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Materials Processing of Diamond: Etching, Doping by Ion Implantation and Contact Formation

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13. ABSTRACT *(Maximum 200 words)*

A summary of work achieved under this grant is given, including ion implantation doping of natural diamond, annealing of implantation damage, characterization methods, contact formation using a sputtering method, and plasma etching.

14. SUBJECT TERMS

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FINAL REPORT

Contract N00014-87-K-0243, Materials Processing of Diamond: M.L. Swanson, P.I.

SUMMARY OF WORK

1. DOPING OF NATURAL DIAMOND

Ion implantation techniques were developed for doping natural diamond, with the eventual goal of doping CVD diamond layers. The major effort was devoted to p-type doping with boron. The use of dual implantation at low temperature (77K) with both C and B was found to enhance the doping efficiency, for two reasons: (i) the C implantation provided vacancies which could trap the implanted B atoms on substitutional sites; (ii) the recombination efficiency of B interstitials and vacancies during annealing was enhanced by the low temperature of the implantations.
The results, including optical and electrical measurements, were summarized in the annual reports 1987-88. 1988-89, 1989-90 and 1990-91, and in refs. 1, 4, 5, 6, and 12.

Attempts to dope diamond n-type via Na, Li or F implantations formed the final part of the doping program, and were intended to be preliminary studies leading to a new diamond program. However, this program was not funded for renewal. Promising results were obtained in the resistivity data, showing a linear R vs 1/T behavior with activation energy near 0.4eV for Na, which is close to the predicted donor level 0.3eV. The problem of residual implantation damage still existed, however. This problem could possibly be overcome by diffusing the dopants past the damaged layer (e.g. by using ionizing radiation to enhance the diffusivity), followed by etching to remove the damaged layer. These results are summarized in the annual report 1990-91.

2. ANNEALING OF IMPLANTATION DAMAGE

For effective activation of dopants, the damage caused by ion implantation must be removed. Optical, electrical and RBS/channeling methods were used to measure the recovery of implantation damage. Complete removal of damage could not be achieved by rapid thermal or furnace annealing up to 1100°C. However, for low ion doses, most of the damage was successfully removed by annealing. For high doses the diamond was graphitized; for deep implants (approx. 1 μm) the dose required for graphitization was increased because of the constraint of the overlying diamond.

For intermediate ion doses, from 1.5×10^{15} - 10^{16} C ions/cm^2, no recovery of the randomization of the lattice was observed by channeling, and the implanted diamond layer became a golden -green color. This "green phase" may represent a stable structure of vacancy clusters in diamond, similar to ordered void structures in irradiated metals.

This work is summarized in the annual reports 1989-90, 1990-91, and in refs. 9, 11 and 14.

3. CHARACTERIZATION

Several characterization methods were used: Raman scattering, electrical resistance, optical absorption RBS/channeling, elastic recoil detection, and coincidence ion beam methods. The results of these methods of analysis are summarized in the annual reports 1987-88, 1988-89, 1989-90, 1990-91, and in refs. 2, 3, 8 and 10.

4. CONTACTS

A new method of forming strongly adhering ohmic contacts on natural diamond was developed and patented. It consisted of sputtering the diamond surface with Ar, then sputter depositing a layer of Au. Since this process was not done in UHV, it probably involved the radiation-enhanced diffusion of Au atoms into the upper few atomic layers of the diamond because of the sputter-induced vacancies. Thes results are given in the annual reports 1987-88, 1988-89

5. ETCHING

An effective method of removing thin layers of diamond is plasma etching. We found that etching in an oxygen or hydrogen plasma removed natural diamond at rates up to 70 nm/min. These results are summarized in the annual reports 1987-88, 1988-89 and ref. 7.

U.S.A. PATENT

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METHODS OF CREATING SEMICONDUCTING DIAMOND

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Diamond films grown by chemical vapor deposition can be used for wear-resistant coatings, heat sinks and optical windows. Future exploitation of diamond films for blue LED's, switching devices and high temperature transistors requires the efficient production of films that are both semiconducting and heteroepitaxial. Doping to create semiconducting diamond is complicated by the low solubility and diffusivity of most elements. Some success has been achieved in doping diamond with B during both CVD and synthetic diamond growth, and by dual implantations with dopants plus C in natural diamond. Implantations of Na and Li for n-type doping have been attempted. The recovery of damage in ion-implanted diamond exhibits a sharp transition at a defect density of about 7%; below this level, almost complete recovery occurs, but above this level a stable damage structure remains. The heteroepitaxial growth of diamond by CVD methods on suitable substrates, and by ion implantation of C into Cu has been reported; this is a very promising development for electronic applications.

1. INTRODUCTION

The economical growth of diamond films by chemical vapor deposition (CVD) has opened up many areas of commercial exploitation, including heat sinks, optical windows, and coatings for wear and corrosion resistance. In addition, when heteroepitaxial semiconducting diamond becomes available, many optoelectronics and microelectronics applications will become feasible, such as blue light-emitting diodes (LED's), high power switching devices and high temperature transistors (Davis et al.1988, Collins 1990).

There are two basic problems in the production of device-quality semiconducting diamond films. One problem is to dope the diamond with electrically active n- and p-type dopants; this is complicated by the low solubility of most elements in diamond, and by the high melting point of diamond (estimated as 4000°C), which makes diffusion of most elements very slow below the temperature of spontaneous graphitization, about 1530°C. One method of overcoming this problem is to implant the desired dopant. In this paper, we shall review the methods of doping diamond, with emphasis on ion implantation methods. The second problem is the economical production of single crystalline diamond. We shall review recent progress in the growth of heteroepitaxial diamond films by CVD and ion beam methods.

2. DOPING METHODS

2.1 Natural diamond

The major dopants in natural diamond are B and N, although large quantities of O and H are also often present (Seal 1989). Type I diamond contains appreciable amounts of N which can occur in substitutional sites, as small clusters or as platelets.

Unfortunately, substitutional N gives a rather deep donor level, about 1.7 eV below the conduction band. Type II diamond is relatively N-free; type IIa is the purest form of natural diamond, although it may still contain variable and considerable amounts of impurities such as H and O (up 1 at%H and 0.2 at%O). Type IIb diamond contains B, up to levels of at least $10^{19}/cm^3$, giving an acceptor level at 0.37 eV above the valence band. This gives suitable semiconducting behavior. Unfortunately, no suitable shallow level n-type dopants are known. Because of the extremely close packing of atoms in diamond, 1.76 x $10^{23}/cm^3$, as compared for example with Si (5 x $10^{22}/cm^3$), the solubility of most elements in diamond is very low.

2.2 High pressure diamond

Dopants can be introduced directly during the growth of synthetic diamond at high pressure and temperature (Spitsyn et al. 1991). Boron concentrations from 10^{17} to $10^{21}/cm^3$ can be introduced, and titanium is also often added to compensate the inevitable nitrogen, whose content can be varied from 10^{16} to $10^{18}/cm^3$. For B concentrations up to $10^{19}/cm^3$, the activation energy from resistance-temperature curves is 0.37 eV, in agreement with data for CVD diamond and natural diamond. For higher B concentrations, the energy decreases gradually to about 0.1 eV at $10^{21}B/cm^3$, just as for CVD diamond. The electrical resistivity varies from 10 to 10^6 ohm cm at 293K, depending on the B and N concentrations.

2.3 Doping of CVD diamond

Doping of CVD diamond can be accomplished by introducing suitable carrier gases during the deposition. The most success has been reported for B doping. As the ratio of B to C in the reactant gas was decreased for homoepitaxial growth on (100) and (110) diamond surfaces, using microwave plasma CVD, the activation energy, as determined by a resistance versus 1/T plot, increased from about 0.1eV (attributed to a hopping mechanism at high dopant levels) to about 0.4 eV, which is close to the value 0.37 eV attributed to substitutional B (Fujimori et al. 1990). These results are shown in Fig. 1. Similar results were obtained using a hot filament method (Geiss 1990), although in that case the activation energy of lightly doped films was 0.32-0.39 eV, and heavily doped films showed almost no change in resistivity with temperature.

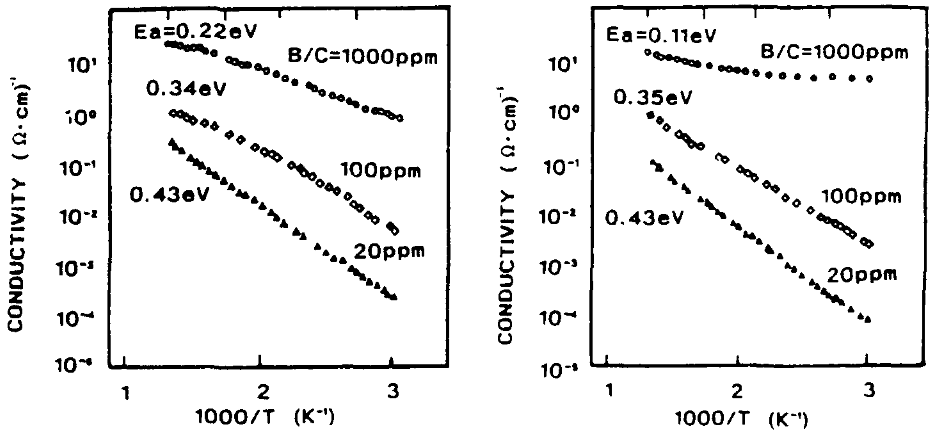

Fig. 1. Conductivity of B-doped diamond films deposited on (100) and (110) diamond (left and right panels, respectively) (from Fujimori et al. 1990).

Attempts to dope CVD diamond with N and P have also been reported, with considerably less success. We note that, according to calculations of Bernholc et al. (1991), B and N have negative formation energies in diamond, while P has a positive formation energy.

2.4 Doping by implantation

Numerous attempts to dope diamond by ion implantation have been made, dating from the early Russian work of Vavilov (1978) and co-workers. The basic difficulty with implantation doping is to anneal out the damage without graphitizing the crystal. Spontaneous graphitization during implantation also is a problem for high implantation doses. If we compare the experience of doping Si by implantation, we see that implanted amorphous Si regrows epitaxially at a temperature of about 550°C or approximately 1/2 of the melting temperature. However, the residual damage in the form of dislocation loops anneals out only at about 1100°C, or 4/5 of the melting point; this corresponds to the temperature required for appreciable self-diffusion. Since diamond graphitizes at less than 1/2 of the melting point, it may be difficult to anneal out residual ion damage in diamond.

3. IMPLANTATION METHODS

3.1 Basic concepts

Doping by implantation has the important advantages that the method is very clean, it gives precise amounts of dopant and the depth of doping can be controlled by varying the implantation energy. A further advantage which has been utilized little, but which may be especially important for diamond, is that the equilibrium solubility can be greatly exceeded by implantation. The major disadvantage is that the radiation damage produced by implantation must be removed by annealing. In many of the early implantation results, the electrical activation attributed to implanted dopants may have been due to residual radiation damage in the form of vacancy clusters.

3.2 Dual implantation

It was suggested by Prins (1988) that some of the problems of dopant activation and damage annealing in diamond could be overcome by dual implantations with C plus the desired dopant. The idea was to implant with C to create vacancies at the same depth as the distribution of the subsequently implanted dopant atoms. The implantation was to be done at 77K, where both self-interstitials and vacancies are immobile. Thus, during annealing, recombination of the vacancies with the implanted dopant atoms would enhance the substitutionality of the dopant. Some success has been reported with this method in the case of B, N and P doping (Prins 1988, Sandhu et al. 1989, 1990a). Recently, Prins (1991) has found that suitable annealing procedures could give equally good results in the case of single B implantations.

3.3 High versus low temperature implantations.

For low temperature implantations (293K or lower), many annealing results have indicated that almost complete recovery of the implantation damage, as measured by RBS/channeling, occurs for low doses, partial recovery occurs for intermediate doses, and graphitization occurs for high doses (Braunstein et al. 1980). However, recent results have indicated that graphitization can be suppressed by using very deep implants, presumably because the expansion of the lattice necessary to create graphite is prevented by the constraint of the overlying relatively undamaged diamond (Sandhu et al. 1990b).

Because of the limited implantation dose possible at low temperatures without graphitization, and because of residual damage remaining after annealing of low dose samples, it was suggested that high temperature implantations, where defects anneal out during the implantations, might be an effective way to reduce the implantation damage and prevent graphitization. Although at high temperatures (1100°C) the implantation dose can indeed be greatly increased without graphitization, considerable residual damage remains (Lee et al. 1978, Braunstein and Kalish 1983, Nelson et al. 1983).

3.4 n-type doping

It is essential to achieve n-type doping for many microelectronics applications. To date, effective n-doping has not been achieved in any type of diamond. Theoretical considerations have indicated that Li and Na will occupy interstitial lattice sites in diamond, and thus act as donors. Of these elements, Na is the preferred candidate for a suitable donor (Bernholc et al. 1991); its predicted energy level is ≈0.3 eV below the conduction band, and its activation energy for diffusion is estimated to be 1.4 eV. Fluorine has been shown by perturbed angular correlation measurements to occupy, at least partially, interstitial lattice sites (Connell et al. 1988).

As mentioned in Section 3.2, partial success in obtaining n-type diamond has been achieved using dual implantations of N or P together with C. However, the problem of residual damage remains a difficult one. We have implanted natural diamond with Na and Li, and have measured the resistivity, optical absorption and RBS/channeling yields before and after annealing. Our preliminary data for Na-implanted diamond are shown in Fig. 2, indicating a large increase in resistance as damage is removed by annealing. In such samples, if a suitably high concentration of electrically activated dopant could be injected by implantation, and then diffused beyond the damaged layer, the damaged layer could be completely removed by plasma etching to leave perfect n-type diamond.

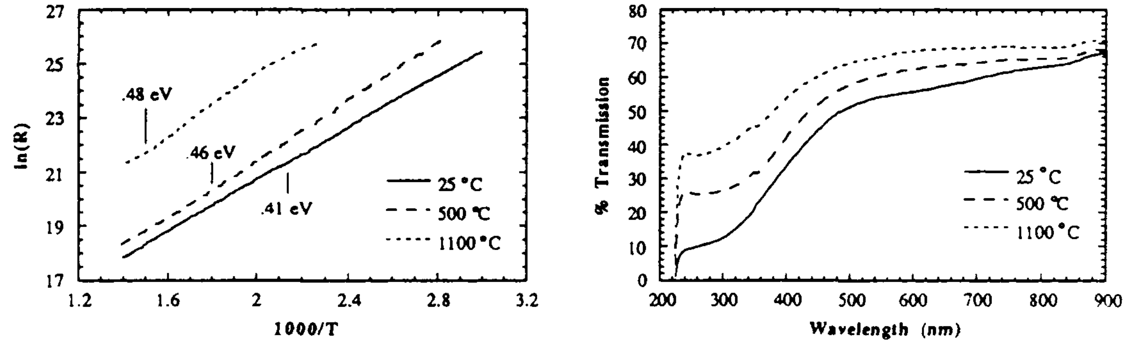

Fig. 2. Resistance versus temperature and optical transmission data for type IIa diamond after implantation at 77K with 5.2×10^{15} Na ions per cm^2, warming to 25°C, and subsequent annealing at 500°C and 1100°C.

4. ANNEALING OF DAMAGE AND REGROWTH

The main methods of damage characterization have been optical absorption, electron paramagnetic resonance (EPR) and Rutherford backscattering/ channeling (RBS). Optical absorption data indicate that several vacancy-associated centers (GR centers) are created by irradiation, and are annealed gradually over a very broad temperature range from 500-1100°C (Clark and Mitchell 1971, Walker 1979). Optical absorption data show that considerable damage in the form of near-edge absorption remains after annealing up to 1100°C (see Fig.2). EPR data also indicate a variety of defects produced by irradiation (Lomer and Wild 1973).

RBS/channeling results show that most of the gross implantation damage anneals out at 900°C or less, for low implantation doses. An example of these results for carbon implantations of natural diamond to low doses is shown in Fig. 3 (Hunn et al. 1991). Of particular interest is the observation that complete recovery of the implantation damage occurred for doses below a critical level, but that no recovery of damage occurred above that level. The critical dose is seen from Fig. 3 to be about 1.2 x 10^{15}/cm^3 for 50 keV C ions. For this dose, most of the damaged layer recovered completely, but at the peak of the damage distribution, no recovery occurred. In contrast, the deeper implant at 100 keV, which corresponds to a lower energy density deposited into nuclear collisions, recovered completely. The critical defect density corresponding to this transition was 7% Frenkel defects, as determined by TRIM calculations. In the damaged layers that did not show recovery of channeling, after annealing the color of the diamond changed from brown to a striking golden green color, and optical absorption data showed strong, broad absorption in the visible range. This "green phase" was stable to at least 1100°C.

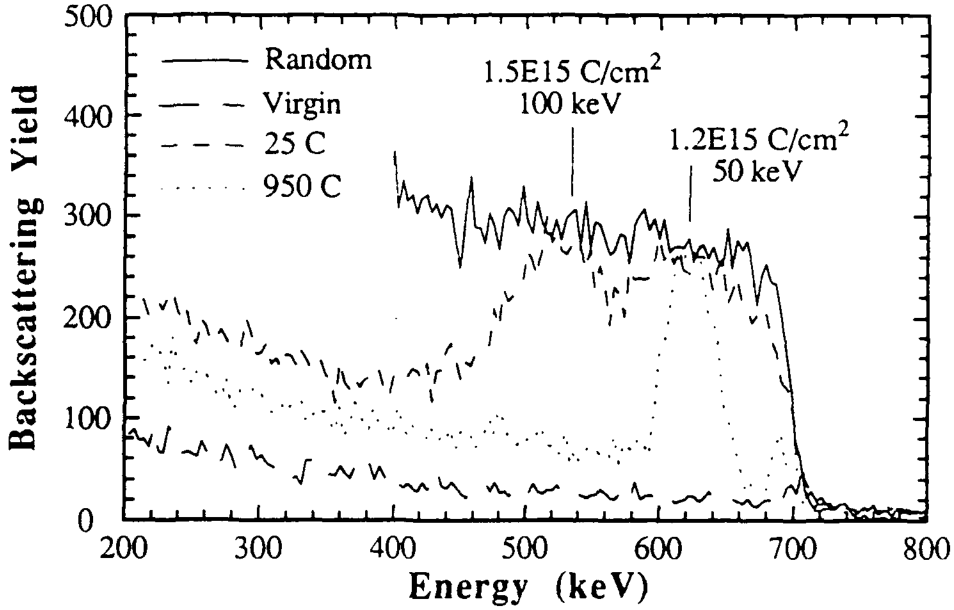

Fig.3. RBS/channeling of 2 MeV He$^+$ along <100> normal of type IIa diamond, implanted with C and annealed at 950°C for 30 min (Hunn et al. 1991).

5. HETEROEPITAXIAL GROWTH OF DIAMOND FILMS

5.1 Chemical vapor deposition.

Polycrystalline CVD diamond films of very good quality, as shown by the sharp 1332 cm^{-1} Raman scattering signal, have been grown using a variety of gas mixtures, mostly on Si substrates (Spear 1989, Davis 1988). Most films were grown using a methane-H mixture, but recently it was found that the addition of oxygen-containing gases is beneficial for growth; films have also been grown using halides instead of H compounds (Rudder et al. 1989). The grain size of these films is small, of the order of micrometers. For electronic applications it is considered necessary to grow single crystalline diamond films, although in principle devices can be easily constructed on micron-sized crystals. Efforts to grow an ordered array of diamond crystallites ("SENTAXY") have also been successful (Kawarada 1990).

Considerable progress has been made in the growth of single crystalline diamond films by CVD on substrates such as c-BN, Cu and Ni (Davis et al., 1988, Itoh et al. 1991, Kawarada et al. 1990, Yarbrough 1991), which have a good lattice match to diamond. However, large area single crystalline regions have not yet been achieved.

5.2 Ion beam methods

Novel ion beam growth methods such as ion beam deposition (Lifshitz 1989) and ion implantation (Prins and Gaigher 1991, Narayan et al. 1991) have been utilized to achieve heteroepitaxial diamond. Lifshitz (1989) has attempted to grow diamond by the low energy deposition of C at about 50 eV energy. At this energy it was hoped that sufficient energy would be imparted to the C layer to displace C atoms in graphite, but not in diamond, resulting in diamond growth. The results of this method are still somewhat in doubt.

An additional novel ion beam method for growth of diamond on Cu crystals has been reported (Prins and Gaigher 1991; Narayan et al. 1991). This method consists basically of implanting C into a material that has a very low solubility for C but a good lattice match to diamond. If the implantation is done at high temperatures where the implanted C atoms are mobile, the Cu lattice will reject the C atoms, resulting in diffusion of the C to the surface and formation of an epitaxial diamond film. Success in this method has been reported by Prins and Gaigher (1991), who implanted C into Cu at elevated temperatures (up to 900^{o}C), and a patent has been issued. However, up to now single crystal diamond has not been formed on single crystal samples of Cu by this method. It appears that the preparation of the Cu surface is very important in such experiments, since the presence of a very small amount of graphitic carbon on the Cu surface will cause nucleation of graphite rather than diamond. A similar method was reported by Narayan et al. (1991), whereby the C ions were implanted into single crystalline Cu at room temperature, and the Cu was subsequently excimer laser-annealed to achieve monocrystalline films of diamond of about 50 nm thickness. Difficulties in reproducing these latter results are probably related to the critical laser pulse energy and duration, and to the fact that the diamond is buried near the Cu surface, so that delicate etching is required to reveal it. Similar experiments have been performed using Ni substrates, but in the case of Ni, carbide formation occurs, which is clearly detrimental.

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