Journal of ELECTRICAL ENGINEERING, VOL. 54, NO. 3-4, 2003, 96–99

COMMUNICATIONS

INVESTIGATION OF MESFET STRUCTURES BASED ON POLYCRYSTALLINE DIAMOND FILMS GROWN BY MODIFIED HF CVD TECHNIQUE

Alexander Kromka — Michal Kubovič — Frantiek Balon
Ján Janík — Viera Dubravcová *

We have investigated the influence of process conditions on growth of diamond thin film on silicon substrates for electronic applications. The developed double-bias HF CVD system allows an igniting of plasma at DC and AC voltages. Low voltage nucleation process results in improved nucleation in the early stage of diamond growth. Furthermore, next post-processing of such films by microwave CVD plasma is used to build conductive layer. The problem associated with the doping has been overcome by using of p-type surface conductive layer, naturally built on and in hydrogen terminated diamond surfaces. The influence of surface morphology on I-V characteristics of realized metal-semiconductor field effect transistors (MESFET) is discussed.

K e y w o r d s: diamond, hot-filament CVD, nucleation, MESFET

1 INTRODUCTION

An excellent combination of intrinsic properties of diamond offers wide-range possibilities for the fabrication of microelectronic devices operating until hostile conditions, like high temperature and high frequency, hazardous chemical environments, radiation, *etc* [1]. Several discrete diamond based electronic devices have been achieved mainly on natural and high-pressure/high-temperature synthetic diamond in the past decade [2]. However, the use of such diamond limits broad applications because of high cost, limited active area, and complicated device integration.

Today, researchers over world try to span these problems by using of diamond thin films grown on foreign substrates by several low-pressure/low temperature techniques. The list of deposition techniques for diamond thin film growth includes various plasma assisted chemical vapour depositions (CVD) at microwave, RF, and DC frequencies, and hot filament CVD [3].

However, CVD diamond films grown on Si substrate are mostly polycrystalline in nature resulting in their limited application for microelectronics. Surface and bulk defects, like grain boundaries and/or impurities, decline their electrical and thermal properties (i.e. carrier mobility, thermal conductivity, *etc*). In addition, limited doping methods shift a diamond based electronic still to state-of-art and know-how position. In 1989, Landstrass and Ravi [4] discovered a surface conductivity on hydrogen-terminated diamond films, known as p-type subsurface conductive layer. This effect substantially extends family of diamond applications without using the doping process.

The aim of this study is to demonstrate abilities of HF CVD technique for large area deposition of polycrystalline diamond films with enough good quality for realization of electronic structures. We have investigated the electrical behavior of diamond based MESFET structures as a function of nucleation conditions and post-processing procedure. In addition, basic technological steps used in the fabrication of H-terminated diamond based active electronic devices are presented.

2 EXPERIMENTAL

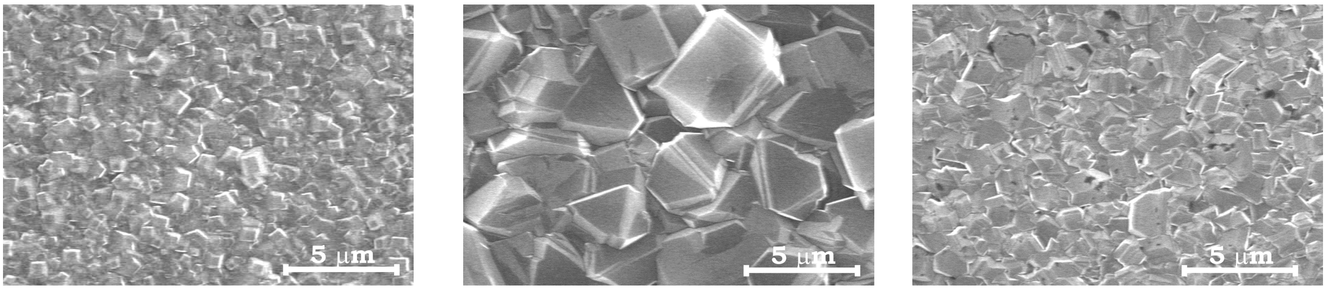
2.1 Diamond growth.

In this study, diamond thin films were grown on mirror polished silicon substrates by dual plasma HF CVD system [5].

Sample A was nucleated for 75 minutes at negative DC substrate bias voltage of 170 V and substrate temperature of 650 °C, in a mixture of methane (3 sccm) and hydrogen (300 sccm), at total gas pressure of 3000 Pa. Then, a standard HF CVD growth started for 3 hours at 1 % of methane and followed by 3 hours growth at decreased methane content ($CH_4 : H_2$ = of 2.5 : 500 in sccm). All other parameters, filament temperature, substrate temperature, total gas flow, *etc* were kept constant. Next, three-step microwave CVD post-processing was applied with introduced nitrogen for 10 minutes. Finally, polycrystalline diamond surface was terminated by MW CVD in hydrogen plasma for 10 minutes, at substrate temperature of 500 °C, and a total gas pressure of 4000 Pa.

* Department of Microelectronics, Slovak University of Technology Ilkovičova 3, 812 19 Bratislava, Slovakia, e-mail: kromka@elf.stuba.sk

ISSN 1335-3632



sample A, film thickness 3 m sample B, film thickness ~4.7 m sample C, thickness 3 m

Fig. 1. Surface morphology of grown diamond layers

Sample B was pretreated for 2 hours, at changed gas flows (methane : hydrogen = 2 : 400), and low negative DC voltage (120 V).

Sample C was nucleated also for 2 hours. In this case, AC bias voltage of 160 V (50 Hz) was applied between the filaments and substrate. All other process conditions (substrate temperature 650 °C, total gas pressure 3000 Pa, filament temperature 2000 °C) were constant in the nucleation and growth step. Both, sample B and C, were grown for 20 hours. Then, chemically and plasmatically cleaned films were terminated by hydrogen in microwave plasma (identically like in the case of sample A). Surface morphology of all samples grown and their thickness are summarized in Fig. 1.

2.2 Diamond preprocessing

Before hydrogen termination, diamond films were cleaned in $CrSO_4$ acid at 150 °C (30 minutes) in order to remove any graphitic components from its surface. Next, purged films in deionized (DI) water were cleaned in nitric acid to etch any chromium rest from the diamond surface. Finally, samples were cleaned in solutions of H_2V_2 : H_2SO_4 (1 : 2) for 10 min and rinsed in DI water. In addition, samples B and C were etch in oxygen plasma for 5 minutes. Such cleaned samples were processed in hydrogen plasma to build the active p-type surface and/or sub-surface channel, as mentioned above.

2.3 Fabrication of FETs

After hydrogen plasma treatment, diamond films were covered by gold layer (300 nm thick). First, basic MESA structures were fabricated by optical lithography (the first lithographical step). Gold layer was patterned in KI/I_2 solution and subsequently washed in DI water. Then, samples were treated in oxygen plasma to build isolation between MESA structures. Thus, the surface p-type layer is restricted to the region under the Au layer. The second lithographical step formed ohmic contacts in rectangular and/or Corbino geometry. Once again, gold was etched in potassium iodide to form source and drain, with a H-terminated channel between them.

Gates were fabricated by using the self-aligned strategy. In this case, aluminum was evaporated onto formed structure. The under-etch of the gold beneath the photoresist defined the separation distance between a gate and Au ohmic contacts. Aluminum gates were patterned by the LIFT-OFF technique. A typical top-view of realized testing structure was previously shown in [11].

3 RESULTS AND DISCUSSION

I-V measurements confirmed near-ohmic nature of Au contacts, whilst Schottky contact was formed under Al. The main origin for this behavior is due to different electronegativities of the used metals and indicates also the state of H-terminated diamond surface, which displays little Femi level pinning [6].

Figure 2 shows I-V characteristic at room temperature for Al contact on sample A. Fabricated Schottky diode remains stable with no leakage current at reverse bias of 100 V. In forward biasing, instability is observed at bias voltage of 30 V. At this bias voltage, slope of the I-V curve slightly decreases down. For higher forward bias range than 30 V, the frequency and amplitude of these instabilities increases resulting in breakdown at voltage of ~ 70 V. This instability could occur due to a slight overheating of conductive layer, and consequent thermally promoted redistribution of hydrogen within the surface region [7]. In addition, H-terminated surface is sensitive not only to temperature (max. to 150 °C) but also to gas environment (mainly to oxygen). The redistribution of hydrogen within the surface could lead in increase of sheet resistance resulting in change of the slope of I-V curve (Fig. 2).

Figures 3–5 show I-V measurements of realized MESFET structures on sample A, B, and C, respectively. Generally, all structures exhibit a clear modulation of channel current as a gate bias voltage is applied. A drain-source current, I_{DS}, considerably increases with increase of U_{GS}.

No-ideal channel pinch off is observed for gate voltages in the case of sample A. This sample shows relatively high

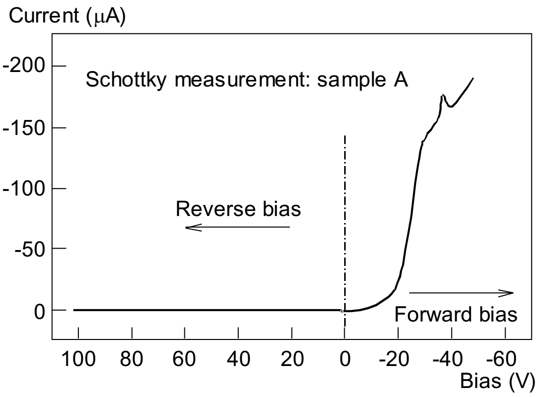


Fig. 2. I-V characteristic for Al contact to H-terminated diamond of sample A

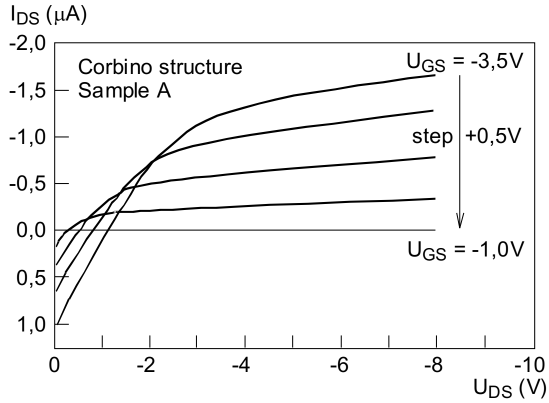


Fig. 3. MESFET source to drain I-V measurements *vs* gate bias of "Pie" structure fabricated on sample A

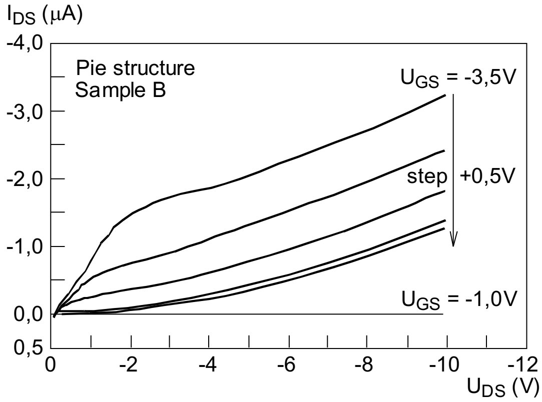


Fig. 4. MESFET source to drain I-V measurements *vs* gate bias of "Pie" structure fabricated on sample B

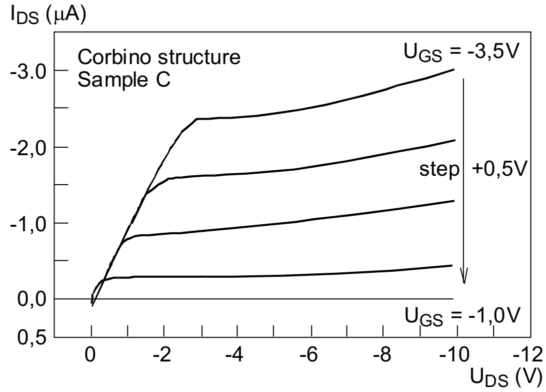


Fig. 5. MESFET source to drain I-V measurements *vs* gate bias of "Pie" structure fabricated on sample C

leakage current, and Corbino structures showed acceptable modulation of source-drain current. Realized "Pie"-type structures exhibited more linear-like modulation and no pinch-off of channel was achieved. It is believed that this is caused by imperfect contact of gate metal to the surface due to relatively high surface roughness, as observed by SEM measurements (Fig. 1).

For sample B, channel pinch-off is observed only for the gate voltage of ~ 3.5 V. Further decreasing of gate voltage down results in modulation of I_{DS} current, and a lack and/or lost of channel pinch-off is observed. On the other side, sample C reveals nearly ideal MESFET characteristics. For all applied gate voltages is observed low leakage current and full channel pinch-off. At gate voltage of -1.0 V, no source-to-drain current is measured indicating p-type MESFET transistor.

The origin of different MESFET characteristic could be explained by varied nucleation conditions. First, nucleation is known to play a crucial role in diamond growth, influencing its homogeneity, crystallography, growth rate, etc. In the case of samples A and B, DC bias voltage was applied during the nucleation step. Previously we found that the DC bias substrate pretreatment influence the film quality, surface roughness, crystal size and orientation [8]. Lower substrate bias voltage results in (100)-like diamond growth. On the other side, higher substrate bias pretreatment yields polycrystalline films with twinning and edge cracking features. Also, non-diamond carbon phases were detectable by Raman spectroscopy. It means that the "quality" of fabricated MESFET structures on such substrates will show non-homogeneous features characterized also by a lack of channel modulation and its pinch-off due to imperfect contact of gate metal to the diamond surface.

In addition, the maximum value of measured current (I_{DS}) of all samples still remains in order of few microamperes. In the case of sample A, this could be explained through incorporated nitrogen in the bulk of grown films. A nitrogen-methane-hydrogen gas mixture was used for a short period of diamond growth in order to improve crystallite formation. Both effects, either change of the positions of Fermi level or compensation of the surface (surface-near) acceptors due to nitrogen donors could result in suppression of the p-type conductivity of H-terminated diamond surface [9].

On the other side, samples B and C were grown without introducing any nitrogen. However, both samples showed relatively low saturation current. This could be explained via no-optimized process conditions for hydrogen termination of diamond surface. In this study we used relatively low substrate temperature (550 °C), whilst some authors prefer temperature of 700 °C and higher [9].

However, I-V measurements over sample C demonstrated interesting results — growth of diamond films on AC bias pretreated silicon substrates reveals enough good film quality to fabricate simple MESFET structures. This result opens new promising applications, where polycrystalline diamond layers could be homogeneously deposited also on non-conductive substrates (like glass — where DC bias pretreatment hindered low nucleation yield [10]) in order to build a diamond-based sensor for monitor of biomedical reactions in human body.

The presented results clearly demonstrate an ability of the modified HF CVD system to successfully achieve diamond nucleation under various substrate biasing (DC and AC bias enhanced nucleation). In addition, we believe that the optimizing of process conditions will lead in improved MESFET quality for both bias pretreatment methods. Such investigations are in progress.

4 CONCLUSIONS

Polycrystalline diamond films were grown on Si substrates by modified HF CVD technique. I-V measurements of fabricated MESFET structures displayed importance to optimize nucleation conditions. Next, diamond films were successfully and homogeneously deposited also on AC bias pretreated silicon substrates. Such films were enough good in quality to demonstrate basic MESFET transistor characteristics. All structures were realized by using p-type surface conductive layer, naturally built during the hydrogen termination of diamond surfaces. Presented results indicated new applications of polycrystalline diamond films grown on insulating substrates. Further optimization of diamond growth conditions is required in order to achieve better MESFET output characteristics.

Acknowledgement

This work has been supported through the project grants VEGA No. 1/7629/20 and 1/9044/02 of the Slovak Republic.

References

[1] DAVIS, R. F.: J. Vac. Sci. Technol. **A11** (1993), 829.

[2] GROT, S. A.: In Diamond — Electronic Properties and Application, edited by L. S. Pan, L.S.— Kania, D.R., Kluwer Academic Publisher, Boston, 1995, p. 443.

[3] LI, H.—DANDY, D. S.: Diamond Chemical Vapor Depositions — Nucleation and Early Growth Stages, Noyes Publication, New Jersey, 1995.

[4] LANDSTRASS, M. I.—RAVI, K. V.: Appl. Phys. Lett. **55** (1989), 1391.

[5] KROMKA, A.—MALCHER, V.—ČERVEŇ, I.: Acta Physica Slovaca **51** (2001), 27.

[6] LOOI, H. J.—PANG, L. Y. S.—FOORD, J. S.—JACKMAN, R. B.: Solid-State Electronics **42** (1998), 2215.

[7] LOOI, H. J.—PANG, L. Y. S.—MOLLOY, A. B.—FORD, F.—JACKMAN, R. B.: Diamond Relat. Mater. **7** (1998), 550.

[8] KROMKA, A. *et al*: to be published in Diam. & Relat. Materials.

[9] KAWARADA, H.: Surf. Sci. Rep. **26** (1996), 205.

[10] KROMKA, A. *et al*: to be published in Thin Solid Films.

[11] KUBOVIČ, M.—BALON, F.—KROMKA, A.—JANÍK, J.—DUBRAVCOVÁ, V.: The First Diamond Based FETs Realized at the Department of Microelectronics, In Final Programme and Book of Abstracts, Proc. of 8th International Workshop on Applied Physics of Condensed Matter — APCOM 2002, (2002, June 19–21), Liptovský Mikuláš — Demänovská Dolina, Slovakia, p. 17–22, (ISBN 80-8040-186-1).

Received 10 December 2002

Alexander Kromka (Ing), born in Nitra in 1971. He received his MS degree in electronic engineering from the Slovak Technical University, Bratislava, in 1995. He has been a PhD student at the Department of Microelectronics of the Faculty of Electrical Engineering and Information Technology, since February 1996. His research interests include diamond thin films deposition techniques and its application for microelectronic devices. He spent 3 months at Institut fü r Festkö rper Elektronik in Ilmenau, Germany, in 1995, where he worked on the project Multilayered and Quasi-Superlattice Structures for Sensors and Photovoltaics in the field of the investigation of AlN and SiC thin films.

Michal Kubovič (Ing), born in Myjava in 1977, received his Ing (MSEE) degree in electronics from the Slovak University of Technology, Bratislava, in 2001. He is currently working towards the PhD degree at the University of Ulm, Germany.

Frantiek Balon (Ing), born in Ilava in 1977, received his Ing (MSEE) degree in electronics from the Slovak University of Technology, Bratislava, in 2001. After graduation, he spend 13 months at University of Ulm, Germany, where he worked on the processing, design and characterization of novel GaN based HFETs. Now he is currently working towards the PhD degree at the University of Surrey, United Kingdom.

Ján Janík (Doc, Ing, PhD), born in Handlová in 1946, received his Ing (MSc) degree in solid state physics, in 1970 and the CSc (PhD) in applied physics, in 1981, both form the Slovak University of Technology, Bratislava. Since 1992 he has been Associate Professor in Electronics at the Departmnet of Microelectronics of Slovak University of Technology. In research he was oriented to ion beam optics and plasma technology. He spent some time at scientific stays abroad. Long-time stay in Dubna, Russia (1987-89) as a member of Cyclotron Design Group, then in CERN, Switzerland (1993-94) as ion-beam optics designer for the AUSTRON project and six short-time stays in GSI Darnstadt, Germany (1992-96) as ion-beam optics designer for a medical accelerator. He collaborate on hadron therapy project, sponsored by oncology 2000 in Czech Republic.

Viera Dubravcová (Doc, Ing, PhD), born in 1942, received the Ing degree in 1964 and the CSc (PhD) degree in 1982, both in microelectronics, from the Slovak Technical University, Faculty of Electrical Engineering, Bratislava. Since 1985 she has been Associate Professor of vacuum physics and technique at the Department of Microelectronics, STU. The measurement of low pressures is the main topic of her research activity.