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1 JUL 91 - 1 MAR 92

# The In Situ Observation of Diamond Film Nucleation and Growth

Submitted by

Martin E. Kordesch
Assistant Professor of Physics
Ohio University, Athens, Ohio
Tel.: (614) 593 - 1703, FAX: - 0433
electronic mail: kordesch@helios.phy.ohiou.edu

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Ohio University
Department of Physics and Astronomy
Athens, OH 45701-2979
Carol J. Blum,Administrative Contact

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**13. ABSTRACT** (Maximum 200 words)

The growth of a two-dimensional ordered carbon monolayer on Mo(100) has been observed under conditions approaching those used for diamond CVD using a photoelectron emission microscope. The method used is a combination of HFCVD and atomic layer epitaxy, using a strongly chemisorbed oxygen layer as a nucleation inhibitor. Site-for-site place exchange of carbon for oxygen, that preserves the surface ordering, has been observed. Extension of this technique to surfaces that are templates for diamond epitaxy is being investigated.

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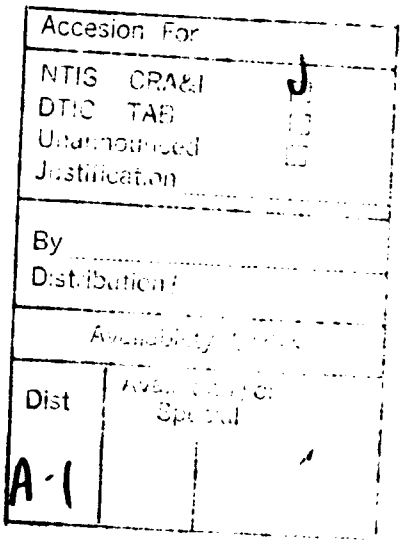

Introduction:

Due to the high temperature and high pressures used in the CVD diamond process, very few analytical methods are applicable to in situ observation of CVD diamond nucleation and growth [1]. In addition, due to the use of plasma discharges to excite or activate the feed gasses, most electron-based analyses are unable to cope with the deposition environment. The common electron microscopes are either "blinded" by substrate temperature (SEM) or unsuited to thick samples (TEM). Emission microscopy offers a possibility for in situ observation, since this non-scanning imaging technique is compatible with 1500 K temperatures and pressures near 0.1 Torr. Several of the non-plasma deposition techniques are adaptable to observation with low energy electron microscopy, photoemission and thermal emission microscopy. The in situ observation of diamond nucleation is essential, since the successful growth (a problem distinct from nucleation) of epitaxial, electronic-grade CVD diamond on non-diamond substrates will depend on the complete control of nucleation.

Emission microscopy has been known since 1930, but was ignored for several decades due to the theoretically postulated resolution limit on the order of 2 um [2-5]. More complete analysis, especially for LEEM [6], has shown that 10nm lateral resolution is possible with uncorrected lenses, and possibly better resolution with advances in electron optics [7]. The emergence of surface science in the last decades has spurred a rediscovery of emission microscopy, because the emission microscope is a high contrast method, able to detect and image singe layers of adsorbed gasses and metals on solid surfaces [8-10], and to provide both real space images and diffraction patterns [11-13] in real time at video rates (30 millisec/frame). We have already applied the photoemission microscope to the deposition of diamond films [14]. The combination of hot filament assisted CVD and atomic layer epitaxy methods has allowed us to observe the initial stages of carbon deposition on Mo surfaces in situ under conditions approaching those used for polycrystalline CVD diamond growth.

The most severe limitation on the PEEM microscope is the operation of the image intensifier at the pressures needed for CVD diamond deposition (see below). In order to eliminate this limitation we have devised a method to shield the substrate from the hot filament, which accomplishes two goals. First, the electrons from the filament are kept from entering the imaging optics, secondly, no filament contamination occurs at the substrate. As a bonus, the directed gas stream increases the partial pressure of the reactants at the substrate, while limiting the total pressure in the analysis chamber.

Experimental:

A diagram of the HFCVD arrangement is shown in Figure 1. The gas mixture is introduced through a stainless steel capillary that is at the end of a variable leak valve and flow controller.

The images in Figure 2 were recorded from a video tape of carbon deposition from 5% methane/hydrogen using the HFCVD method and dosing device described above. The substrate is Mo(100), held at 1200 K during observation. Pressures in the chamber were regulated to control the advance of the film deposition front. This last point is significant: the advance of the nucleation front (the bright side of the image is an ordered overlayer of carbon) was controlled by pressure variation, and could be halted or advanced at will.

In the series of micrographs in fig. 2 a form of atomic layer epitaxy was used to control secondary nucleation of carbon. The Mo(100) surface was covered with a single layer of oxygen. The presence of adsorbed oxygen was confirmed with both LEED and ESCA. Oxygen is strongly adsorbed on Mo(100), and it does not desorb at the typical HFCVD substrate temperatures [15,16]. At 1200 K, only a chemical reaction to form a species less strongly bound will displace the adsorbed oxygen. In the methane/hydrogen gas stream, such a reaction occurs during the deposition of carbon. The reaction is inhomogeneous, and proceeds across the crystal surface as a reaction front or wave. The reaction front may be controlled by decreasing the flux of reactants to the surface of the crystal, or by raising the temperature to increase the reaction rate. The first micrograph shows the boundary between the carburized (upper left) surface and the oxygen covered (lower right) surface. In the second micrograph, the crystal was heated slightly, causing the formation of the bright band at the interface between the two surfaces.

The bright band is the explanation of the two wider bands to the left of the wavefront. The process was stopped and started by heating several times before these micrographs were taken, so that the irreversible change in the adlayer can be seen by the change in relative brightness (photoemission yield) of the bands. The width of the older bands is the lateral progression of the front during the active cycle. The brightness, and hence the electronic structure [17], of the active front is very different from the older, cooled and re-heated carbon areas.

The fact that the boundary moves forward can be observed by noting that the reaction front moves toward and surrounds the impurity nucleus (round spot with dark, raised center) in the last two micrographs. In previous experiments, it was shown that

impurities such as the one shown could serve as nucleation sites for carbon deposition. These micrographs are the first observation of controlled lateral growth of a single CVD carbon monolayer.

A further observation made with the HFCVD doser is that nuclei formed outside the field being viewed during the experimentation with the growth fronts. These structures were formed on the part of the crystal closest to the nozzle, i.e. at the point of highest surface carbon concentration. Some of these nuclei are shown in figure 3. Although the conclusive identification of the material deposited was not possible, (process stopped before detectable bulk diamond was grown), comparison with diamond grown on molybdenum foil suggests that polycrystalline nucleation occurs through the process shown in the micrographs in figure 2 and 3. The diamonds shown in figure 4 were grown in the usual manner using HFCVD on molybdenum foil in a standard reactor.

The underside of each crystallite contains the nucleus and a radiating growth pattern similar to the star-like carbon structures in figure 3. In the PEEM micrograph, several of the star-like arms are oriented at right angles to one another, along a common axis, which may be an epitaxial effect resulting from the (100) molybdenum surface orientation. It must be recalled however, that the crystallites were nucleated at 20 Torr, and the PEEM image is from a process at about $10^{-4}$ Torr. Even if the morphological similarities are strikingly similar, the actual evolution of the low pressure seeds to bulk diamond crystals must still be confirmed by direct observation [18].

Discussion:

A number of conclusions may be made from these images. First of all, lateral layer growth is possible, using the oxygen ALE method. No secondary nucleation or three-dimensional growth was observed on the oxygen covered surface for several mm to the right of the wavefront. The actual deposition of carbon occurs through an intermediate that is only present at the deposition temperature of diamond, and is irreversibly altered by cooling or removal from the gas stream. This second point may not directly hinder what are called "stop 'n grow" investigations of nucleation (i.e., where the growth is interrupted to observe the progress of the film growth or analysis), since the site may be identified ex situ. However, analysis of growth dynamics must be performed in situ.

From the work of Weber and Ostwald, it is known that semiconductors have a temperature dependent electron yield. The empirically determined equations and constants are in Ref. 17. It is likely that the bright bands observed at the leading edge of the wavefronts are locally heated by the reaction. The energy released through the recombination of atomic hydrogen in the CVD process has been measured to increase the substrate temperature by Angus and

Gat [22]. We may also have recombination of atomic oxygen and water formation at the C-O interface on the Mo surface. These reactions are spatially localized in our experiment, so that the local heating may significantly alter (and increase) the temperature at the deposition line, what we have called the "wavefront". A second possibility, is the temporary creation of a very low work function species (possibly a form of graphite) is created at the interface, but does not persist at lower temperatures. Future experiments will be directed at spectroscopic determination of the transient species in the bright bands at the growth interface.

To date, ours are the only PEEM studies of in situ carburization of substrates at conditions suited to diamond growth. Several papers have been directed at the oxidation of CO on Pt [19,9-11], and one each on molybdenum carbide formation [20] and oxidation of carbon [21] on Pt(100). In both of the latter cases, definite orientation effects have been observed between the surface lattice and the overlayer of carbide, or the direction of carbon oxidation along high symmetry surface directions. In our study, the carbon layer grown in the modified ALE-HFCVD process showed a 3X3 LEED pattern after growth. The oxygen precoverage is also a two domain 3X1 LEED structure, so that it is possible that a direct replacement of oxygen by carbon takes place on a site-by-site basis. Investigation of this phenomenon by LEEM, and of course, continuation of the growth to bulk diamond is still necessary.

SUMMARY:

Progress 1 JUL 91 to 1 MAR 92:

1. In Situ HFCVD diamond Observation with PEEM:

Using the ALE-HFCVD doser we have been able to observe the deposition of ordered carbon monolayers on Mo(100). Initially, the growth of carbon layers was attempted in order to determine if the excess electrons from the hot filament interfered with the electron microscope. In these first experiments, nucleation was observed at impurity sites, and the subsequent film growth was observed at low pressures ($10^{-6}$ Torr) so that growth of films with 100 to 500 um lateral size took several hours.

After the successful testing of the HFCVD doser, higher pressures were used, with estimated equivalent pressures at the sample surface of $10^{-3}$ Torr 5% methane/hydrogen. It was discovered that the crystal edge was sufficient to nucleate macroscopic (10mm) wavefronts of carbon deposition, which resulted in an ordered 2x1 or 3x1 C overlayer on M(100) depending on temperature. Such wavefronts are shown in Figure 2. The deposition could be controlled, and secondary nucleation on the crystal inhibited by a full monolayer of oxygen preadsorbed on the Mo(100) surface. These results will be presented at the March APS meeting (Abstract enclosed)

Mechanical work for the "stop n' grow" observation of CVD diamond growth at 20 Torr or higher has also been completed. We now have a working transfer mechanism, that can transfer a sample from the growth chamber, with heating and temperature measurement contacts, to the uhv system for PEEM, AES, ESCA and EELS analysis and back into the growth chamber without breaking vacuum. This special mount also allows sample tilt in order to align the specimen with the optical axis of the PEEM.

2. LEEM:

The LEED microscope is now almost complete. We have made PEEM observations of natural diamond substrates at much higher resolution and magnification than in the dedicated PEEM. Electron reflection and LEEM images have been obtained from Pd(100) as a test specimen. We are currently waiting for a C(100) sample of sufficient size and quality to begin LEEM studies. Completion of the LEEM will entail the installation of a stigmator, the small area diffraction apertures, the lanthanum hexaboride cathode and the differential pumping for CVD. None of these additions will be necessary for the observation of the C(100) reconstruction, which

will be investigated immediately upon receipt of the sample specimen.

3. Substrates:

A source of Si(310) substrates has been identified. Ten 1" wafers will be ordered after 1-APR-92.

4. Negative electron affinity emitters/films.

It has been reported that the current density for the polycrystalline low-field-cold-emitters ( see below) may be as high as 5 Amperes/sq cm. We have filed a patent application for this technology, and will further investigate this phenomenon.

PUBLICATIONS:

An Emission Microscopy Study of Carbon Surface and Thin Film Morphology, C. Wang and M.E. Kordesch, **Ultramicroscopy**, 36 (1991) 154-163.

An UHV Photoemission Microscope for use in Surface Science W. Engel, M.E. Kordesch, H.H. Rotermund, S. Kubala and A.van Oertzen, **Ultramicroscopy**, 36 (1991) 148-153.

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In Preparation:

An UHV Sample Transfer and Tilting Mechanism for High Temperature Thin Film Deposition and UHV Electron Microscopy, Adrian Garcia, Congjun Wang and Martin E. Kordesch, to be submitted J. Vac. Sci. Tecnol.

Chemical Vapor Deposition of an Ordered Two - Dimensional Carbon Layer on Mo(100) observed With Emission Microscopy, Adrian Garcia and Martin E. Kordesch, to be submitted to Appl. Phys. Lett.

PATENTS:

Patent on cold field emitters applied for, MAR- 92.

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Figures:

Figure 1: The gas doser and PEEM geometry.

Figure 2: A time sequence of activation and progress of the 2-D deposition of carbon. The left half of the image, which is light, is the carbon film. The dark half, on the right is the pre-adsorbed oxygen layer on Mo(100). The edge of each frame is 50um. A 10 um bar is in the Upper Left image, by the "33".

Figure 3: Star shaped 3-dimensional nuclei formed in the area of highest flux from the gas jet. 1 cm = 10 um.

Figure 4: The underside of diamonds grown at 20 Torr with HFCVD on a Mo foil substrate. The star shaped pattern is visible at the center of the crystallites.

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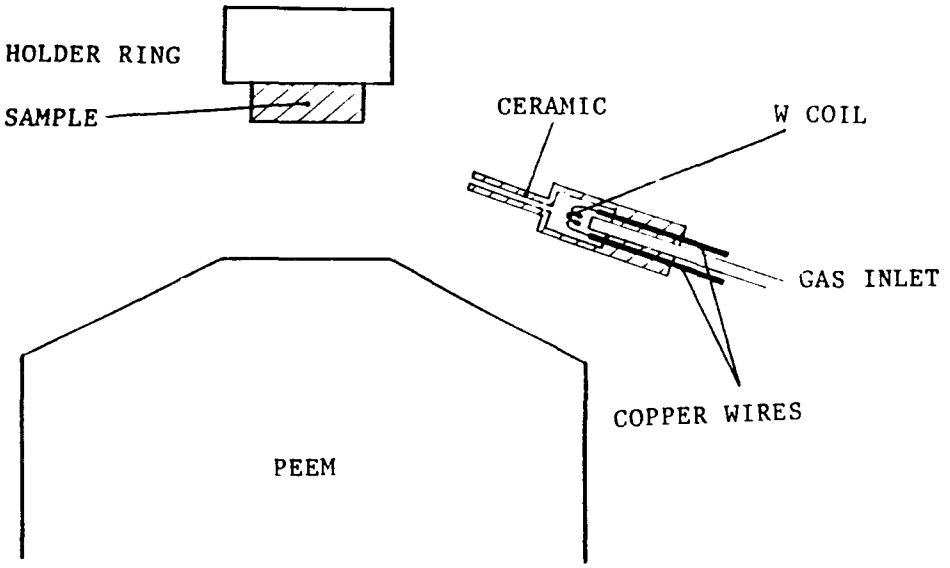
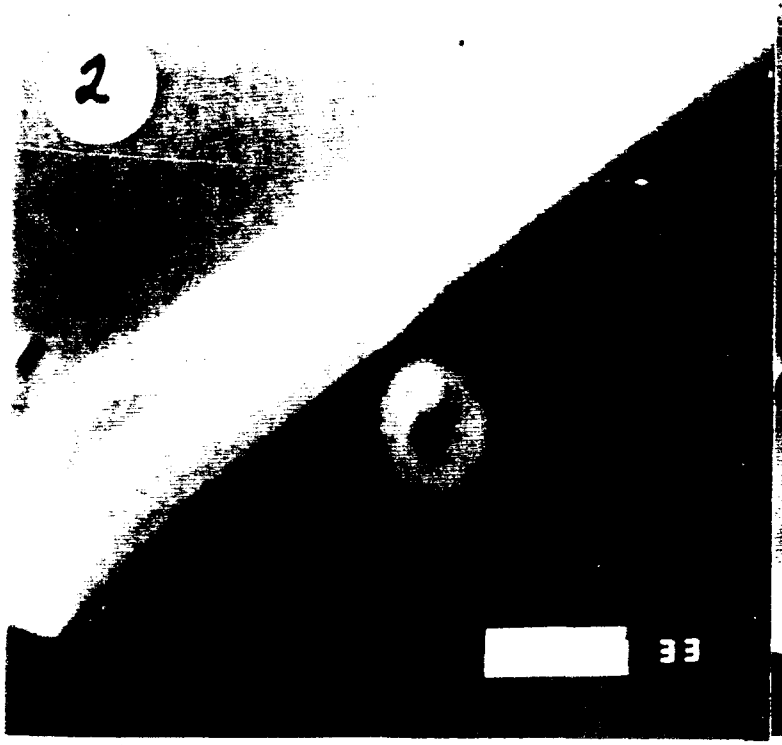



Figure 1: Schematic diagram of the HFCVD dosing apparatus. The sample is about 12 mm in diameter. The distances are approximately to scale. The dosing system condsists of a ceramic jacket with appropriate feedthroughs for the tungsten filament power, and an orifice for the Stainless Steel capillary gas inlet.



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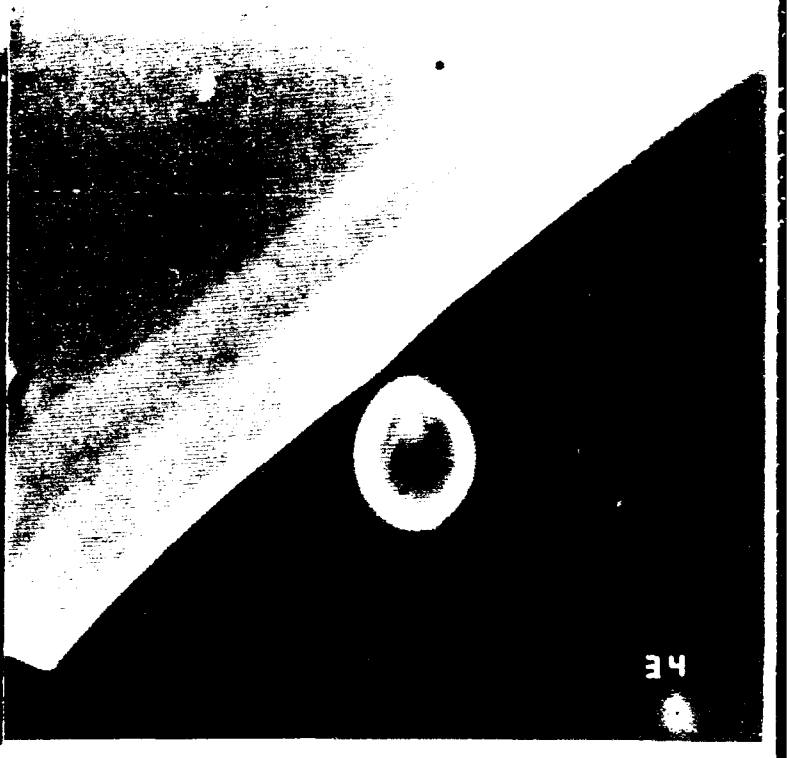

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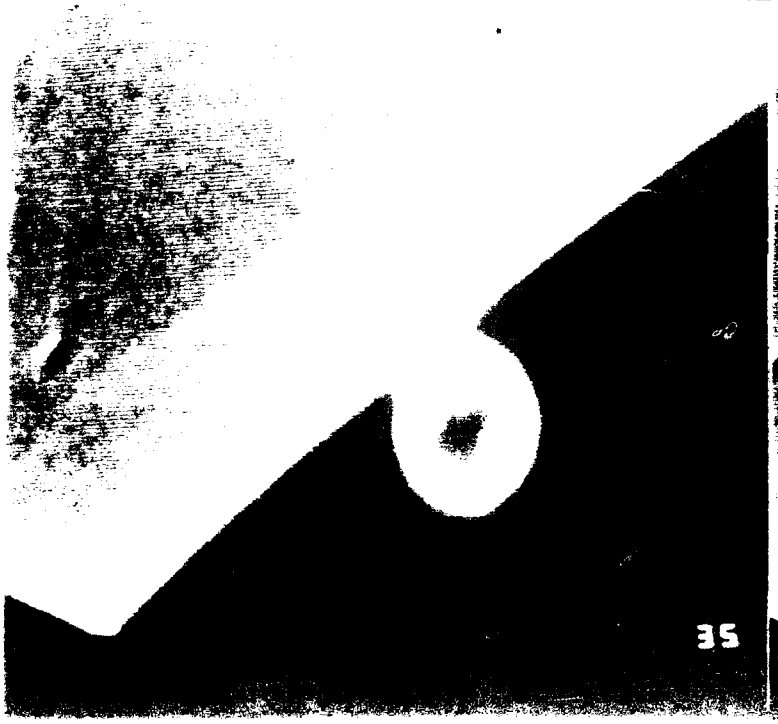

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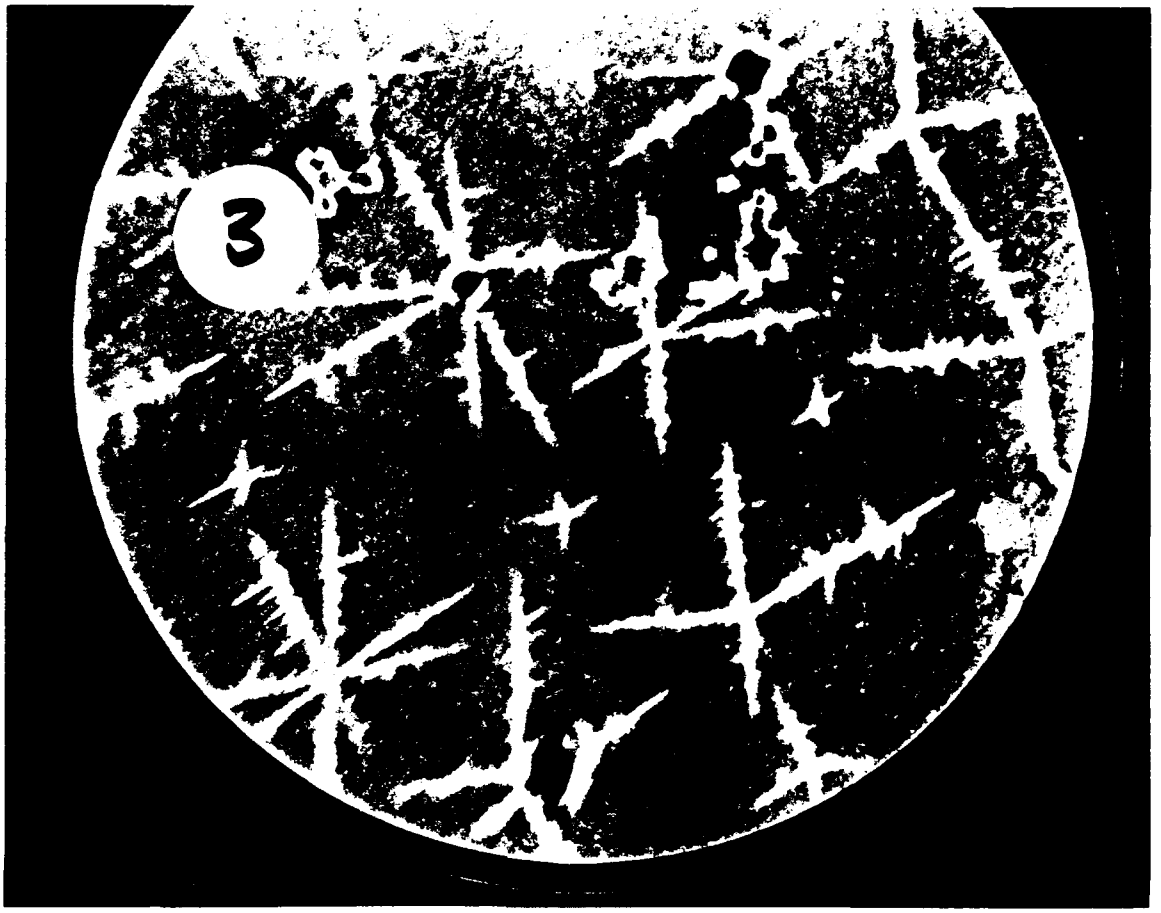


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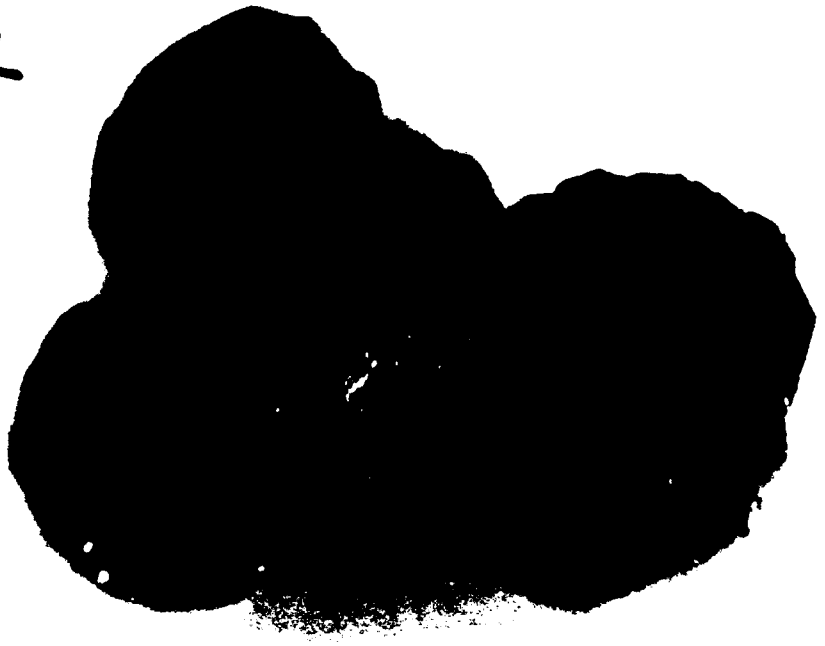

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Abstract Submitted
for the 1992 March Meeting
16-20 March 1992

Suggested Session Title:
Surfaces: Metallic

March Sorting Category:
30e

Chemical Vapor Deposition of an Ordered Carbon Monolayer on Mo(100) by the Propogation of a Two-Dimensional Reaction Wavefront. Adrian GARCIA and Martin E. KORDESCH, Ohio U. -- The reactive deposition of carbon on an oxygen-covered Mo(100) single crystal surface has been observed in situ with photoelectron emission microscopy (PEEM). The Mo(100) surface is heated to 1000 K and covered with a single monolayer of atomic oxygen. LEED and XPS data confirm the O-layer purity and coverage. A stream of 5% methane in hydrogen is directed at the surface over a hot W filament which dissociates the hydrogen. The effective pressure is about $10^{-4}$ Torr. The deposition of carbon on the Mo surface can be observed as a travelling, wave-like reaction front, where oxygen is consumed at the leading edge, and carbon is deposited at the trailing edge. The deposited carbon layer gives a C(2x2) LEED pattern. The wave front extends over several mm, without secondary nucleation of carbon on the oxygen covered surface. Implications for atomic layer epitaxy of diamond and the dynamics of the observed reaction will be discussed.

Submitted by

Martin E. Kordesch
Department of Physics
Ohio University
Athens, Ohio 45701

Prefer Standard Session

A2

# Emission Microscopy of CVD Diamond Film Nucleation

Martin E. Kordesch
Department of Physics and Astronomy, Ohio University
Athens, Ohio 45701-2979 Tel.: (614) 593 - 1703

The growth of device quality diamond films for electronics applications depends on the ability to control the nucleation site and growth habit of the deposited diamond. To date, direct observation of diamond nucleation and growth has not been possible. The most common growth techniques are not adaptable to conventional electron microscopes, due to geometry and temperature restrictions and the lack of contrast in the images that is a result of using high voltage electron illumination. Photoelectron emission microscopy (PEEM) images are dependent on the surface photoyield for contrast, so that the local surface work function determines the image composition. Graphite, diamond and atomic carbon can be distinguished by this technique, and monolayer adsorption of reactants can be viewed in real time, at video tape rates. The photoemission microscopy technique will make the direct observation of diamond growth possible, and is therefore complementary to standard ex-situ microscopy already extensively applied to thick diamond films after growth.

The critical, initial stages of diamond growth from the vapor phase beginning at monolayer thickness may be imaged using PEEM. A differentially pumped, ultra-high vacuum microscope equipped with a miniature "hot filament" diamond CVD system has been used to observe the deposition of carbon films from a methane/atomic hydrogen gas stream directed at a Mo(100) single crystal surface at 1000 K. The growth of strongly textured two-dimensional "disks" centered at impurity nucleation sites has been observed (see fig. 1). The (video taped) complete growth process, beginning with nucleation, growth of the "disks", and subsequent coalescence into a continuous two-dimensional film have been documented, and the evolution of the film texture during cooling to room temperature have been documented (fig. 2).

Polycrystalline films observed in the emission microscope have also been observed to emit electrons without external illumination. Low field cold electron emission from polycrystalline diamond films may be suited to field emitter arrays for high frequency switching devices or electron sources with low chromatic aberration.

Thick (100 um) chemical vapor deposited (CVD) polycrystalline diamond films emit electrons with an intensity sufficient to form an image in the accelerating field of an emission microscope without external excitation (< 3 MV/m). The individual crystallites are on the order of 1-10 um. The combined crystallite diameter and the electric field strength in these "self-emitting" films are far below those typical for cold metal field emitters. The investigation of these cold field emitters is underway, however, it is believed that graphitic inclusions in the film, possibly on the scale of individual bonds, are responsible for a cascade breakdown of the film that causes electrical conduction.

Abstract/accomplishment paragraph/ Kordesch/Ohio University

A3

Thick ( about 100 micrometers) microwave plasma deposited polycrystalline diamond films have been observed to emit electrons with an intensity sufficient to form an image in the accelerating field of an emission microscope without external excitation (< 30 kV/cm). The individual crystallites are on the order of 10 um. The combined crystallite diameter and the electric field strength in these "self-emitting" films are far below those typical for cold filed emitters ( $10^7$ V/cm, 50 -100 nm tip radius). The "self-emitted" images persist indefinitely, and no charging effects are present. Emission currents are estimated to be near 10 mA/sq cm. When these films are illuminated with UV light ( 100 W mercury arc lamp), a photoemission image may also be observed. The photoemission image is primarily of the crystallite tips, superposed upon the "self-emitting" image. The low field, cold emission images are uniform, with no preference for sharp topographical features. A graphitic conduction mechanism, based on cascade breakdown of the insulating diamond film is suspected as a source of this type of electron emission. The films may be useful as field emission arrays.

KORDESCH/OHIO UNIVERSITY

LOW FIELD COLD ELECTRON EMISSION FROM A POLYCRYSTALLINE DIAMOND FILM. < 3 MV/m AT 10 mA/sq cm.

## OBJECTIVES

OBSERVATION OF DIAMOND NUCLEATION ON Mo AND Si

OBSERVATION OF HETERO- AND HOMOEPITAXY OF DIAMOND IN SITU

DETERMINE THE NECESSARY CHEMICAL PARAMETERS FOR EPITAXIAL DIAMOND GROWTH

DETERMINE THE ROLE OF THE SUBSTRATE CRYSTALLINITY AND ORDER ON DIAMOND GROWTH USING PHOTO-ELECTRON EMISSION MICROSCOPY AND DIFFRACTION TECHNIQUES

## PROGRESS

OBSERVATION OF THERMALLY ASSSITED PHOTOEMISSION FROM CARBON FILMS. MATERIAL CONTRAST IN DIAMOND FILMS GROWN WITH ACETYLENE TORCH ULTRAMICROSCOPY **36** (1991) 154-163.

OBSERVATION OF LOW-FIELD COLD ELECTRON EMISSION FROM POLYCRYSTALLINE DIAMOND FILMS ELECTRONICS LETTERS IN PRESS.

IN SITU OBS. AND GROWTH OF 2-DIMENSIONAL CARBON LAYERS AT 1000 K IN PEEM FROM METHANE/HYDROGEN MIXTURE.

CHARATCTERIZATION OF CARBON TETRACHLORIDE ADSORPTION ON SI(100)

CONSTRUCTION OF LEED MICROSCOPE FOR IN SITU DIFFRACTION STUDIES AND OBSERVATION OF DIAMOND RECONSTRUCTION AND HOMOEPITAXY

## APPROACH

LOCALIZATION OF THE NUCLEATION SITES ON THE SURFACE USING AN ADSORPTION-SENSITIVE, IMAGING PROBE (PEEM). ANALYSIS USING XPS, AES, SEM AND RBS.

IN SITU OBSERVATION OF THE SUBSTRATE AND REACTANTS DURING DEPOSITION USING PHOTOELECTRON EMISSION MICROSCOPY

MICROCAPILLARY GAS DOSER WITH HEATERS FOR HYDROGEN DISSOCIATION AND HALOCARBON ACTIVATION INCORPORATED INTO DIFFERENTIALLY PUMPED MICROSCOPE CHAMBER.

OBSERVATION OF DIAMOND GROWTH FROM SEEDS

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# for
# 1 Oct 90 through 30 Sept 91

A5

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Contract/Grant Number: N0001491J1596

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Principal Investigator: Dr. Martin E. Kordesch

Mailing Address: Ohio University
Clippinger Research Labs, Rm 357B
Athens, OH 45701-2979

Phone Number (with Area Code): (614) 593-1703

E-Mail Address:

a. Number of Papers Submitted to Referred Journal but not yet published: 0

b. Number of Papers Published in Referred Journals: 3
(list attached)

c. Number of Books or Chapters Submitted but not yet Published: 0

d. Number of Books or Chapters Published: 0
(list attached)

e. Number of Printed Technical Report & Non-Refereed Papers: 0
(list attached)

f. Number of Patents Filed: 0

g. Number of Patents Granted: 0
(list attached)

h. Number of Invited Presentations at Workshops or Prof. Society Meetings: 0

i. Number of Presentation at Workshop or Prof. Society Meetings: 3

j. Honors/Awards/Prizes for Contract/Grant Employees:
(list attached, this might include Scientific Soc. Awards/Offices, Promotions, Faculty Award/Offices etc.) 0

k. Total number of Graduate Students and Post-Docs Supported at least 25%, this year on this contract,grant:

Grad Students 0 and Post Docs 0

How many of each are females or minorities? (These 6 numbers are for ONR's EEO/Minority Reports; minorities include Blacks, Aleuts Amindians, etc and those of Hispanic or Asian extraction/nationality. This Asians are singled out to facilitate meeting the carrying report semantics re "under-represented")

- Grad Student Female ____
- Grad Student Minority ____
- Grad Student Asian e/n ____
- Post-Doc Female ____
- Post-Doc Minority ____
- Post-Doc Asian e/n ____