

# DIAMOND FILMS

Chemical Vapor Deposition for  
Oriented and Heteroepitaxial Growth

Koji Kobashi

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## CHEMICAL VAPOR DEPOSITION FOR ORIENTED AND HETEROEPITAXIAL GROWTH

Koji Kobashi

2005



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# **DIAMOND FILMS**

## **CHEMICAL VAPOR DEPOSITION FOR ORIENTED AND HETEROEPITAXIAL GROWTH**

## Preface

The major purpose of this monograph is to review process technologies for oriented and heteroepitaxial growth of diamond by microwave plasma chemical vapor deposition (CVD). There are many CVD methods to synthesize diamond films using, for instance, hot filament, radio-frequency (rf) plasma, microwave plasma, DC plasma, arc-jet plasma, and combustion flame. Among these, this monograph mainly focuses on microwave plasma CVD. The microwave plasma is an electrode-free discharge so that the contamination of diamond films by electrode materials can be avoided. The microwave plasma is very stable over many days, and a subtle control of the plasma, and hence the properties of synthesized diamond films, is possible. This is particularly important to quantitatively investigate the diamond nucleation and the film morphology in oriented and heteroepitaxial growth. This monograph also includes research results that are related to oriented and heteroepitaxial growth.

Historically, a vapor growth of diamond was first demonstrated by W. G. Eversole, which was followed by an intensive research in B. V. Derjaguin's group in Russia and J. Angus' group in the USA. A revolutionary advancement in diamond CVD occurred in the early 1980s, when B. V. Spitsyn et al. demonstrated a growth of diamond particles by CVD (by the chemical transport method), and N. Setaka and co-workers disclosed three methods to synthesize diamond films: hot filament, rf plasma, and microwave plasma. This triggered intensive R&D in Japan, Europe, US and then all around the world. The progress of diamond film research has been very steady and consistent, and numerous findings have been made during the past two decades.

One of the most remarkable findings in the area of diamond CVD is the growth of azimuthally oriented diamond films with (100) faces aligned in the same direction at the film surfaces by B. Stoner and J. Glass in 1992. Such films are now called highly oriented diamond (HOD) films. A key to make HOD films is to use bias-enhanced nucleation (BEN) discovered by S. Yugo, and thus a number of works have been done on diamond nucleation using BEN. The standard substrates for HOD film synthesis are  $\beta$ -SiC(100) and Si(100). One of the best-coalesced films was successfully synthesized by H. Kawarada, P. Koidl and their co-workers, as well as X. Jiang and his co-workers. In the mid 1990s, it was discovered that (111) and (100)-oriented, spontaneously coalesced diamond films could be synthesized on Pt(111) and Ir(100) by Y. Shintani and A. Sawabe, respectively, showing the possibility of producing single crystal diamond films by CVD. Since the growth rate of diamond films has

been remarkably increased in recent years from the past rate of  $\sim 0.2 \mu\text{m/h}$  to  $\sim 50 \mu\text{m/h}$ , and large CVD reactors of maximum 60–100 kW are commercialized, production of single crystal diamond plates is expected to begin in the not-too-distant future.

Under these circumstances, it would be of significance to review the articles on oriented and heteroepitaxial growth of diamond films by CVD, and particularly summarize the processing conditions for the readers to further develop and elaborate the science and technology of diamond CVD. It is expected that this monograph would be useful for such purposes.

In completing this monograph, I must acknowledge many people. First of all, I wish to thank late V. Chandrasekharan, D. Fabre, M. L. Klein, and R. D. Etters for my education in physics before I became an independent researcher. On diamond CVD, I am particularly indebted to N. Setaka, Y. Sato, and M. Kamo for kindly allowing me to learn diamond CVD in their group. I certainly would like to thank my colleagues in Kobe Steel, Ltd.: K. Nishimura, K. Miyata, T. Tachibana, Y. Yokota, K. Hayashi, and N. Kawakami. I wish to particularly thank T. Tachibana for supplying a number of papers related to heteroepitaxy. I would also like to thank my superiors in the company: T. Horiuchi, Y. Kawate, Y. Kawata, Y. Sugizaki, J. Miyazaki, H. Sato, and S. Suzuki for their consistent support of R&D of diamond films over two decades. A longtime friendship with J. Glass, R. Nemanich, and A. Gicquel was a strong encouragement for me to write this monograph. Finally, I would like to thank my wife Toshiko and my son Akira for creating a calm environment for me to work at home. This monograph would not have been published without the support of the people mentioned above.

Finally, I wish to acknowledge I. Craig, S. Foster, and N. Jones of Elsevier Ltd. for their very kind assistance in publishing this monograph.

Koji Kobashi  
2005

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In this monograph, a number of figures and tables are quoted from different sources, and the author would like to thank the following organizations and individuals for generously giving permission to use them in this monograph.

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Chapter 1

**Overview of Oriented Growth**



## Chapter 1

### Overview of Oriented Growth\*

Science and technology of diamond film growth by chemical vapor deposition (CVD) have markedly advanced during the past decade. One of the most notable achievements is the growth of azimuthally oriented diamond particles on  $\beta$ -SiC by Stoner and Glass [1, 2] using Yugo's method of bias enhanced nucleation (BEN) [3], which then led to the growth of (100)-oriented diamond films on Si(100) that were later named as highly oriented diamond (HOD) films [4]. This technique was further elaborated by the groups of Glass, Koidl, Klages, and Kobe Steel [5] amongst others. Most recently, Kawarada et al. [6, 7] were successful in growing perfectly coalesced, (100)-oriented, 300- $\mu$ m thick HOD films, where there was no grain boundary at the film surface. In the meanwhile, a new method of diamond heteroepitaxy was heuristically found by Shintani [8, 9], i.e. spontaneously coalesced, (111)-oriented diamond films can be grown on (111) surfaces of Pt that have been polished with diamond powder for diamond nucleation. This finding was followed by diamond heteroepitaxy on Ir(100) by Sawabe's group [10] using direct current (DC) plasma CVD, in which (100)-oriented, perfectly coalesced diamond films were grown. This work was reproduced by Morooka's group [11], and Schreck and Stritzker's group [12] using microwave plasma CVD (MPCVD), where the diamond nucleation was done by BEN.

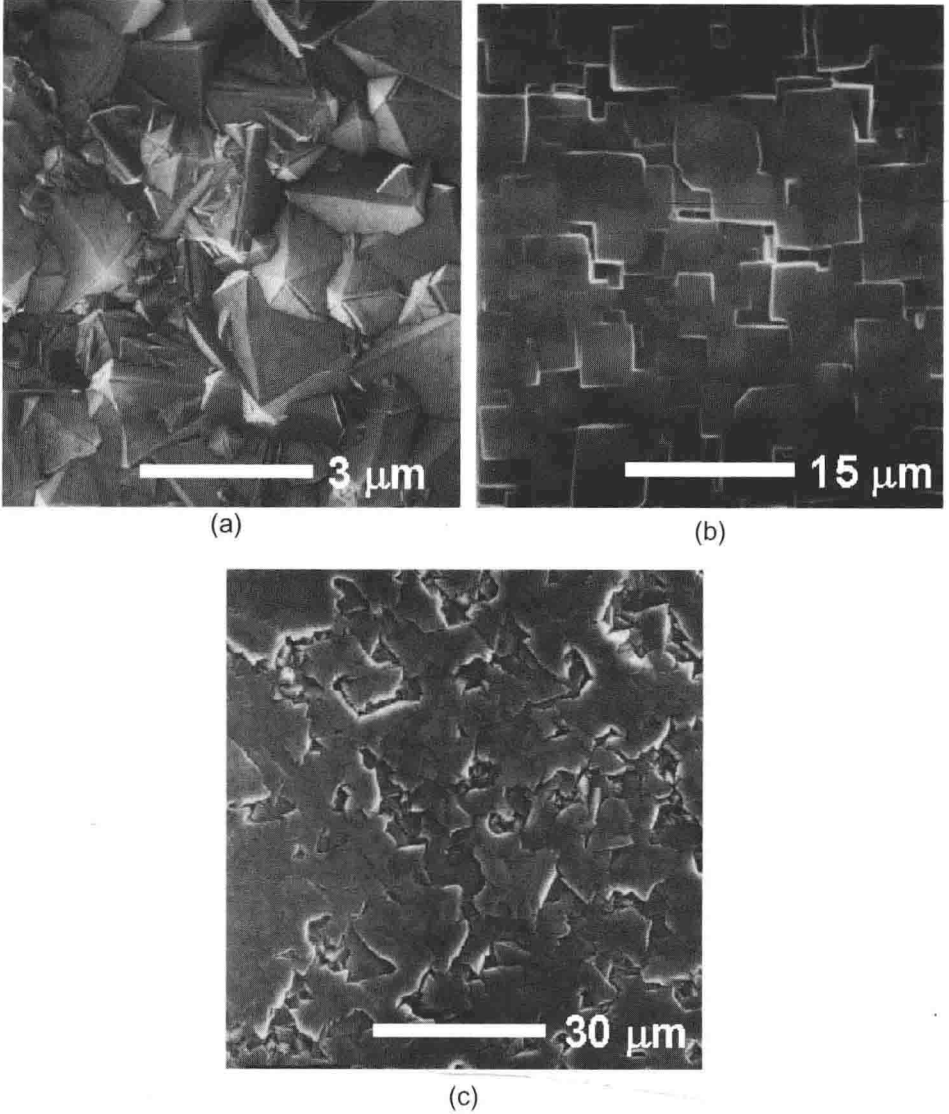
Figures 1.1 (a)–(c) show a polycrystalline diamond film deposited on Si substrate, an HOD film grown on Si(111) surface, and a partially coalesced film on Pt(111) substrate, respectively, all synthesized by MPCVD. Diamond films grown on Ir(100) are so perfectly coalesced that there is no feature in the film surface image by scanning electron microscopy (SEM) (see Figure 12.18 [13]). At the present stage, the coalesced area is the largest for diamond films on Ir(100) [25 mm] followed by those on Pt(111) ( $\sim 10$  mm, see Figure 12.8 [14]), while the crystal facets are the smallest for polycrystalline diamond films (see Figure 1.1 (a)). In the latest technology, however, the edge lengths of crystal facets become extremely large even for polycrystalline diamond films. For instance, they are

---

\*In this monograph, a number of notations, units, and abbreviations will be used, and they are summarized in Appendix A. It contains lists of notations for crystal orientations, process parameters for CVD, analytical techniques, CVD reactors, crystal growth, and carbon materials in addition to a description of standard diamond film characterizations, i.e. Raman spectroscopy and cathodoluminescence (CL). The readers are recommended to just quickly read through Appendix A at this point.

30–50  $\mu\text{m}$  for few hundred- $\mu\text{m}$  thick polycrystalline diamond films, and 50–100  $\mu\text{m}$  for HOD films.

Judging from the recent progress of R&D on heteroepitaxial growth of diamond, it seems that the growth of single crystal diamond films over significantly large areas,



**Figure 1.1.** (a) Polycrystalline diamond film deposited on Si substrate, (b) HOD film grown on Si(100) substrate, and (c) partly coalesced diamond film on Pt(111) substrate.



such as one inch in diameter, will soon be achieved by CVD, and thence electronic applications of diamond films [15–17] will proceed much faster in the coming years than in the past. One of the purposes in the present monograph is to follow the trail of science and technology of oriented diamond film growth that has involved a number of researchers mostly in the past decade. This monograph was not able to cover all the papers related to oriented and heteroepitaxial growth of diamond films, and the readers are recommended to consult excellent reviews for heteroepitaxy [18–20], and nucleation and growth [21]. The readers can also consult Ref. [22] for defects, and Ref. [23] for a brief history of diamond research before 1988. Also, useful books on bulk diamond and diamond films are available [24–31].

Apart from MPCVD for diamond film synthesis, a number of works have been published on oriented growth using hot filament CVD, DC plasma CVD, and molecular beam epitaxy (MBE) [32], but most of them are left out because this monograph mainly concentrates on the MPCVD process technology. Among such papers that we missed, interesting and important papers are; homoepitaxial growth kinetics [33]; texture and morphology of diamond films [34]; HOD film growth by electron-assisted hot filament CVD (EACVD) [35]; diamond growth on highly oriented pyrolytic graphite [36]; TEM observation of the interface between the Si(100) substrate and the HOD layer [37]; heteroepitaxial nucleation of diamond particles on Si(100) using BEN [38]; heteroepitaxially (111)-oriented diamond films on Si(100) [39]; growth of both (100)- and (111)-oriented diamond films on Si(100) and Si(111) using BEN, respectively, where  $D[110]//Si[110]$  and  $D\{100\}//Si\{100\}$ , while  $D[1\bar{1}0]//Si[1\bar{1}0]$  and  $D\{111\}//Si\{111\}$  [40]. In the above expressions, D stands for diamond, and this abbreviation will be used throughout this monograph. Likewise, a notation G will also be used for graphite. Reference [40] stated that diamond could epitaxially grow directly on Si, and the existence of the native oxide on the Si wafer gave deleterious effects on heteroepitaxy. An observation of interface structures by high-resolution transmission electron microscopy (HRTEM) [41,42] showed that there was a misorientation angle of  $7.3^\circ$  between D(100) and Si(100), but later a more detailed study showed that it was actually  $9^\circ$  [43]. Other papers left out in this monograph are; growth of (111)-oriented diamond films on 6H-SiC(0001), demonstrating that  $D[110]//6H-SiC[11\bar{1}0]$  and  $D\{111\}//6H-SiC\{000\bar{1}\}$  [44,45]; heteroepitaxial growth of diamond film on scratched Si(100), followed by  $\beta$ -SiC(111) formation at the Si surface, which demonstrated that  $D\{111\}//\beta-SiC\{111\}$  and  $D\{771\}//Si\{111\}$  [46]; effects of biasing time in the BEN step to synthesize HOD films [47]; study of BEN process by ellipsometry, showing that  $\beta$ -SiC is unnecessary for HOD film growth [48, 49]; bias current effects on nucleation density, showing that a nucleation density of  $10^9/\text{cm}^2$  at a nucleation rate of  $10^6/\text{cm}^2 \cdot \text{s}$  is most appropriate