Los Alamos National Laboratory is operated by the University of California for the United States Department of Energy under contract W-7405 ENG-36

RECEIVED
JAN 16 1996
OSTI

Correlation of Electrical Properties with Defects in a Homoepitaxial Chemical-Vapor-Deposited Diamond

Author(s): Sung S. Han, George Rodriguez, Antoinette Taylor, MST-11
M. A. Plano, M. Dale Moyer, M. A. Moreno, Crystallume, Inc.
L. S. Pan, Sandia National Laboratories, Livermore
D. B. Black, H. Burdette, NIST
J. W. Ager III, A. Chen, Lawrence Berkeley National Laboratory

Submitted to: Materials Research Society Fall Meeting
Boston, MA
November 27 - December 1, 1995

By acceptance of this article, the publisher recognizes that the U.S. government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or to allow others to do so, for U.S. Government purposes.

The Los Alamos National Laboratory requests that the publisher identify this article as work performed under the auspices of the U.S. Department of Energy.

Los Alamos

Los Alamos National Laboratory
Los Alamos, New Mexico 87545

CORRELATION OF ELECTRICAL PROPERTIES WITH DEFECTS IN A HOMOEPITAXIAL CHEMICAL-VAPOR-DEPOSITED DIAMOND

S. HAN¹, G. RODRIGUEZ¹, A. TAYLOR¹, M.A. PLANO^{2*}, M.D. MOYER², M.A. MORENO², L.S. PAN³, D.B. BLACK⁴, H. BURDETTE⁴, J.W. AGER III⁵, AND A. CHEN⁵

¹Los Alamos National Laboratory, D429, Los Alamos, NM 87545, U.S.A., han@lanl.gov

²Crystallume, Inc., 3506 Bassett Street, Santa Clara, CA 95054, U.S.A., MAPlano@aol.com

³Sandia National Laboratories, MS9162, 7011 East Avenue, CA 94551, U.S.A.,

LSPan@sandia.gov

⁴NIST, 223/A163, Gaithersburg, MD 20899, U.S.A., Black@enh.nist.gov

⁵Lawrence Berkeley National Laboratory, University of California, Berkeley, CA 94720,

U.S.A., ager@lbl.gov

ABSTRACT

A high-quality, low-stress 200 μm epitaxial diamond film has been grown on a 400 μm thick high-temperature-high-pressure IIa diamond. X-ray diffraction images of the film indicate that a large region of the film is fairly defect free and individual dislocations have been imaged in this region. Depth-resolved Raman results indicate that the region of the film with a low density of defects also has lower stress than in the higher defect density region. Transient photoconductivity measurements were performed on the high and low line defect density regions of the homoepitaxial diamond film to determine the effects of the stress and defect density on the combined electron-hole mobility and carrier lifetime. The correlation between the electrical properties and the x-ray diffraction imaging suggests that line defects may not be the limiting factor in the carrier transport at the present film quality.

INTRODUCTION

Currently, the heteroepitaxy of single-crystal chemical-vapor-deposited (CVD) diamond is not possible. The only method of obtaining a single-crystal CVD diamond is to grow it homoepitaxially on a single-crystal diamond substrate. Because grain-boundary in polycrystalline CVD diamond introduces another unknown in evaluating electronic devices, CVD diamond device physics should be investigated using single-crystal material. Therefore, the knowledge of how the single-crystal substrate affects the quality of the CVD epilayer needs to be obtained.

From previous work,¹ the plane and line defects such as stacking faults and dislocations have been found to propagate into the epilayer, especially dislocations. Such defects usually have detrimental effects on the carrier transport properties by acting as additional scattering and trapping centers which leads to degradation in electronic device performance.

In order to study the structural quality of single-crystal CVD diamond, an epitaxial layer was grown on an oriented, type IIa, single-crystal, high-pressure-high-temperature (HPHT) substrate with the crystal dimensions of 4x4x0.4 mm³. The growth of the epilayer was accomplished using a microwave-assisted CVD technique with the substrate temperature of 800 °C and the growth rate of about 1 $\mu\text{m/hr}$. The total thickness of the epilayer was 200 μm .

After the growth, x-ray diffraction imaging and depth-resolved, micro-Raman spectroscopy were performed. X-ray diffraction imaging was used to examine the distribution of structural defects such as dislocations and stacking faults. Examination showed regions of high line defect density, primarily dislocations, as well as regions that were relatively free of line defects (Fig. 1A). The regions that were relatively free of line defects appeared as light-regions in contrast to the defected regions where the images were very dark. Details of the examination can be found elsewhere.² By performing depth-resolved, micro-Raman spectroscopy, it was found that as much as 130 MPa of compressive stress was incorporated on the surface of the heavily defected regions. In the defect-free regions, the stresses were essentially non-existent as expected (Fig. 1B). Details of the stress measurement technique using micro-Raman spectroscopy can be found

* Present address: Novellus Systems, San Jose, 95051, U.S.A., MAPlano@aol.com

elsewhere, as well.³ The uniqueness of the sample was having both defected and defect-free regions in the epilayer which resulted in an opportunity to examine the influence that these structural defects have on carrier transport properties. In this work, the magnitude of the effects on the transport properties due to these defects is measured with transient photoconductivity.

Figure 1: X-ray diffraction image of the epilayer (A) and the corresponding depth-resolved micro-Raman stress measurements at different regions on the epilayer (B).

EXPERIMENT

Sample Preparation

Before the sample was prepared for electrical measurements, it was cleaned by a standard procedure. After the growth and x-ray and Raman spectroscopic examinations, the samples were cleaned with hot chromic acid which was prepared as a saturated solution of chromium trioxide in sulfuric acid to eliminate any non-diamond phases on the sample surface. The samples were then rinsed in de-ionized water followed by rinses in 0.1 M NH_4OH and 0.1 M HCl . The samples were again rinsed in de-ionized water followed by RCA-I and -II clean to eliminate any organic and inorganic residues. Finally, the sample was rinsed in de-ionized water and dried.

In order to examine the carrier transport properties of defected and defect-free regions, microstrip transmission line structures were fabricated on the regions (Fig. 2). The structures were fabricated using Ti/Au metals with the thicknesses of 500 Å and 5000 Å, respectively and with the stripline width of 1 mm. The metal deposition was accomplished with a sputter deposition system. Gaps of 1 mm in the striplines were opened to be used as an photoexcitation area. The total area for photoexcitation was $1 \times 1 \text{ mm}^2$. After the fabrication, the sample was incorporated into a 50-Ω, microstrip transmission line on an alumina substrate (Fig. 3). One millimeter-wide indium strips were used to make the electrical connection between the striplines on the sample to the striplines on the alumina.

Photo-excitation Source and Photocurrent Recording Apparatus

The photo-excitation of the sample was accomplished using a ultraviolet laser. The laser was Ar-ion laser pumped, mode-locked Ti:Sapphire laser. The output of the mode-locked laser was amplified with a 1 kHz Ti:Sapphire regenerative amplifier which was pumped by a frequency-

doubled Nd:YLF laser. At this stage, the pulse width was compressed to 150 fs at a wavelength of 830 nm with the output energy of 800 μ J. Using KDP and BBO non-linear crystals, the

A)

B)

Electrically Probed Region

Microstriplines

Figure 2: An x-ray image of the sample and the locations of the probed regions (A) and the micrograph of the sample with the enlarged area showing the locations of the microstriplines (B).

Figure 3: A schematic diagram of the sample/transmission line package used for the measurements.

frequency of the laser was doubled, then quadrupled to a wavelength of 207 nm and a pulse width of 150 fs at a repetition rate of 1 kHz. The energy per pulse at 207 nm was nominally 1-3 μ J with pulse-to-pulse variation of 5-10%. The absorption depth at this wavelength was 1-2 μ m from the epilayer surface. The laser-energy drift during 8 hours of operation was less than 10%, typically. About 10% of 1 ω light was split from the main beam in order to excite a neutron-damaged, 50-ps GaAs photoconductor which in turn was used to trigger the 20-GHz sampling oscilloscope used for recording the photocurrent.

At the output of the BBO-crystal, the laser spot size was approximately 4-5 mm in diameter. In order to minimize the electron-hole scattering effects on the carrier mobility and lifetime measurements, the laser intensity was reduced with the use of neutral density filters. The spot was apertured to 1 mm² so that only the active region on the sample was excited. The nominal carrier density excited was approximately $2\text{-}5 \times 10^{13} \text{ cm}^{-3}$. Temporally-resolved photocurrent was delayed by approximately 50 ns with respect to the trigger. Electrical transient was recorded with a 20 GHz sampling Oscilloscope.⁴ The deconvolved system recording time was approximately $(65 \pm 10) \text{ ps}$. The complete schematic diagram of the photoexcitation and the recording system can be seen in Fig. 4.

Figure 4: A schematic diagram of the transient photoconductivity excitation and recording apparatus. Signal waveforms were recorded with a 20 GHz sampling oscilloscope.

RESULTS

Temporally-resolved transient photoinduced current waveforms are recorded. The waveforms are used to deduce carrier mobility and lifetime. Because the absorption of 207 nm photons occurs within 1-2 μm in diamond, the measurement only probes that distance from the surface. Because the pulse width of the excitation source is much shorter than a nominal carrier lifetime in CVD diamond of 0.1-1 ns, the excitation is considered to be an impulse. The carrier lifetime is deduced from the photoinduced current decay and the mobility is deduced from the integration of the induced current and the deduced carrier lifetime. Full detail of this technique to deduce carrier lifetime and mobility can be found elsewhere.⁵

The mean carrier lifetimes in the defected and defect-free regions measured with transient photoconductivity are shown in Fig. 5 as a function of applied electric field. The mean lifetime in the defected region over the entire electric field range is about 120 ps whereas the defect-free region has a lifetime of 110 ps. In fact, the lifetime on the average appears to be slightly larger in the defected region. The carrier lifetime appears to have very little electric field dependence for both regions except at the lowest field. This is mainly due to the baseline noise in the waveform at low fields that makes the lifetime deduction difficult. The electric field dependence shown here is contrary to what was reported in the past.⁶

Results are contrary to the expectation that the line defects have an effect on the carrier lifetime through trapping and recombination at these sites. The measurement suggests that the dislocations may not play a dominant role in determining the carrier trapping/recombination in the epilayer. Instead, the lifetime is likely to be dominated by impurities such as nitrogen and other point defects because no special efforts were made to reduce the impurities that may be present in

the precursors for the epilayer growth. Nitrogen is already known to be incorporated in CVD diamond at a level of 10^{17} - 10^{18} cm^{-3} due to impurities in the feed-gases.⁷

Figure 5: Mean carrier lifetime as a function of applied electric field for the defect-free and defected regions.

Figure 6: Mean carrier mobility as a function of applied electric field for the defect-free and defected regions.

The measured mean carrier mobility as a function of applied electric field is shown in Fig. 6. The major source of uncertainty in the measurement is the energy variation from pulse to pulse.

Furthermore, any variation in the baseline of the recorded waveforms will also contribute to the error in the mobility measurement. The error is estimated to be approximately 15%.

The mobility appears to be similar in the two probed regions. The decrease in the mobility as a function of applied electric field in both cases is due to carrier drift velocity saturation. Within the errors of the measurement, the mean carrier mobility for both regions appears to be approximately the same. Again, similar to the lifetime measurements, the line defects causing the stresses in the epilayer appear to have very little effect on the carrier mobility. This implies that the line defects play only a minor secondary role in carrier scattering compared to other scattering mechanisms.

CONCLUSIONS

Effects of line defects on carrier transport properties have been measured for a high quality homoepitaxial film using transient photoconductivity. The mean carrier mobility and lifetime appear not to be affected by the existence of these defects in the epilayer. Even with the incorporation of line defects at large densities, the mean carrier mobility appears to suffer little degradation. Presently, the transport properties are dominated primarily by impurities and other point defects. Therefore, the effects of line defects are not observable. Unless concentrations of point defects and impurities can be greatly reduced, the elucidation of the effects of line defects such as dislocations on the transport properties in diamond may be difficult.

ACKNOWLEDGMENTS

We thank Dr. William Banholzer of General Electric, Co. for supplying the single crystal substrate. We would also like to thank Brent Stuart and Steve Herman at Lawrence Livermore National Laboratory for their help. One of us (S. Han) would like to thank A. Lalime for her assistance. This work was performed under auspices of the U. S. Department of Energy by Los Alamos National Laboratory under Contract No. W-7405-Eng-48.

REFERENCES

- [1] D.R. Black, H.E. Burdette, L. Robbins, M.A. Plano, M. Dale Moyer, M.A. Moreno, L.S. Pan, D.R. Kania and W.F. Banholzer, *Advances in New Diamond Science and Technology*, S. Saito, N. Fujimori, O. Fukunaga, M. Kamo, K. Kobashi and M. Yoshikawa, Editors, MYU, Tokyo (1994) 251.
- [2] D.R. Black, H.E. Burdette, M.A. Plano, M. Dale Moyer, M.A. Moreno, J.W. Ager III and A.L. Chen, "Observation of Individual Defects in a Homoepitaxial Diamond Film", the Fourth International Symposium on Diamond Films, K.V. Ravi, J.P. Dismukes, J.L. Davidson, K.E. Spear, R.H. Hange, B. V. Spitsyn, Editors, Reno, Nevada, U.S.A, May 1995.
- [3] J.W. Ager III and M.D. Drory, *Phys. Rev. B*, **48** (1993) 2601.
- [4] Hewlett Packard 54120A Main Frame with 54121A 4-channel 20-GHz Sampling Head.
- [5] L.S. Pan, D.R. Kania, P. Pianetta, J.W. Ager III, M.I. Landstrass, S. Han and O.L. Landen, *J. Appl Phys*, **73** (6) (1993) 2888.
- [6] L.S. Pan, S. Han, D.R. Kania, M.A. Plano and M.I. Landstrass, *Diamond and Related Materials*, **2** (1993) 820.
- [7] M. Hoinkis, E.R. Weber, M.I. Landstrass, M.A. Plano, S. Han and D.R. Kania, *Appl Phys Lett*, **59** (15) (1991) 1870.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.