonds, a necessity for the formation of a well behaved graded-bandgap ohmic contact. A summary of experimental results is presented in Table II. The average specific contact resistivity of Au/Ta/diamond contacts is comparable with that of ion mixed Si/diamond contacts. However, the standard deviation in contact resistivity for the Si/diamond contacts is half that of Au/Ta/diamond contacts. Au/Ta/diamond alloyed contacts may have metallic penetration into the diamond, as evidenced by the substantial roughness observed on the contact surface. This could lead to contact spiking or difficulties in producing the planar device structures favored in integrated circuit fabrication.
### Table II

<table>
<thead>
<tr>
<th>Sample Type</th>
<th>Contact System</th>
<th>Thickness (Å)</th>
<th>Treatment</th>
<th>Specific Contact Resistance (Ω-cm)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>S/diamond</td>
<td>500</td>
<td>ion mixed and annealed at 1000°C, 1 hour</td>
<td>$1 \times 10^{-7}$</td>
<td>uniform contact</td>
</tr>
<tr>
<td>2</td>
<td>S/diamond</td>
<td>500</td>
<td>annealed at 1000°C, 1 hour</td>
<td>$5 \times 10^{-5}$</td>
<td>uniform contact</td>
</tr>
<tr>
<td>3</td>
<td>Au/Ta/diamond</td>
<td>940/100</td>
<td>annealed at 1000°C, 1 hour</td>
<td>$1 \times 10^{-5}$</td>
<td>non-uniform alloyed contact</td>
</tr>
</tbody>
</table>

(1) Average value for each sample type.
(2) Although type 1 and type 3 contacts have a similar mean value, the distribution of measured values is much more closely spaced for the S/diamond (type 1) contacts.

### IV. Summary

Kr$^+$ implantation into diamond with different doses and sample temperatures was investigated. It was found that the surface layer of the diamond could be made n-type with a five to six orders of magnitude decrease in sheet resistance. IR absorption measurements show that annealing of Si on diamond at 1200°C leads to the formation of SiC. High-temperature ion mixing prior to annealing enhances the formation of SiC. TLM measurements showed that well behaved ohmic contacts on n-type diamond can be formed with a graded-bandgap Si/SiC/diamond structure formed by ion mixing. The uniformity of such contacts is much better than that of conventional Au/Ta/diamond contacts. Au/Ta/diamond alloyed contacts may have metallic penetration into the diamond.

### Acknowledgment

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### References


Fang Fang was born in Shanghai, People’s Republic of China. She was a graduate student from 1982 to 1985 at the Shanghai Institute of Metallurgy, Academia Sinica, majoring in semiconductor physics and devices. Since graduation, she has been on the research staff of the Ion Beam Laboratory, Shanghai Institute of Metallurgy.

Charles A. Hewett was born in Columbia, MO. in 1940. He received the B.S. degree in materials science and engineering from Cornell University in 1962 and the M.S. degree in electrical engineering from the University of California at San Diego in 1966. He is currently working toward the Ph.D. degree in electrical engineering at the University of California at San Diego. His doctoral research is in the area of ion-solid interactions and their applications.

Mr. Hewett is a student member of the Materials Research Society and the American Physical Society.

Mark Fernandes received the B.Tech. degree in chemical engineering from I.I.T., Kharagpur, India, in 1980 and the Ph.D. degree in materials science and engineering from McMaster University, Canada, in 1986. Since then, he has been a Research Engineer in the Department of Electrical and Computer Engineering at the University of California, San Diego. His research interests include ion-beam effects on materials and materials analysis.

Dr. Fernandes is a member of the Materials Research Society.

S. S. Lau has been a Professor of Electrical and Computer Engineering at the University of California, San Diego, since 1980. His research interests include ion-beam processing of electronic and optoelectronic materials and thin films.
Thermal and ion beam induced reactions in Ni thin films on BP(100)

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Thermal and ion beam induced reactions in Ni thin films on BP(100) have been investigated. For thermally annealed samples the reaction of the Ni layer on BP started at temperatures between 350 and 400 °C and the formation of the crystalline phase corresponding to a composition of Ni₂BP was observed at 450 °C. It was observed that oxygen in the Ni layer could retard the progress of the reaction. The full reaction of the Ni layer with BP induced by energetic heavy ion bombardments (600 keV Xe) was observed at 230 °C. The crystalline phase has the same composition and x-ray diffraction pattern for both thermal and ion-induced reactions.

BP (boron phosphide) is one of the attractive electronic materials as a refractory semiconductor with a wide band gap of 2.1 eV. This compound is also characterized by several outstanding properties such as a high melting point above 3000 °C, a high decomposition temperature of about 1130 °C under 1 atm, extreme hardness, and high oxidation resistance at high temperatures. The fabrication of the Schottky barrier diodes using BP wafers has been reported. Recently, attention has been focused on the ion beam induced epitaxial crystallization (IBIEC) in BP.

The investigation of the mechanism of the reaction of metals with BP attracts interest from the technological view of the device applications of BP and also physical and chemical views of the reaction of metal thin films on the refractory compound semiconductor. Ion-induced reactions in thin-film systems, on the other hand, have been used to form many phases from equilibrium to amorphous phase and can provide a knowledge of production of innovative materials. From these points of view, this research work was intended to make an investigation on the fundamental mechanism of reactions of the metal BP system. In this letter we present the first observation of both thermal and ion beam induced reactions of Ni thin films on BP(100). The striking result is that the reaction temperature was around one-fifth of the melting point (K).

Wafers with BP film thicknesses of around 1 μm were prepared by the thermal decomposition of B₂H₆ and PH₃ in a hydrogen atmosphere on Si(100) substrates at temperatures between 950 and 1150 °C. All wafers were confirmed to be grown epitaxially by reflectance high-energy electron diffraction (RHEED) and channeling experiments using MeV He ions. Ni thin layers with a thickness between 50 and 150 nm were deposited onto BP(100) wafers at room temperature (RT) by evaporation with the resistive method and with the electron beam (EB) method. The pressure during evaporation was 4 × 10⁻⁵ Torr for the resistive method and was less than 4 × 10⁻⁷ Torr for the EB method. For the thermally induced reaction experiments, samples were annealed in the vacuum (1.5 × 10⁻¹ Torr) at a given temperature for 30 min. In order to investigate the ion beam induced reactions, samples with the thickness of 50 nm of the Ni layer were bombarded with 600 keV Xe²⁺ ions. The projected range and the straggling of 600 keV Xe ions in Ni are 71.4 and 12.7 nm, respectively.

Analyses of the thickness, compound composition, and homogeneity of the reacted layers of Ni with BP were performed with the Rutherford backscattering (RBS) measurements using a 2 MeV He⁺ ion beam. Observations with x-ray diffraction were also performed in order to investigate the phase of the reacted layer. Auger electron spectrometry (AES) was employed for the depth analysis of the composition in the layers.

For the samples deposited in a poor vacuum with the resistive method (sample I), the reaction of the Ni layer started at 350 °C and proceeded up to the formation of the reacted layer with 100 nm thickness at 400 °C. Although the Ni layer was only partly consumed, the reaction proceeded no further up to 500 °C. Figure 1 shows the RBS spectrum of this partly reacted Ni layer on BP after annealing at 450 °C.

![FIG. 1 RBS spectra of partly reacted Ni layer on BP(100) [sample I (clean)] and fully reacted layer [sample II (clean)] with 2 MeV He ions after the thermal annealing at 450 °C.](https://example.com/rbs-spectra.png)
method (sample II), the reaction started at 400 °C. Thermal annealing at 450 °C has induced the fully reacted layer of Ni with BP. The RBS spectrum of this sample is also shown in Fig. 1 after the annealing. The compound composition of the reacted layer determined by the RBS measurements has the atomic ratio for Ni:B:P to be 4:1:1 both in samples I and II.

The depth profile of the composition of the partly reacted layer (sample I) observed with the AES measurements is shown in Fig. 2. The composition of the reacted layer derived from the AES analysis has coincided with that derived from the RBS analysis. One can observe oxygen atom accumulation at the interface between the Ni layer and the reacted layer. This suggests that oxygen atoms have an important role in the retardation of the progress in the reaction in the Ni layer on BP.

Ion bombardment experiments were performed for the samples with a thin clean Ni layer deposited with the EB method (sample III). Figure 3 shows the RBS spectra of the sample before and after the ion bombardment with 600 keV Xe²⁺ ions. One can see the fully reacted layer of Ni on BP after the ion bombardment at 230°C with the fluence of 5 × 10¹⁵ ions/cm² together with the spectrum of the sample before bombardment. The reacted layer has the same composition ratio as that of the thermally reacted layer from the analysis of the RBS measurements.

Thin-film x-ray diffraction experiments on the fully reacted layer (sample II) have revealed that this layer has a crystalline phase. The ion-induced layer (sample III) has also shown the same diffraction pattern. As an example of the reaction of metal films on a refractory compound semiconductor, the reaction of Ni on SiC was reported. In this case, the first phase is thought to be a binary phase (Ni₃Si) and no reaction of Ni with C atoms was observed. In contrast to the case of SiC, a ternary phase (Ni₃BP) or mixed binary phase (NiB + Ni₃P) is thought to be the candidate for the reacted phase of Ni thin film on BP from the x-ray diffraction experiments. The dominant moving species in the reaction of Ni on BP is thought to be Ni from the results of the oxygen accumulation between the reacted layer and the nonreacted layer.

In conclusion, we have observed the reaction of Ni thin films on BP(100) at temperatures around 400 °C for the thermal reaction and around 200 °C for the ion beam induced reaction. Metal-rich ternary phase or mixed binary phase is thought to be the first crystalline phase formed both in the thermal and ion beam induced reactions. Although further studies on kinetics of the compound growth are necessary for the detailed investigation of the reaction mechanism, it is noted that the reaction of Ni thin films on refractory BP was found to occur at temperatures as low as one-fifth of the melting point (K).

The authors would like to express their sincere gratitude to Randy Phillips and Lynn Rathbun for AES measurements and to Shi-Qing Wang for x-ray diffraction measurements at Cornell University. One of the authors (N. K.) acknowledges the Agency of Science and Technology of Japan for the financial support of his stay at Cornell. The Cornell work was supported in part by the Office of Naval Research (M. Yoder).

![Figure 2](image_url)

**FIG. 2.** Depth profile of the composition observed in the partly reacted Ni layer on BP(100) (sample I) after the thermal annealing at 450°C by the AES measurements.

![Figure 3](image_url)

**FIG. 3.** RBS spectra of Ni on BP(100) (sample III) before and after the 600 keV Xe²⁺ bombardments with the fluence of 5 × 10¹⁵ ions/cm² at 230°C.