

5th International Conference on
Hot-Wire Chemical Vapor Deposition
 HWCVD-5

Massachusetts Institute of Technology
 Cambridge, Massachusetts, U.S.A.
 August 20-24, 2008

Book of Abstracts and Program

Edited by Craig W. Abernethy and Karen K. Gleason
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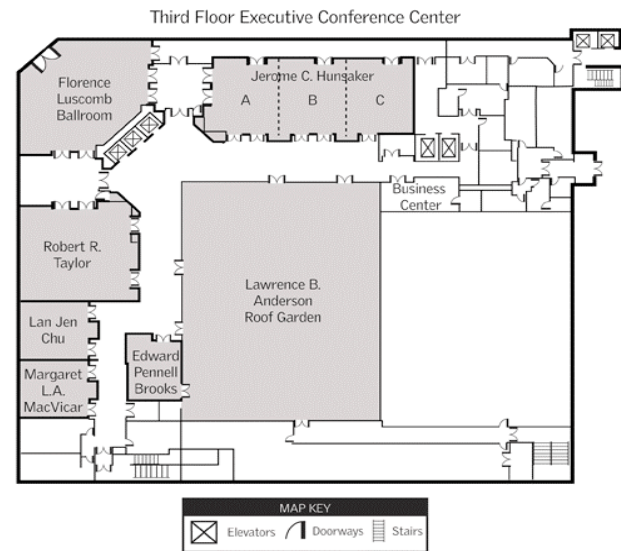
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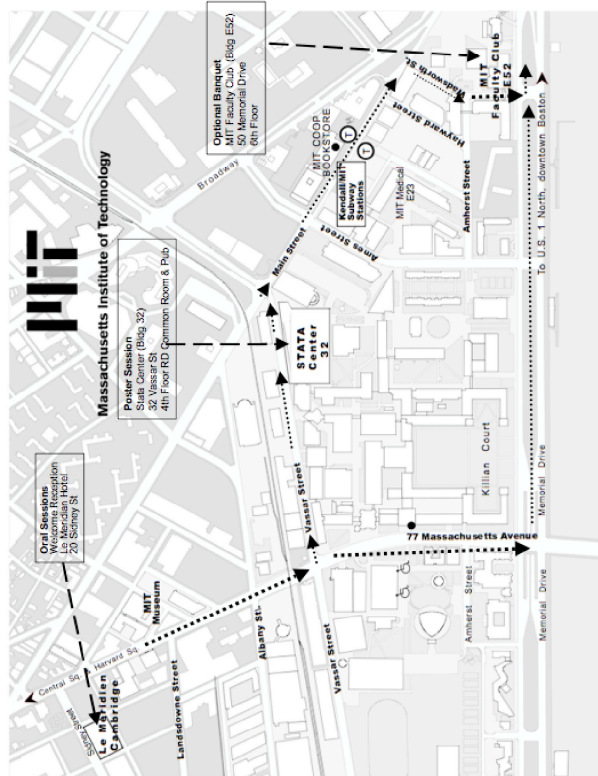
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Map of Hotel Floor Plan



Map of Local Area showing hotel and campus locations



Locations

Tuesday, August 19

A welcome reception for all participants will be held in the Luscomb Ballroom on the third floor of Le Méridien Hotel from 18:30-20:30. Please check in at the registration desk outside of the ballroom to pick up your registration materials and badge.

Wednesday, August 20 to Sunday, August 24

Continental Breakfast is included for all registered participants and will be available outside of the Hunsaker Ballroom from 8:00-9:00.

The break from approximately 12:20 to 13:40 is provided in order to allow you to choose from among the many dining options on your own. Many are within walking distance of the hotel. A list of some local area dining venues is included in your welcome packet. For those wanting to stay close, Sydney's Grille (first floor) is open for lunch and the Star Market next to the hotel has a deli with made-to-order sandwiches.

Thursday, August 21

Tour of GVD – optional, ticket required

A tour of GVD's commercial facility, featuring various types and sizes of hot-wire reactors. Light refreshments will be provided at GVD. If you signed up for the tour, a ticket was provided behind your name badge. The shuttle bus will leave from the hotel entrance at 17:45 and 18:05, returning approximately 90 minutes later. Please refer to your ticket for the tour time. Tickets will be collected at the shuttle.

Friday, August 22

Boston Duck Tour – optional, ticket required

If you registered, a ticket was provided behind your name badge. You must be in the lobby of the hotel entrance at 14:00 to board the duck. The tour returns at approximately 15:20. Tickets will be collected at the duck.

Banquet – optional, ticket required

If you selected the banquet, a ticket was provided behind your name badge. Please bring this ticket with you. The conference banquet will be held at the MIT Faculty Club located on the 6th floor of the Sloan Building (Building E52, 50 Memorial Drive) starting at 19:00 and ending at approximately 22:00.

Saturday, August 23

The poster session and reception - included

The poster session and reception will be held in the R&D Common Area and Pub located on the 4th floor of the MIT Stata Center (Building 32, 32 Vassar St) from 17:30 to 19:30.

General Information

Conference Venue

Le Méridien Cambridge - Hunsaker Ballroom, Third Floor
20 Sidney Street
Cambridge, MA 02139
Phone: 617-577-0200
Toll Free: 800-754-7130

Registration and Information Desk

The registration and information desk will be located on the third floor outside of the Hunsaker Ballroom and will be open during the following hours:

Tuesday, August 19	18:30-20:30
Wednesday, August 20	8:00-15:45
Thursday, August 21	8:00-15:45
Friday, August 22	8:00-12:00
Saturday, August 23	8:00-15:45
Sunday, August 24	8:00-13:00

Badge

Please be sure to wear your badge for all conference sessions and social events.

Session Chairs

Session chairpersons are requested to be present in the lecture room during the break preceding their session. Please meet your authors and make sure that their presentations are properly installed on the available laptop computer. During the session, please make sure that the time schedule is strictly adhered to.

Lecturers

Please bring your presentation on a memory stick and upload it during the break before your session to the laptop PC with PowerPoint, which is available in the lecture room. The chairperson of your session will be there to meet you.

Poster Presenters

Please set up your poster either before the conference dinner on Friday, between 17:15-19:00 or anytime after 7:00 on Saturday. An area of 1.3 meters x 1.3 meters feet is available for each poster. Please affix your poster on the board labeled with your abstract number. Food and refreshments will be provided.

Wireless Internet

Complimentary wireless internet is available in the first floor lobby of the hotel.

Insurance

The organizers cannot be held responsible for injury to conference attendees or for damage to or loss of their personal belongings, regardless of the cause. Attendees are advised to make their own insurance arrangements.

Program**Tuesday August 19**

18:30 Welcome Reception

Wednesday August 20

08:00 Continental Breakfast
 08:50 Opening
 09:00 Introduction and Overview
 10:20 Break
 10:40 Session: Filaments 1
 12:20 Lunch — On your own
 13:40 Session: Filaments 2
 15:20 Break
 15:40 Session: Devices 1
 17:40 End

Thursday August 21

08:00 Continental Breakfast
 09:00 Deposition Mechanism I
 10:20 Break
 10:40 Deposition Mechanism II
 12:20 Lunch — On your own
 13:40 Deposition Mechanism III
 15:20 Break
 15:40 Film Properties I
 17:40 End
 Departure for lab tour (by sign-up)

Friday August 22

08:00 Continental Breakfast
 09:00 Novel Materials I
 10:20 Break
 10:40 Novel Materials II
 12:20 Lunch — On your own
 14:00 Excursion (by sign-up)
 19:00 Banquet (by sign-up)
 22:00 End

Saturday August 23

08:00 Continental Breakfast
 09:00 Applications 1
 10:20 Break
 10:40 Applications 2
 12:20 Lunch — On your own
 13:40 Nanostructures and Patterning
 15:20 Break
 15:40 Processing
 17:30 Poster Session
 19:30 End

Sunday August 24

08:00 Continental Breakfast
 09:00 Commercialization and Wrap Up
 10:20 Break
 12:00 Closing
 12:20 End

Wednesday August 20

Introduction and Overview

Chair: Shuichi Nonomura

Day: **Wednesday August 20** Time: **08:50**

08:50 Opening Remarks: Karen K. Gleason

09:00 – 09:40 Invited Lecture:

New Applications of Cat-CVD Technology and Recent Status of Industrial Implementation**Hideki Matsumura** (h-matsu@jaist.ac.jp)JAIST (Japan Advanced Inst. Sci. & Tech.)
Asahidai, Nomi-shi, Ishikawa-ken 923-1292, Japan

Recent progress in application of Cat-CVD technology is overviewed, along with recent status of industrial implementation of this technology. For instance, highly stable amorphous-silicon thin film transistors are fabricated with sufficiently high deposition rates over 3 nm/s by Cat-CVD technology. A new patterning method of metal lines in flat panel displays is developed by using super-hydrophobic Cat-CVD PTFE films prepared on display substrates. In the method, by dipping such substrates into functional solution containing nano-particles of metals, metal lines are formed automatically by avoiding the patterned super-hydrophobic area. Additionally, such metal lines are converted to low resistivity lines by the exposure of hydrogen atoms generated in Cat-CVD apparatus, through the process of reduction of metal-oxide surrounding metal nano-particles and removing organic binders. Other examples to demonstrate the progress of Cat-CVD technology are also reviewed. The Cat-CVD technology appears industrially implemented in the fabrication of ultra-high frequency devices including lasers, solar cells, and other special devices. Such movements and promising future of this Cat-CVD technology are mentioned briefly in the talk.

Wednesday August 20

09:40-10:20 Invited Lecture:
Frontiers in Hot Wire CVD**R.E.I. Schropp** (R.E.I.Schropp@uu.nl)Utrecht University, Faculty of Science, Department of Physics and Astronomy,
Nanophotonics - Physics of Devices, P.O. Box 80.000, 3508 TA Utrecht, the Netherlands.

The research effort in the field of Hot Wire CVD (also called Catalytic CVD or initiated CVD) has increased considerably over the last 10 years. HWCVD is an elegant low pressure deposition technique to deposit functional films, both inorganic and organic, based on the decomposition of precursor sources at a heated metallic surface. An increasing variety of thin film materials can be obtained with this method, at high deposition rate and with good feedstock utilization. The properties of the deposited films are notably different from those of films made with conventional methods. A number of applications, such as diamond deposition, functional polymer deposition, and passivating silicon nitride deposition, has already found its way to commercial manufacturing.

The technique is attractive in many ways. The most important advantages are:

- The deposition of thin films is plasma-free (i.e. without the risk of a damaging bombardment of energetic ions on the deposited films or of losing functionality of precursor molecules).
- It is an easily scalable method. Scaling to large areas merely requires an increase in catalytic surface along with a proportionally larger supply of source gases.
- Substrates (whether rigid or flexible) can easily be handled as they do not have a role in the decomposition process. Step coverage is excellent, and uniformity can easily be optimized as substrates can be moved during deposition without any difficulty.

As can be deduced from the evolution of the number of publications over the last 20 years (see Fig. 1), the growth rate in the interest in HWCVD has increased from 1999.

The peaks in this graph are due to the biannual international conferences, which have been held since 2000 in the even years. The four different names that are listed relate to the various forms of this technique. The term Hot Filament CVD (HFCVD) is the oldest and normally used in the field of deposition of

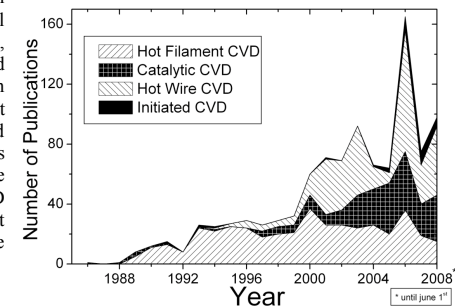


Fig. 1: Number of publications in the field of HWCVD over the last 20 years (courtesy of V. Verlaan).

Wednesday August 20

diamond and other carbon-containing layers. The term i-CVD is the newest and is used to describe polymer deposition using initiator molecules and monomer precursors. HWCVD and Cat-CVD are mutually exchangeable terms for the process of depositing (mainly) inorganic thin films in general.

In this paper we discuss the modern research issues and progress in the realm of Hot Wire CVD (Cat-CVD) and i-CVD as presented at the 5th International Conference, held in Cambridge, MA, USA, in August 2008.

Wednesday August 20

Session: **Filaments 1**

Chair: Yujun Shi

Day: **Wednesday August 20** Time: **10:40**

10:40-11:20 Invited Lecture:

Hot Filament Activation of B₂H₆/CH₄/H₂ Gas Mixtures: Filament seasoning and its effects on gas phase chemistry and composition

M.N.R. Ashfold^a (mike.ashfold@bris.ac.uk), **Dane W. Comerford**^a and **Yuri A. Mankelevich**^b

^a School of Chemistry, University of Bristol, Bristol, U.K., BS8 1TS

^b Nuclear Physics Institute, Moscow State University, 119992 Moscow, Russia

B-doped diamond produced by chemical vapour deposition (CVD) methods is attracting growing interest in electronic device applications and as a bio-sensor. Such material can be produced using B₂H₆/CH₄/H₂ gas mixtures, activated either by a microwave plasma or a hot wire (HW).

This presentation describes systematic studies of HW activation of pure H₂ and of CH₄/H₂, B₂H₆/H₂ and B₂H₆/CH₄/H₂ gas mixtures monitoring (i) the power consumption and dissipation by the HW, and (ii) the gas phase H and, when appropriate, B atom concentrations adjacent to the HW surface by *in situ* laser spectroscopy methods, as functions of process parameters (*i.e.* HW temperature, gas mixing ratio and/or pressure, and time, after introducing a perturbation to any of the above).

Such studies serve to highlight, and allow some unraveling of, the complex inter-relation between the HW and the gas phase. Changes in the gas phase can affect the bulk resistivity (and thus the power consumption) of the HW, and its surface properties, on very different timescales. Surface modification, in turn, can affect the efficiency of gas activation (*e.g.* H₂ dissociation), and thus the gas phase chemistry and composition and, ultimately, the growth rate and properties of material grown by such HW-CVD methods.

Wednesday August 20

11:20 – 11:40

Detection of radical species produced by catalytic decomposition of H₂, O₂ and their mixtures on heated Ir surfaces

Hironobu Umemoto^a (thumemo@ipc.shizuoka.ac.jp), **Hiroki Kusanagi^a**, **Kazuaki Nishimura^b**, **Mitsuru Ushijima^b**

^a Faculty of Engineering, Shizuoka University, Johoku, Naka, Hamamatsu, Shizuoka 432-8561, Japan

^b Tokyo Electron LTD., FPD Division, Akasaka, Minato, Tokyo 107-8481, Japan

Production of H atoms, O atoms and OH radicals was confirmed by laser spectroscopic measurements in the catalytic decomposition of H₂, O₂ and their mixtures on a heated Ir filament. No change in electric resistivity was observed when the filament was kept at 2350 K in the presence of 1 Pa of pure O₂, showing that oxidation is minor. Arrhenius type temperature dependences were observed for the densities of H (O) atoms in pure H₂ (O₂) systems. Both increased with their pressures but showed saturation. The O-atom density could be increased up to $2 \times 10^{12} \text{ cm}^{-3}$. In the H₂/O₂ mixed system, the H-atom density was almost independent of the O₂ partial pressure, although the O-atom and OH-radical densities increased with the O₂ pressure. Such O₂ pressure dependences are completely different from those observed for W. Ir is less poisoned by O₂ compared to W. The sharp increase in the OH density against the O₂ pressure suggests that the production processes of OH and H₂O on catalyzer surfaces are competing. This conclusion is consistent with the result of mass spectrometric measurements that the consumption efficiency of O₂ exceeds 40% when the catalyzer temperature is as low as 1100 K.

Wednesday August 20

11:40 – 12:00

The role of hot filaments in plasma enhanced and hot filaments catalytic chemical vapor deposition of carbon nanotubes and other graphitic carbons

F. Le Normand^a (Francois.Le-Normand@ipcms.u-strasbg.fr), **M. Guláš^{a,b}**, **P. Veis^b**, **S. Farhat^c**, **J.E Bouree^d**

^a Groupe Surfaces and Interfaces, IPCMS, UMR 7504 CNRS, 23, rue du Loess, 67034 Strasbourg Cedex, FRANCE

^b Department of Experimental Physics, Faculty of Mathematics, Physics and Informatics, Comenius University, Mlynská dolina, F2, 84248 Bratislava, SLOVAKIA

^c LIMHP, Université Paris 13, Av. J.B. Clément, 93430 Villetaneuse, FRANCE

^d LPICM, Ecole Polytechnique 91128 Palaiseau, FRANCE

Various catalytic chemical vapor deposition (CCVD) processes are investigated, namely Hot Filament CCVD (producing radicals) and Plasma Enhanced Hot Filament CCVD (where electron impact dissociation occurs) in comparison to thermal CVD for the growth of carbon nanotubes or other carbon films or nanostructures, at low temperature. Gas phase kinetics and surface chemistry are taken into account. The role of tungsten filament temperature (300 K, 1900 K and 2200 K) is discussed. Modeling of the gas phase chemistry was carried out by CHEMKIN[®] collection of software, using more than 200 reactions involving 31 species distributed as 15 neutral species and additional 16 charged species. Surface reactions were taken from model presented by Grujicic *et al.* [1] and extended to employ more hydrocarbon species which are believed to participate in the nanotubes growth [2]. Optical emission spectra were monitored to support the modeling, as well as chemical, structural and morphological characterizations of the carbon deposits. Using our surface reaction model, catalytic growth rate in different conditions can be predicted in agreement with experimental observations. Long tubular structures were not observed in HF CCVD, when electric discharge was turned off [3]. This can be explained by lack of the ions, generated by plasma, which participate in the pre-treatment and activation of the catalytic particles.

[1] M. Grujicic, G. Cao, B. Gersten, *Applied Surface Science* **191** (2002) 223

[2] M. Guláš, C. S. Cojocar, F. Le Normand, S. Farhat, *Plasma Chemistry and Plasma Processing* **28** (2008) 123

[3] S. Lee, S. Choi, K.H. Park, K.W. Chae, J.B. Cho, Y. Ahn, J.Y. Park, K.H. Koh, *Thin Solid Films* **516** (2008) 700.

Wednesday August 20

12:00 – 12:20

**Reversibility of silicidation of Ta filaments in HWCVD
of thin film silicon**

C.H.M. van der Werf¹ (c.h.m.vanderwerf@uu.nl), **H. Li¹**, **V. Verlaan¹**, **C.J. Oliphant^{2,3}**,
R. Bakker¹, **Z.S. Houweling¹**, and **R.E.I. Schropp¹**

¹Utrecht University, Debye Institute for Nanomaterials Science, SID-Physics of Devices,
PO Box 80,000, 3508 TA Utrecht, the Netherlands

²National Centre for Nano-Structured Materials, Council for Scientific and Industrial
Research, PO Box 395, Pretoria, 0001, South Africa

³Department of Physics, University of the Western Cape, Private Bag X17, Bellville
7535, South Africa.

One of the main issues in the use of the hot wire chemical vapor deposition (HWCVD) technique for the fabrication of thin film silicon and its alloys is the reaction of the silicon precursor (usually silane) with the filament metal. In case tantalum filaments are used various types of tantalum silicide can be formed depending on the filament temperature. This silicide formation leads to a decreased filament lifetime, which may lead to higher costs when employed in industrial processes. Under standard deposition conditions for device quality material ($T_{\text{wire}} \approx 1750 \text{ }^\circ\text{C}$) a Ta_5Si_3 (as determined by XRD) shell is formed around the Ta core. After 8 hours of accumulated deposition time this shell has a thickness of around $25 \text{ }\mu\text{m}$. Upon annealing in vacuum at $2100\text{-}2200^\circ\text{C}$ the silicide shell becomes thinner while a Ta layer is reappearing at the surface of the wire. After 4 hours the silicide is completely removed whereas the total diameter of the wire has not significantly changed. The resistance of the filament has also been monitored and after the annealing procedure it completely recovers to that of a fresh wire. This regeneration procedure greatly helps to avoid frequent replacement of the filaments.

Wednesday August 20

Session: **Filaments 2**

Chair: Sanju Gupta

Day: **Wednesday August 20** Time: 13:40

13:40 – 14:20 Invited Lecture:

**Epitaxial growth of gallium nitride films under a resource saving
condition using a Cat-CVD (hot-mesh CVD)**

Kanji Yasui (kyasui@vos.nagaokaut.ac.jp)

Nagaoka University of Technology, Nagaoka 940-2188, Japan

Blue LEDs and laser diodes have already been put to practical use by using GaN and InGaN films grown on sapphire substrates. If the GaN films with quality comparable with those grown on sapphire can be grown on Si, it will become possible to fabricate new optoelectronic devices by combining GaN growth techniques with already established Si device technology. In our study, the heteroepitaxial growth of the GaN films on SiC/Si substrates with AlN buffer layer was investigated using a Cat-CVD (hot-mesh CVD). Using the hot-mesh CVD, which utilizes the decomposition reaction on heated tungsten (W) mesh surface, the GaN film growth was achieved with less ammonia consumption compared with conventional MOCVD. In order to further improve the decomposition efficiency of ammonia, the GaN growth by the hot-mesh CVD using Ru-coated W-mesh was also investigated. When using the Ru-coated mesh, the crystallinity of the GaN films did not degrade until a mesh temperature of 1000°C , while the crystallinity markedly degraded at lower than 1100°C when using the W-mesh. From the PL spectra of GaN films grown using the Ru coated W-mesh, strong near band edge emission without yellow luminescence was observed.

Wednesday August 20

14:20 – 14:40

Silicide formation of Ta- and W-filaments and filament lifetime for different filament temperatures

N. Kniffler (n.kniffler@hs-mannheim.de), **A. Pflueger**, **D. Scheller**,
and **B. Schroeder (1)**

University of Applied Science, Department of Power Systems, Paul-Wittsack-Str. 10,
68163 Mannheim, Germany

(1) Department of Physics, University of Kaiserslautern, 67653 Kaiserslautern, Germany

Using typical conditions for hot wire chemical vapour deposition (HWCVD) of high quality thin silicon films in a UHV deposition chamber, we studied the silicidation of different filaments mainly varying the filament temperatures between 1700 °C and 2100 °C. The experiments were done with constant current, running the filament for 5 to 8 hours and even longer. We investigated the material changes over the hole filament length by Raster Electron microscopy (REM). The thickness of the formed silicide layers and the change of filament resistance depending on the filament temperature was also monitored, pointing out the different behaviour related to wolfram and tantalum filaments. We will show and discuss the changes of filament resistance and filament temperature with time and point to the different behaviour of tantalum and wolfram filaments. As a result, optimum deposition parameter regimes for tantalum and wolfram filaments could be derived with respect to their specific problems concerning the filament lifetime.

Wednesday August 20

14:40 – 15:00

Resistance characteristics to oxidation of the metal-catalyzer for Cat-CVD

Tsugutomo Kudoh (tsugu@ele.kanagawa-it.ac.jp) and **Yoh-Ichiro Ogita**

Kanagawa Institute of Technology

1030 Shimo-Ogino, Atsugi, Kanagawa, 243-0292, Japan

Oxide films are needed to fabricate a high-k gate of MOSFET (metal oxide semiconductor field effect transistor) in ULSI (ultra large scale integration) and coating films to block O₂. One of the authors has formed alumina films on a Silicon crystal from TMA (tri-methyl aluminum) using Iridium and Tungsten catalyzers in Cat-CVD (catalytic chemical vapor deposition) process which is characterised by non-plasma damages and low temperature deposition. However, Iridium is expensive and Tungsten is soft resistance to oxidation. In this study, we examined resistance characteristics to oxidation in various metals such as Advance, Alumel, Al Chrom-O, Chromel, Constantan, Dumet, Hastelloy, Kanthal, Kovar, Inconel-600, Inconel-601, Inconel X-750, Iron Chrom 30, Molecuoy, Molybdenum, Monel, Nickel, Ni Chrome, 42 Invar, Piano Wire, Platinum, SUS-304, SUS-316, Super Invar and Titanium. We measured time variation of resistivity in the metal catalyzers under oxygen atmosphere of 95 Pa and a temperature of catalyzer at 900°C. Moreover, we also measured extent of oxidation by XPS (X-ray photoelectron spectroscopy). We found that Alumel, Nickel, Platinum, Al Chrom-O, Chromel, Hastelloy, Inconel-600, Inconel-601, Inconel X-750, Iron Chrom 30, Kanthal, SUS-304, Molecuoy and SUS-316 are a resistive to oxidation, of which metals are characterised by inclusion of Chromium.

Wednesday August 20

15:00 – 15:20

Effect of ammonia on Ta and W wires during the hot wire CVD process

V. Verlaan (V.Verlaan@uu.nl), **C.H.M. van der Werf**, **C.J. Oliphant**^{1,2}, **R. Bakker**,
Z.S. Houweling and **R.E.I. Schropp**

Utrecht University, Faculty of Science, Debye Institute for NanoMaterials Science,
SID - Physics of Devices

1 National Centre for Nano-Structured Materials, Council for Scientific and Industrial
Research, PO Box 395, Pretoria, 0001, South Africa

2 Department of Physics, University of the Western Cape, Private Bag X17,
Bellville 7535, South Africa

Although NH₃ is extensively used in HWCVD processes, surprisingly little is known about its influence on the filaments. We have studied the effect of NH₃ on heated Ta and W wires. When using pure NH₃, the wire temperature is an important parameter. Below 2000°C, N dissolves in the Ta, however no compositional modifications occur. Due to the fast decline in N-solubility in Ta upon cooling, the N-atoms are rapidly forced out of the wire, causing surface roughening. At higher temperatures, TaN_x formation takes place. The behaviour of W wires differs greatly from Ta wires, no N dissolves into the wire or nitride formation.

When combining NH₃ and SiH₄, no structural changes are observed. After shutting down the gas flows, it takes only 60s for the complete recovery of the resistance to pre-deposition values, which confirms the absence of compositional modifications. On the “cold”-spots of the wires, SiN_x films are observed. Noticeable are five SiN_x films, corresponding to the five depositions the wire was used for. After exposure to an NH₃ and CH₄ atmosphere, a crystalline TaC_{0.275}N_{0.218} layer is formed at the surface. Further heat treatment causes phase separation of the wire in which Ta, Ta₂C and TaC_{0.275}N_{0.218} regions are formed.

Wednesday August 20

Session: **Devices 1**

Chair: Bernd Schroeder

Day: **Wednesday August 20** Time: 15:40

15:40 – 16:20 Invited Lecture:

Trace Detection of Peroxides Using Microcantilevers

John P. Lock (jlock@tritonsys.com), **Edward Geraghty**, **Lawino C. Kagumba**, and
Ken K. Mahmud

Triton Systems, Inc., 200 Turnpike Rd., Chelmsford, MA 01824

Peroxides have proven extremely useful as a free-radical polymerization initiator of organic films deposited via initiated Chemical Vapor Deposition (iCVD). Triton has combined the vapor-phase mechanism of iCVD and microcantilever detector technology to develop a trace sensor of peroxide vapors. Microcantilevers are highly sensitive to the adsorption of molecules onto their surface. The cantilever surface is typically functionalized with coatings that promote adhesion of the target molecule, but low selectivity (high false alarm rates) stemming from the adsorption of interferent molecules remains an inherent problem for many microcantilever detectors. Triton's unique iCVD-based functionalization scheme enables an extremely sensitive and selective self-amplified response to peroxide vapors. The detector response is also reversible, which is critical for deployment in an operational setting. Triton's peroxide sensor addresses a gap in aviation security where new technology is needed for the reliable detection of peroxide explosives and their liquid precursors. The threat of these homemade explosives has resulted in the restriction of liquids and gels on commercial aircraft. Triton also anticipates a growing role for peroxide sensors in a range of industrial applications, and for leak detection as hydrogen peroxide rocket fuel systems are assessed as candidate replacements of extremely toxic hydrazine.

Wednesday August 20

16:20 – 16:40

Development of Flexible Electrochromic Devices Based on Crystalline WO₃ Nanostructures Produced with Hot-Wire Chemical-Vapor-Deposition

C. M. White^{a,b} (Christine.Dolliver@colorado.edu), **D. Gillaspie^b**, **E. Whitney^b**,
S.-H. Lee^a, and **A. C. Dillon^b**

a Department of Mechanical Engineering, University of Colorado at Boulder, Boulder CO
b National Renewable Energy Laboratory, 1617 Cole Boulevard, Golden CO

Nanostructured tungsten oxide (WO₃) films exhibit great potential for a variety of applications, such as energy-efficient electrochromic windows, high energy density batteries, displays, and gas sensors. Crystalline WO₃ nanoparticles synthesized using a hot-wire chemical-vapor-deposition (HWCVD) process show promising results for the advancement of electrochromic applications. Nanoparticle films deposited on SnO₂:F coated glass substrates by a unique electrophoresis technique are more porous than bulk films, resulting in increased surface area and improved ion-insertion kinetics. Compared to state-of-the-art amorphous technologies, the crystalline nanoparticle films were shown to have superior electrochemical-cycling stability (1). We are currently investigating the potential of these durable nanoparticles for flexible electrochromic devices. The development would reduce the costs for current electrochromic windows as electrochromic layers could be added to windows that are already in place. A variety of techniques are being explored for the deposition of WO₃ nanoparticles onto transparent conducting polymers and polymers coated with a conducting layer. Non-conducting polymers are coated with thin conducting oxides including IZO (Indium Zinc Oxide) and ITO (Indium Tin Oxide) with sputtering techniques. Both electrophoresis and spraycoating techniques are employed to deposit the WO₃ nanoparticles on the polymers, and the electrochromic properties of the films are determined. The optimization of a HWCVD generated nanoparticle-based flexible electrochromic device will be discussed.

(1) Lee, S-H.; Deshpande, R.; Parilla, P.A.; Jones, K. M.; To, B.; Mahan, A. H.; Dillon, A.C., *Adv. Mater.* 2006, 18, 763-766.

Wednesday August 20

16:40 – 17:00

Novel Back-Contact Heterojunction Solar Cells Fabricated Using Cat-CVD a-Si and SiN_x films

Koichi Koyama (k-koyama@jaist.ac.jp), **Keisuke Ohdaira**, and **Hideki Matsumura**

JAIST (Japan Advanced Inst. Sci. & Tech.)
Asahidai, Nomi-shi, Ishikawa-ken 923-1292, Japan

It is well known that Heterojunction with Intrinsic Thin layer (HIT) solar cells demonstrate higher efficiency than the conventional crystalline cells by high temperature processes. A back contact type HIT solar cell is expected to have even higher efficiency than the normal HIT solar cell, due to lack of shadowing loss. We proposed a novel structure of back contact HIT cells by using Cat-CVD films. In this structure, zebra electrodes for p-type are covered with insulating SiN_x films. Then a part of SiN_x is opened at the middle of zebra electrodes, and n⁺-a-Si are deposited on it. This process is so simple compared with the conventional back contact solar cell process. All a-Si and SiN_x films were deposited by Cat-CVD. SiN_x films were used for passivation and antireflection on the front surface and interlayer dielectric films on the back surface. We have succeeded to fabricate back contact solar cells. For instance, although it is really first preliminary attempt without intrinsic a-Si layer and optical confinement structure, we could fabricate solar cells with efficiency of 4.33%, open-circuit voltage of 405 mV, and short-circuit current of 27.64 mA/cm².

Wednesday August 20

17:00 – 17:20

Microcrystalline silicon carbide thin films grown by HWCVD at different filament temperature and their application on n-i-p microcrystalline silicon solar cells

Tao Chen^{a,b} (t.chen@fz-juelich.de), **Yuelong Huang^a**, **Arup Dasgupta^a**, **Reinhard Carius^a**, **Friedhelm Finger^a**, and **Deren Yang^b**

^a IEF-5 Photovoltaik, Forschungszentrum Jülich GmbH, 52425 Jülich, Germany

^b State Key Lab of Silicon Materials, Zhejiang University, 312007 Hangzhou, P.R. China

To further optimize the performance of microcrystalline silicon carbide ($\mu\text{-SiC:H}$) window layers in n-i-p type microcrystalline silicon ($\mu\text{-Si:H}$) solar cells, we studied the influence of hot wire chemical vapor deposition (HWCVD) parameters on the properties of $\mu\text{-SiC:H}$ films and corresponding solar cells. We report on a variation of the filament temperature (T_f) of the Rhenium wires in the range $T_f=1800 - 2100$ °C, keeping the substrate temperature at 275 °C. The material was investigated with Raman and Infrared Spectroscopy, optical absorption and electrical conductivity measurements. All $\mu\text{-SiC:H}$ films are strongly n-type as reported earlier. With increasing filament temperature, the crystallinity of these films increases. Both conductivity σ and the transparency according to the energy E_{04} value (where absorption is 10^4 cm^{-1}) show maximum values of $\sigma=2.1 \times 10^{-2} \text{ S/cm}$ and $E_{04}=3.0 \text{ eV}$ respectively for $T_f=1900 - 2000$ °C. The material is used in n-side illuminated n-i-p $\mu\text{-Si:H}$ solar cell on a texture etched ZnO transparent contact with 1.1 μm thick i-layer and silver back reflector. Cell efficiency of more than 8.0% and high short-circuit current density above 23 mA/cm^2 are obtained for the cell with $\mu\text{-SiC:H}$ window layer prepared at $T_f=2000$ °C.

Wednesday August 20

17:20 – 17:40

Comparison of a-Si TFT's fabricated by Cat-CVD and PECVD method

Shogo Nishizaki (s-nishi@jaist.ac.jp), **Keisuke Ohdaira** (Ohdaira@jaist.ac.jp) and **Hideki Matsumura** (h-matsu@jaist.ac.jp)

JAIST (Japan Advanced Inst. Sci. & Tech.)

Asahidai, Nomi-shi, Ishikawa-ken 923-1292, Japan

This demonstrates the characteristics of amorphous silicon thin film transistors (a-Si TFT's) and their stability on bias current stress, fabricated by both plasma enhanced chemical vapor deposition (PECVD) and catalytic (Cat) CVD method. The Cat-CVD a-Si TFT's using an optimized low-defect-density a-Si active layer and a SiN gate insulator have lower off current (I_{off}) under 10^{14} A and higher on current (I_{on}) than PECVD one. In addition, under the bias and current accelerated test, the V_{th} shift and degradation of I_{on} is smaller than PECVD a-Si TFT's. These effects imply that Cat-CVD a-Si TFT's are superior to conventional PECVD a-Si TFT's. The superiority in stability for Cat-CVD TFT's are observed for fabrication temperatures at both 320°C and 180°C, while low temperature fabrication of TFT's is a key for cost reduction by using low cost glass substrate. Thus, the a-Si TFT's fabricated by Cat-CVD method may have the possibility as pixel controlling devices for a next generation flat panel displays.

Thursday August 21

Deposition Mechanism 1

Chair: Hideki Matsumura

Day: **Thursday August 21** Time: **09:00**

09:00 – 09:40 Invited Lecture:

Silicon surface passivation by hot-wire CVD Si thin films studied by in situ surface spectroscopy**J.J.H. Gielis, B. Hoex, P.J. van den Oever, M.C.M. van de Sanden, and W.M.M. Kessels** (w.m.m.kessels@tue.nl)

Department of Applied Physics, Eindhoven University of Technology, The Netherlands

Silicon thin films can provide an excellent surface passivation of crystalline silicon (*c*-Si) which is of importance for high efficiency heterojunction solar cells or diffused emitter solar cells with well-passivated rear surfaces. Hot-wire chemical vapor deposition (hotwire CVD) is an attractive method to synthesize Si thin films for these applications as the method is ion-bombardment free yielding good quality films over a wide range of deposition rates. The properties of the interface between Si thin films and H-terminated *c*-Si substrates have been studied during film growth by three complementary in situ techniques. Spectroscopic ellipsometry has been used to determine the optical properties, film thickness and surface roughness whereas information on the H-bonding modes and H-depth profile has been obtained by attenuated total reflection infrared spectroscopy. Second-harmonic generation (SHG), a nonlinear optical technique sensitive to surface and interface states, has been used to probe two-photon resonances related to modified Si-Si bonds at the interface. The observations have been correlated with ex situ lifetime spectroscopy experiments. On the basis of the results, the growth and surface passivation mechanism of the films will be discussed, including the role of defect states, built-in electric fields, (nanometer-level) epitaxial growth, influence of the substrate temperature, etc.

Thursday August 21

09:40 – 10:00

Structural properties of hydrogenated microcrystalline silicon ($\mu\text{c-Si:H}$) deposited by the hot-wire chemical vapor deposition technique at low substrate temperature**S. Halindintwali** (shalindintwali@uwc.ac.za), **D. Knoesen, T. Muller, A. Malape, B. Julies, M. Moodley¹, C. Arendse¹ and R. Bucher²**

Physics Department, University of the Western Cape, Private bag X17, Bellville 7535, South Africa

¹ Nanoscience Research Centre, Council for Scientific and Industrial Research, P.O. Box 395, Pretoria 0001, South Africa² Materials Research Group, iThemba LABS, P.O. Box 722, Somerset West 7129, South Africa

A series of samples have been deposited at substrate temperature below 200°C by the Hot-Wire Chemical Vapor Deposition (HWCVD) technique. The temperature of the substrate heater was increased from room temperature (RT) to 200°C and the hydrogen dilution ratio in the silane gas was maintained at 98% while other deposition parameters were kept fixed. This contribution will report on the structural properties of the films deposited. They have been characterized for microcrystallinity by Raman scattering spectroscopy and X-ray diffraction. An average crystalline volume fraction is estimated around 40% with a crystal size ranging between 13.7 and 26 nm. The photoluminescence studies reveal a peak situated at around 1.42 eV in case of high energy excitation and another one at between 1 - 1.2 eV for low energy excitation suggesting that the films are indeed crystalline with a residual network of amorphous silicon. Another additional peak, likely due to the Si-O effects, has been observed at around 1.6 eV. The hydrogen content was estimated by Fourier Transform Infrared spectroscopy at around between 2.9 and 3 at.% in the samples. Additional structural details will be given by the micrographs obtained by our newly acquired High Resolution Cross Section Transmission microscope.

Thursday August 21

10:00 – 10:20

Hot wire CVD deposition of nanocrystalline silicon on rough substrates

Hongbo Li (h.li@uu.nl), **C. H. M. van der Werf**, **Jatin K. Rath**, **Ruud E. I. Schropp**

Utrecht University, Debye Institute for Nanomaterials Science, SID - Physics of Devices, P.O. Box 80.000 3508 TA Utrecht, the Netherlands

In silicon thin film solar cell technology, frequently rough or textured substrates are used to scatter the light and enhance its absorption. To further promote the use of HWCVD for the deposition of efficient nc-Si:H thin film solar cells, the important issue of the influence of substrate roughness on silicon nanocrystal growth has been investigated. It is shown that silicon grown on the surface of an unoptimized rough substrate contains densely distributed structural defects, which deteriorate solar cell performance. The defect development in $\mu\text{c-Si:H}$ cells may be caused by hydrogen etching of strained Si-Si bonds formed within the valleys at the surface onto which the film is grown. This finding has helped us to set a criterion to the morphology of light trapping substrates for silicon thin film solar cells. The formation of structural defects in the nc-Si:H absorber layer is avoided if the substrate surface local opening angle is larger than a certain value.

Thursday August 21

Deposition Mechanism 2

Chair: Saibal Mitra

Day: **Thursday August 21** Time: **10:40**

10:40 – 11:20 Invited Lecture:

Involvement of Silylene and Silene Intermediates in the Hot-wire Chemical Vapor Deposition (HWCVD) Process with Silacyclobutanes as Source Gases

Yujun Shi (shiy@ucalgary.ca) and **Ling Tong**

Department of Chemistry, University of Calgary, Calgary, Alberta, Canada T2N 1N4

Silacyclobutane (SCB) molecules, with significant ring strain energy in their four-member ring structures, are expected to be more susceptible to hot filament activation. In view of this, the gas-phase reaction products of three different SCB molecules in a HWCVD reactor were diagnosed in-situ using vacuum ultraviolet laser ionization/time-of-flight mass spectrometry. Both Si- (silylenes and silenes) and C-containing (ethene and propene) species were found to be produced in-situ from the SCB decomposition on W filament. It is demonstrated that the formation of ethene is favored over that of propene for monosilacyclobutane(mono-SCB) molecules. With both Si-H bonds replaced by Si-CH₃ connections in mono-SCB, more ethene species were produced. The highly reactive silylene and silene intermediates undergo insertion reactions into the Si-H bonds of the parent molecule, Π -type addition reactions across double or triple CC bonds, or cycloaddition reactions, forming a variety of stable products that were detected by the mass spectrometer. Among all the Si-containing reactive intermediates produced with SCB as source gases, silene (H₂Si=CH₂) formed from decomposition of parent SCB molecule (SiC₃H₈) is a potentially useful precursor for silicon carbide film formation due to its built-in 1:1 Si:C ratio and an existing Si-C bond.

Thursday August 21

11:20 – 11:40

Effects of growth temperature and gas-phase diffusion on carbon-nanotube growth in Hot-filament CVD: two-stage growth model

**Nguyen Tuan Hong, Kim Sang Yong, Bun Hee Jun, Ken Ha Koh,
and Soonil Lee** (soonil@ajou.ac.kr)

Division of Energy Systems Research, Ajou University, Suwon 443-749, Korea

We investigated the growth kinetics of a vertically-aligned dense carbon-nanotube (CNT) forest in hot-filament CVD (HFCVD) processes. A two-stage growth model that took the temperature and gas-phase-diffusion effects into consideration was developed to account for the experimentally observed CNT-forest growth behavior. In the first stage, in which direct radiation from the heated tungsten filament resulted in monotonic increase of substrate temperature, the CNT-forest growth showed exponential temporal dependence. It seemed that the substrate temperature was more important, compared to the gas-phase diffusion, in this stage. In a typical example, a CNT forest grew as tall as ~240 μm within 3 minutes. In the second stage, after substrate-temperature's becoming stable, the height of CNT forest was observed to increase linearly with time. When the growth continues for 20 minutes, the CNT forest grew up to about 2 mm. However, it was observed that the growth-rate decreased, and the growth of CNT-forest became almost completely terminated after about 25 minutes.

Thursday August 21

11:40 – 12:00

Preparation of CNW films by hot-wire CVD with DC substrate bias

T. Itoh¹ (itoh@gifu-u.ac.jp), **S. Shimabukuro²**, **Y. Hatakeyama¹**, **K. Tada¹**,
Y. Iwatake¹ and **S. Nonomura²**

¹ Department of Electrical Engineering, Gifu University

² Environmental and Renewable Energy Systems Division, Gifu University,
1-1 Yanagido, Gifu 501-1193, Japan

Carbon nanowall (CNW), which has wall structures stood on a substrate, can be prepared by hot-wire chemical vapor deposition (HWCVD) [1]. In the preparation of diamond films by hot-wire CVD, DC substrate bias enhances the nucleation [2]. In the growth of CNW, however, the effect of the DC substrate bias has been unknown. In this work, therefore, the effect of the DC substrate bias in the growth of the CNW has been studied. Samples have been prepared by hot-wire CVD with the DC substrate bias V_S from -150 to 150 V. As $V_S > 0$, the I_G/I_D increased with increasing V_S . As $V_S < 0$, however, the I_G/I_D was almost constant. Here, I_G and I_D are the intensity of G and D peaks in Raman spectra, respectively. This result suggests that the positive V_S enhances the growth of CNW. In the preparation by HWCVD with $V_S > 0$, current between filament and substrate was observed. This current would be due to thermoelectron. The effect of V_S in the growth of CNW will be discussed with other results.

[1] T. Itoh, et al., Thin Solid Films, 501 (2006) 314.

[2] X. Li, et al., Thin Solid Films, 308-309 (1997) 163.

Thursday August 21

12:00 – 12:20

HWCVD of Polymer Film Using Supersonic Precursor Jet

A.K.Rebrov (rebrov@itp.nsc.ru), A.I. Safonov, P.A.Skovorodko, N.I. Timoshenko

Institute of Thermophysics of the Siberian branch of the Russian Academy of Sciences, Novosibirsk, Russia

The comprehensive study of the HWCVD polymer deposition was performed by the group of K. Gleason [1]. New possibilities are arising in the case of deposition from supersonic precursor jets. The properties of deposited films depend on the state of precursor molecules colliding with the substrate surface, that is chemical composition, translational and internal energy of molecules. In the case of the nonequilibrium supersonic precursor flow there is a possibility to control the internal energy as far as translational one.

We have studied the influence of the net wire temperature on properties of the teflon-like film using C_2F_4 as the precursor gas. The tetrafluoroethylene was generated from the bulk Teflon in the reactor with stagnation temperature about 800K and pressure on the level a few torrs. In distinct to usual HWCVD we have situation, when temperature of the wires is usually lower than stagnation temperature: the precursor translational energy is decreased, but vibrational one is elevated.

In experiments the supersonic jet was formed behind the sonic nozzle with the diameter 10mm; the substrate was located on the distance 25mm from the nozzle, wire net was installed in 15mm ahead of the substrate. The change of wire temperatures from recovery ones (without wire heating) up to 850K resulted in the deposition of teflon-like films with essentially different morphology (crystalline, fractal, nanosize particle forms). The difference of films by deposition from supersonic and subsonic jets for the same stagnation temperature was discovered as well. The computational modeling shows, that the impulse and heat on the supersonic precursor flow would be governing factor for flow parameters behind the net. It can be either subsonic or supersonic.

Thus the wire activation of nonequilibrium supersonic and subsonic flow enriches HWCVD methods by specific variation of precursor gas state before deposition.

1. Kenneth K. S. Lau, Jeffrey A. Caulfield and Karen K. Gleason. Structure and Morphology of Fluorocarbon Films Grown by Hot Filament Chemical Vapor Deposition. *Chem. Mater.* 2000, 12, 3032-3037

This work is supported by grants RFBR 06-01-00292-a and 08-08-00344-a.

Thursday August 21

Deposition Mechanism 3

Chair: Guohua Chen

Day: Thursday August 21 Time: 13:40

13:40 - 14:20 Invited Lecture:

Design and Synthesis of Polymers via HWCVD for Biotherapeutics and Beyond

Ranjita K. Bose and Kenneth K. S. Lau (klau@drexel.edu)

Department of Chemical and Biological Engineering, Drexel University
3141 Chestnut Street, Philadelphia, PA 19104

Initiated chemical vapor deposition (iCVD) is a low temperature hot-wire chemical vapor deposition (HWCVD) process for making polymer thin films. iCVD utilizes a well defined, thermally activated pathway through the gas phase activation of an initiator to induce surface polymerization on a cooled substrate. As a result, the polymers are clean and stoichiometric, analogous to polymers formed from liquid phase synthesis. This is expected since iCVD directly makes use of liquid polymerization chemistries, only without the use of a solvent medium. We have applied the iCVD paradigm to create hydrogel polymers for the purpose of making hydrogel scaffolds for tissue regeneration. Specifically, iCVD has been used to design and synthesize hydrogel polymers based on poly(2-hydroxyethyl methacrylate) (PHEMA) and poly(ethylene glycol) (PEG). We will show spectroscopic and microscopic data to determine polymer chemistry, morphology, and structural topography. We will present data on the *in vitro* compatibility with living cells and biodegradation of these materials. We will offer insights on the prospects of extending these materials beyond biotherapeutics.

Thursday August 21

14:20 – 14:40

Heat transfer model of an iCVD reactor

R. Bakker (R.Bakker@uu.nl), **V.Verlaan**, **A.D. Verkerk**, **C.H.M. van der Werf**, **J.K. Rath** and **R.E.I. Schropp**

Utrecht University, Faculty of Science, Debye Institute for Nanomaterials Science, SID – Physics of Devices, P.O. Box 80.000, 3508 TA Utrecht, the Netherlands

Initiated chemical vapour deposition (iCVD) is a novel method for depositing polymers from the gas phase by free radical polymerization. The most important difference between iCVD and conventional hot-wire (HW)CVD is the wire temperature. While the typical wire temperature for HWCVD is about 1750-2200°C, for iCVD the temperature is only 250-500°C. Since the irradiated power is proportional to T^4 , the *relative* contribution of the conduction by the source gases to the overall heat transfer is increased in the iCVD process compared to conventional HWCVD. To study this mode of heat transfer, we developed a model and compared it to measurements of the electrical power dissipation and the temperature of the heated filaments.

Our results show that, contrary to conventional HWCVD, the power consumption in the iCVD process is dominated by conduction rather than radiation. Typical pressures are in the transition regime between the collision free (pressure dependent) and the collision mediated (pressure independent) regimes. The heat conductivities of molecular nitrogen, glycidyl methacrylate (GMA) and tert-butylperoxide (TBPO) gases have been determined experimentally for all pressure regimes. The required power input to the filaments can be explained mainly by considering dissipation by radiation and gas conduction, leaving only little power consumption for the dissociation process.

Thursday August 21

14:40 – 15:00

Initiated Chemical Vapor Deposition of Polymer Films on Nonplanar Substrates

Salmaan H. Baxamusa (baxamusa@mit.edu), **Karen K. Gleason**

Massachusetts Institute of Technology
Department of Chemical Engineering
77 Massachusetts Avenue
Cambridge, MA. 02139

The preparation of continuous and conformal thin polymer films can be challenging when utilizing solution or plasma chemistry. The lack of surface tension (from solvents) or directional effects (from plasma) makes initiated chemical vapor deposition (iCVD) an ideal candidate for applications in which a continuous and conformal thin polymer film is required. The sticking coefficient of the reactive species in iCVD is measured by observing the deposition profiles in a model system of microtrenches. By altering the process conditions, the sticking coefficient of reactive species in the iCVD process can be varied from 1.1×10^{-2} – 5.0×10^{-2} .

Understanding of the process that governs the film profiles allows for the preparation of thin polymer films on particle substrates. The deposition of the hydrogel poly(hydroxyethylmethacrylate) (pHEMA) homopolymer and copolymers on particle substrates will be shown. These polymers have favorable biocompatibility properties and their application on particle biosensors allows for enhanced resistance to nonspecific protein adhesion. X-ray photoelectron spectroscopy confirms that the hydrogel copolymers reduce protein nonspecific adhesion by 90%.

Thursday August 21

15:00 – 15:20

**Mechanism of hot-wire chemical vapor deposition
epitaxial silicon growth at 600 to 800°C**

Ina T. Martin (Ina_Martin@nrel.gov), **Charles W. Teplin**, **Kim M. Jones**,
Manuel J. Romero, **Bobby To**, **Howard M. Branz**, and **Paul Stradins**

National Center for Photovoltaics, National Renewable Energy Laboratory,
Golden, CO 80401

Hot-wire epitaxial thickening of thin, high-quality crystalline seed layers on inexpensive substrates is a promising route to next-generation film silicon solar cell technology. For high photovoltaic efficiency and low cost, the epitaxial growth step must satisfy the following conditions: 1) high rate (> 200 nm/min); 2) high quality; 3) low growth temperature compatible with substrates such as borosilicate glass ($< 700^\circ\text{C}$); 4) high silane gas utilization and 5) industrial scalability. We describe a HWCVD epitaxial growth regime above about 620°C that satisfies all these conditions and discuss why epitaxy below 600°C has not been equally successful. We report the epitaxial deposition rate on (100) silicon wafers for a wide range of growth conditions and present a model of the gas-filament interaction consistent with the observed dependence on gas pressure and flow rate. The model predicts the maximum epitaxial growth rate obtainable in a typical HWCVD reactor as well as the silane utilization fraction. Comparison with experimental data allows estimation of the radical sticking coefficient and the silane decomposition probability at the filament. We also report and discuss characteristic epitaxial HWCVD growth morphologies. Initially, oriented islands coalesce to form a smooth film. Spontaneous pyramidal texturing can develop after the film thickens.

Thursday August 21

Film Properties 1

Chair: Chair: Se-hee Lee
Day: **Thursday August 21** Time: **15:40**

15:40 – 16:20 Invited Lecture:

**Microcrystalline silicon carbide alloys prepared with HWCVD as highly
transparent and conductive window layer for thin film solar cells**

F. Finger¹ (f.finger@fz-juelich.de), **O. Astakhov**¹, **T. Bronger**¹, **R. Carius**¹, **T. Chen**¹,
A. Dasgupta¹, **A. Gordijn**¹, **L. Houben**², **Y. Huang**¹, **S. Klein**¹, **M. Luysberg**²,
H. Wang¹, **L. Xiao**¹

1 IEF-5 Photovoltaik, Forschungszentrum Jülich GmbH, 52425 Jülich, Germany
2 IFF, Forschungszentrum Jülich GmbH, 52425 Jülich, Germany

Crystalline silicon carbon alloys have a very high potential as transparent conductive window layer in thin-film solar cells provided they could be prepared in thin-film form and at compatible deposition temperatures. The low-temperature deposition of such material in microcrystalline form ($\mu\text{-Si:C:H}$) was realized by use of monomethyl-silane precursor gas diluted in hydrogen with the Hot-Wire Chemical Vapor Deposition process. We have investigated a wide range of deposition parameters and have studied the structural, electronic and optical properties of the $\mu\text{-Si:C:H}$ thin films. The material, which is strongly n-type, has been used as window layer in n-side illuminated n-i-p microcrystalline silicon solar cells. We obtain exceptionally high short circuit current densities due to the high transparency of the material with solar cell conversion efficiencies of well above 9% for this type of device. We will report on the details of the material and device development.

Thursday August 21

16:20 – 16:40

**Chemical Vapor Deposition of Low-Dielectric
Constant Organosilicon-based Thin Films**

Narine Malkhasyan (malkhasyan.n@neu.edu), **Daniel Burkey**

Department of Chemical Engineering, Northeastern University

Lowering the dielectric constant, k , of interconnect films in semiconductor devices aids in reduction of signal propagation delay, cross-talk noise, etc. Among chemical vapor deposited (CVD) low- k films, the main focus is on organosilicon-based films, the low dielectric constant (about 2.7-3.0) of which is associated with the low polarizability and high porosity of the materials. This research is aimed at attaining porous organosilicon films using precursors with built-in molecular-size porosity, such as 1,3,5-trivinyl-1,3,5-trimethylcyclotrisiloxane (V_3D_3), and employing secondary linear spacer precursors to extend film porosity even further. Hot filament (HF) CVD is employed to selectively activate the more reactive vinyl side-chain chemistry of V_3D_3 while retaining the methyl groups which help lower polarizability as well as the siloxane ring structure which creates intrinsic porosity. Use of *tert*-butyl peroxide as an initiator allows polymerization at low temperature, further preserving the siloxane ring structure. Linear molecules of different lengths with end-capping vinyl groups (divinyldimethylsilane, 1,3-divinyltetramethyldisiloxane, and 1,5-divinylhexamethyltrisiloxane) are used as spacers between V_3D_3 moieties in the films in order to tailor film porosity. Film refractive indices decrease proportionally with increasing length of spacer precursors, indicating control of film structure and functionality through manipulation of the vinyl chemistry.

Thursday August 21

16:40 – 17:00

**Deposition of Silicon Nitride Thin Films by Hot-Wire CVD
at 100°C and 250°C**

L.M. Goncalves¹, **E.S. Marins**², **P. Alpuim**² (palpuim@fisica.uminho.pt),
T.M.R. Viseu³, **J.E. Bourée**⁴

¹ Departamento de Electrónica Industrial, Universidade do Minho 4800-058 Guimarães, Portugal

² Departamento de Física, Universidade do Minho, 4800-058 Guimarães, Portugal

³ Departamento de Física, Universidade do Minho, 4710-057 Braga, Portugal

⁴ Laboratoire de Physique des Interfaces et des Couches Minces, CNRS UMR 7647, Ecole Polytechnique, 91128 Palaiseau, France

Silicon nitride thin films for use as gate dielectric in thin-film transistors or as passivation layers in organic electronics were deposited by the Hot-wire chemical vapor technique at a high deposition rate (1-3 Å/s) and low substrate temperature, as an alternative to the conventional high-temperature deposition techniques. Using an S-shaped Ta filament and a reactive gaseous mixture of silane and ammonia, films were deposited at NH_3/SiH_4 flow rate ratios between 1 and 70 and substrate temperatures of 100°C and 250°C. The structural properties of the films were studied by FT-IR and UV-VIS spectroscopy. For flow rate ratios between 40 and 70, dense films with good dielectric properties and refractive index between 1.93 and 2.08 were obtained. Metal-insulator-metal test structures, with interface areas between $100^{\circ} \times 100 \mu m^2$ and $400^{\circ} \times 400 \mu m^2$, were fabricated. Electrical conductivity between $1.2 \times 10^{-14} \Omega^{-1} cm^{-1}$ and $2.2 \times 10^{-11} \Omega^{-1} cm^{-1}$ and breakdown fields $>1 MV cm^{-1}$ were measured in films deposited at both substrate temperatures. Etch rates in BHF <0.5 and <3.5 nm/min were obtained for films deposited at 250°C and 100°C, respectively. High-frequency and quasi-static $C-V$ measurements will be performed and metal-dielectric interface defect density will be accessed. Temperature-dependent electrical conduction mechanisms in the dielectric films will be studied.

Thursday August 21

17:00 – 17:20

Low Temperature Deposition of Silicon with Crystallinity Improved by HCl Addition during Hot Wire Chemical Vapor Deposition

Yung-Bin Chung, Dong-Kwon Lee, Chan-Soo Kim and Nong-Moon Hwang
(nmhwang@snu.ac.kr)

National Research Laboratory of Charged Nanoparticles,
Department of Materials Science & Engineering, Seoul National University,
Sillim-dong, Gwanak-gu, Seoul 151-744, South Korea

Hot wire chemical vapor deposition (HWCVD) has been developed as a cost-effective way to deposit crystalline silicon directly on a glass. However, the silicon films deposited on glass at temperature less than 400°C by HWCVD has relatively poor crystallinity, containing an appreciable amount of amorphous silicon. According to the two-step growth mechanism, the crystalline silicon nanoparticles, which are formed in the high temperature region near the hot wire, contribute to crystalline silicon deposition whereas the silicon precipitation in the low temperature region near the substrate contributes to amorphous silicon. Therefore, the formation of amorphous silicon can be minimized by suppressing the low temperature precipitation of silicon. In an effort to suppress the low temperature precipitation of silicon, we added HCl during HWCVD. The silicon films were deposited on a Corning 1739 glass at 320°C under a reactor pressure of 10 Torr at the wire temperature of 1600°C. SiH₄ gas was fixed at 10 standard cubic centimeters per minute (sccm) whereas HCl was varied at an interval of 2 sccm from 0 to 28 sccm. Raman spectra showed the systematic increase in the crystallinity of silicon with increasing HCl, approaching the Raman spectra of a single crystal wafer.

Thursday August 21

17:20 – 17:40

Multifunctional nanocarbons for field emission displays: PECVD syntheses and characterization

S. Gupta (guptas@missouri.edu)

ECE Department, University of Missouri-Columbia, MO 65211

Unprecedented worldwide activity in the investigation of nanostructured carbons was initiated by the discovery of the C₆₀ molecule and the development of the arc-discharge technique producing their elongated members i.e. nanotubes followed by nanocrystalline diamond synthesized by varying the traditional gas phase chemistry used to deposit traditional polycrystalline diamond. The present work will talk about the recent activities relating plasma-enhanced chemical vapor deposited nanotubes and nanodiamond wherein materials science played a vital role [Gupta et. al. APL **86** (2005)]. Electron field emitting materials known as *cold cathodes* are of vital importance enabling myriads of vacuum microelectronics applications (*flat panel displays*, RF/MW amplifiers for communication and radar, bright electron beam for microscopes, electric propulsion for microsatellites and portable X-ray sources for medical diagnostics). Nanodiamond and carbon nanotubes proved to be the potential candidate materials as *planar cold cathodes* having nanoscale heterogeneities for vacuum field emission displays. The traditional field emission properties and temperature dependent field electron emission microscopy (*T-FEEM*) enabling real-time imaging of electron emission with high spatial resolution providing evident information on emission site density, temporal variation or flicker of the emission intensity and insight into the *role of adsorbates* from nanotube films will be discussed. The latter results will also be related to vacuum thermionic energy converters.

Friday August 22

Novel Materials 1

Chair: João Pedro Alpuim

Day: **Friday August 22** Time: **09:00**

09:00 – 09:40 Invited Lecture:

Metal Oxide Nanoparticles for Advanced Energy Applications**S.-H. Lee^a** (sehee.lee@colorado.edu), **A. Dillon^b**, **Y. Kim^b**, **D. Benhammou^a**, **S. Lee^b**,
E. Whitney^b, **D. Gillaspie^b**, **P. Parilla^b** and **A. Mahan^b**^a Department of Mechanical Engineering, University of Colorado at Boulder, Boulder CO^b National Renewable Energy Laboratory, 1617 Cole Boulevard, Golden CO

Hot-wire chemical vapor deposition (HWCVD) has been employed as an economically scalable method for the deposition of crystalline metal oxide nanoparticles at high density. Under optimal synthesis conditions, only crystalline nanostructures with a smallest dimension of ~ 10 - 50 nm are observed with extensive transmission electron microscopy analyses. The incorporation of these particles into porous films led to profound advancement in state-of-the-art electrochromic technologies. HWCVD has also been employed to produce crystalline molybdenum oxide nanostructures at high density. The incorporation of crystalline molybdenum oxide nanoparticles into battery electrodes has led to profound advancements in state-of-the-art negative electrodes (anodes) in lithiumion batteries. The nanoparticle materials exhibit a high rate capability as anticipated for the reduced solid-state Li-ion diffusion length. Density functional theory calculations elucidate the complex Li-ion insertion process and reveal a novel mechanism confirming the nanoscale, high-rate, reversible capacity. The MoO₃ nanoparticles have also been examined as a positive electrode (cathode) material and exhibit a capacity of ~ 120 mAh/g without any degradation for 1500 cycles at C rate. Unfortunately, the cathode operating potential is ~ 2.3 V vs. Li/Li⁺. Using theory as a guide, experimental efforts are underway to make doped MoO₃ nanoparticle cathodes with an increased potential.

Friday August 22

09:40 – 10:00

Hot-Wire Chemical Vapor Deposition of Silicon Nanoparticles on Quartz**N. Salivati** (ekerdt@che.utexas.edu), **J. G. Ekerdt**

University of Texas at Austin

Silicon nanoparticles 2-6 nm in diameter have potential as recombination centers in infrared detectors. These particles can be grown using disilane by chemical vapor deposition (CVD), hot-wire chemical vapor deposition (HWCVD) or by using HWCVD to seed the surface with Si adatoms followed by CVD. Typical CVD growth conditions are 10⁻⁴ Torr disilane and a 600 °C surface temperature. HWCVD can be done at the same temperature but with a lower disilane pressure of 8×10⁻⁸ Torr and a filament current of 4 amps. Seeding the surface using HWCVD prior to CVD ensures a smaller nanoparticle size distribution. It is well known that the presence of dangling bonds at the surface of the as-grown nanoparticle retards the optical properties. Surface passivation can terminate these dangling bonds and form a surface with fewer defects. HWCVD is used to deposit nitrogen, carbon and atomic hydrogen on these particles to form a passivating layer. This structure is then capped with silicon nitride. This talk will describe surface pretreatment necessary to realize nanoparticles in HWCVD on quartz, nanoparticle growth, surface passivation, and optical analysis of the heterostructures.

Friday August 22

10:00 – 10:20

Crosslinking of Copolymer Thin Films by Initiated Chemical Vapor Deposition for Hydrogel and Sensor Applications

Wyatt E. Tenhaeff (tenhaeff@mit.edu), **Karen K. Gleason**

Massachusetts Institute of Technology
Department of Chemical Engineering
Institute for Soldier Nanotechnologies
77 Massachusetts Avenue
Cambridge, MA. 02139

The ability of initiated chemical vapor deposition to finely tune crosslinker contents in copolymer thin films has been exploited to develop two functional, reactive polymer systems. The first system consists of hydrogel films of poly[maleic anhydride-co-dimethylacrylamide-co-di(ethylene glycol) di(vinyl ether)] (PMaDaDe) covalently attached to silicon substrates using the coupling agent 3-aminopropyl-ethoxydimethylsilane. The film composition, characterized by x-ray photoelectron spectroscopy (XPS), was 14 mol% maleic anhydride, 76 mol% dimethylacrylamide, and 10% di(ethylene glycol) di(vinyl ether). The swelling of the films in water is pH-dependent, with a maximum swelling ratio of 14 at pH = 8. The hydrogels were exposed to 0.1 M cysteamine solutions in 2-propanol for 30 minutes to convert 97% of the anhydride functional groups to carboxylic acid and amide functionalities, confirmed by XPS and Fourier transform infrared spectroscopy. The functionalization yielded free thiol groups at the surface, which were used to covalently attach CdSe/ZnS core-shell semiconductor nanoparticles to the hydrogels. In the second system, reactive films were deposited on 100nm-thick silicon nitride cantilevers. A low power micromechanical sensor switch was designed based on the reactivity of the polymer. Upon exposure to 0.87 mol% hexylamine in nitrogen, the resistance of the sensor dropped by over six orders of magnitude within two minutes.

Friday August 22

Novel Materials 2

Chair: Jean-Eric Bourée

Day: **Friday August 22** Time: **10:40**

10:40 – 11:20 Invited Lecture:

Amorphous Silicon Germanium Films and Solar Cells Deposited by HWCVD

L.M. Gedvilas, Y. Xu, R.C. Reedy Jr.,^aG. Yue, ^aB. Yan, ^bD.L Williamson, and A.H. Mahan (harv_mahan@nrel.gov)

National Renewable Energy Laboratory, Golden, CO 80401

^aUnited Solar Ovonic LLC, Troy, MI 48084,

^bColorado School of Mines, Golden, CO 80401

We report the results of incorporating a-SiGe:H alloy films grown by HWCVD into n-i-p solar cell devices. A previous collaboration with United Solar enabled an initial device efficiency of 8.64% for an ungraded a-SiGe:H i-layer containing ~ 40 at.% Ge (no Ge profiling). The deposition rate for this i-layer was ~ 4.1Å/s, and the efficiency was quite favorable compared to a (double Ge profiled) PECVD device deposited at a similar (high) deposition rate. We also deposited companion i-layers on c-Si to compare and contrast the film structure, H bonding and impurity content of the HWCVD and PECVD i-layers used in these devices. Increasing the GeH₄ depletion in the HWCVD films causes an increase in both the H preferential attachment and R* (microstructure fraction). These trends are attributed to excess H in the gas phase progressively etching the weaker bonds (Ge-H versus Si-H) in the increasingly porous film, with the H decorating the microvoid structures. Preliminary results show that the XRD medium range order is already optimum in the highest efficiency HWCVD i-layers, suggesting that energetic bombardment may not be necessary to achieve well ordered films. Where possible, additional comparisons will be made between HWCVD and PECVD a-SiGe:H device layers.

Friday August 22

11:20 – 11:40

**Crystallization and Hydrogen Effect in TiO₂ and SnO₂
by Hot-wire CVD**

S. Nonomura (Nonomura@gifu-u.ac.jp), **T. Iida**, **T. Masuda**, **R. Koie**, **N. Yoshida**,
and **H. Natsuhara**

Environmental & Renewable Energy Systems, Graduate School of Engineering,
Gifu University, National University Corporation

There are few reports concerning oxide materials made by a hot-wire CVD technique and the details are not cleared. This paper deals with depositions of oxide films, TiO₂ and SnO₂, using the hot-wire CVD. Both films are used as a transparent conductive oxide (TCO) and a protective layer for deoxidization by atomic hydrogen for large area Si thin film solar cells. The problem is expensive because the commercially available TCO films are prepared at around 550 C by a thermal CVD. We have tried to reduce the crystallization temperature by using the hot-wire CVD technique and studied a role of atomic hydrogen.

The crystallization of SnO₂ starts at 150 C of surface temperature. A fully nano-crystallized TiO₂ films are obtained at 170 C. The addition of hydrogen to source gases during the deposition of TiO₂ and SnO₂ films enhanced the surface reaction. The nano-crystalline structure of TiO₂ changes from anatase to rutile. In SnO₂, a (221) orientation of rutile structure changes to a (101) orientation. In oxide materials, these effects are limited in the deposition condition of small amount of hydrogen. Addition of much higher hydrogen concentration starts an etching effect of oxygen atoms.

Friday August 22

11:40 – 12:00

**iCVD Growth of Poly(vinylimidazole) and Poly(vinylimidazole)-
vinyl(pyrrolidone)**

Guohua Chen[†] (kechengh@ust.hk), **Kenneth K.S. Lau**[†] and **Karen K. Gleason**
(kkg@mit.edu)

Department of Chemical Engineering, Massachusetts Institute of Technology
77 Massachusetts Avenue, Cambridge, MA, 02139, USA

* On sabbatical leave from Department of Chemical Engineering, The Hong Kong University of Science and Technology, Clear Water Bay, Kowloon, Hong Kong, China, kechengh@ust.hk

[†] Current address: Department of Chemical and Biological Engineering, Drexel University, 3141 Chestnut Street, Philadelphia, PA 19104, USA

Imidazole group plays an important role in a-chymotrypsin catalysis, metal-ion complexation, counterion or dye binding. Poly(vinylimidazole), PVI, is also a good model polymer interacting with neutral salts. The Poly(vinylimidazole)-vinyl(pyrrolidone) copolymer PVI-PVP, can be used to produce highly functionalized polymers.

PVI and PVI-PVP thins films were achieved via initiated chemical vapor deposition (iCVD) method, a solvent-free process to form films under mild conditions. The polymerization was initiated by hot wire heated *tert*-butyl Peroxide (TBPO). The chemical structure and compositions of the polymers were analyzed using FTIR and XPS. The rate of the growth of PVI as a function of the pressure inside the iCVD reactor measure to be 1 nm/h·mTorr.

Friday August 22

12:00 – 12:20

**Precipitation of Si nanocrystals in Si-rich Silicon Nitride layers
deposited using HWCVD**

Antisen A M, Solanki C S (chetanss@iitb.ac.in)

Department of Energy Science and Engineering, Indian Institute of Technology Bombay,
Mumbai – 400076, India Ph: 91-22-2576-7890 Fax: 91-22-2576-4890

The optical and electrical properties of silicon can be changed by controlling its band gap. Si band gap can be increased than the bulk material by reducing the size of Si crystals smaller than its Bohr's exciton diameter (4.9 nm) due to Si quantum confinement effects. These are known as nanocrystals or quantum dots.

This work is focused on fabrication of silicon nanocrystals embedded in silicon nitride matrix using Hot Wire CVD. The Si nanocrystals are obtained by the precipitation of silicon from silicon rich nitride layer. Samples were deposited by varying silane to ammonia ratio. Initially the conditions were optimized to get the Si rich SiN layers. Silane and ammonia flow rates were varied from 1/5 to 1/35 sccm and variation of refractive index from 2.23 to 1.89 were obtained from Ellipsometry measurements. It is known that silicon rich silicon nitride samples have refractive index greater than 2. These results were confirmed by FTIR Si-N peak position of samples.

In order to deposit the Si rich SiN layers, SiH₄ to NH₃ flow ratio of 1:10 was used. Thick Si rich SiN layers, instead of thin alternative multilayer of a-Si/SiN, are deposited to obtain Si nanocrystals. The samples with a thickness of about 100 nm were annealed at 1000 °C to for precipitation of Si nanocrystals. Raman, XRD and TEM analysis of the samples will be reported in the full paper.

Saturday August 23

Applications 1

Chair: Kenneth K.S. Lau

Day: **Saturday August 23** Time: **09:00**

09:00 – 09:40 Invited Lecture:

Hot-wire Chemical Vapor Deposition (HWCVD): a useful technique for tailoring surfaces for biomedical applications

**Laura Montero⁽¹⁾, Núria Mari⁽¹⁾, Carlos E. Semino^(1,3) Karen K. Gleason⁽²⁾,
Salvador Borrós⁽¹⁾** (salvador.borros@iqs.url.edu)

(1) Institut Químic de Sarrià-Universitat Ramon Llull, Via Augusta 390, 08017, Barcelona (Spain).

(2) Department of Chemical Engineering, Massachusetts Institute of Technology, 77 Massachusetts Avenue, Cambridge, MA, 02139-4307, USA

(3) Center for Biomedical Engineering, Massachusetts Institute of Technology, 77 Massachusetts Avenue, Cambridge, MA, 02139-4307, USA

Of special interest in biomaterials is the interface between the synthetic material and the biological environment, since most of the biological reactions occur at this level. The broad interdisciplinary area where properties and processes at this interface are investigated and biofunctional surfaces are fabricated is called Biological Surface Science.

There is a need to reexamine many of the current approaches to control biological interactions at surfaces. Instead of preparing a surface, absorbing proteins onto such surfaces and examining the consequence, the new paradigm is to try to mimic what nature does all the time – i.e., design surfaces that will extract precisely the biological reactions. The final goal is to obtain a controlled interaction between cells and synthetic substrates. Usually the most important parameter in this area is biomolecular recognition. However, the mechanical signaling of the surface on the cells has to be taken into account when biointerfaces are designed. In many cases the cell mechanical interaction with the extracellular matrix (ECM) governs cell behavior.

In this work, the design of nanostructured surfaces that combines both chemical and mechanical cell signaling using HWCVD is described. The polymeric structures obtained can be easily tailored using such technique allowing to control very easily the film viscoelasticity and the biomolecules attached to it.

Saturday August 23

09:40 – 10:00

Initiated Chemical Vapor Deposition of Biopassivation Coatings**W. S. O'Shaughnessy¹** (shannan@scoat.com), **D.J. Edell²**, and **K. K. Gleason³**¹ SteriCoat Corp, Cambridge MA² Innersea Technology Inc., Bethesda MA³ Department of Chemical Engineering, Massachusetts Institute of Technology

Recent advances in the field of neuroprosthetics have brought the possibility of human utilization into the near term. However, current implant technology still suffers from loss of functionality due to scar tissue buildup at the implant site. Implant coating chemistries currently in use require thicknesses of 10-25 microns in order to provide the required electrical insulation, significantly increasing the diameter of the neural probe shanks and consequently the amount of damage upon implantation.

In this work, a novel biopassivation coating is created using initiated chemical vapor deposition (iCVD). Trivinyl-Trimethyl-Cyclotrisiloxane is utilized as a self crosslinking monomer, initiated by radical fragments generated from the thermal breakdown of Tert-Butyl Peroxide. The resulting coating is a highly crosslinked organosilicon polymer matrix which is synthesized directly on the surface of the substrate. This novel material possesses an electrical resistivity of 5×10^{15} Ohm-cm, allowing for a coating thickness on the order of only 5 microns to provide the required electrical protection. In addition, the material is insoluble, flexible, and extremely adherent to silicon substrates. This material has been demonstrated to retain its electrical properties in a simulated biological environment for over 3 years and initial biocompatibility with neural cells has also been demonstrated.

Saturday August 23

10:00 – 10:20

“Clickable” Polymer Film via Initiative Chemical Vapor Deposition (iCVD)**Sung Gap Im, Byeong-Su Kim, Wyatt E. Tenhaeff, Paula T. Hammond, and Karen K. Gleason** (kkg@mit.edu)

Department of Chemical Engineering and Institute for Soldier Nanotechnologies, Massachusetts Institute of Technology, 77 Massachusetts Avenue, Cambridge, MA 02139

A new “click chemistry” active functional polymer film was directly obtained from a commercially available monomer of propargyl acrylate (PA) via initiative chemical vapor deposition (iCVD). Fourier transform infrared (FTIR) spectra confirmed that the click-active acetylene functional group was retained after the iCVD process. The degree of crosslinking could be controlled by intentionally adding crosslinker, such as ethylene glycol diacrylate (EGDA) that was polymerized with PA to form click-active, completely insoluble copolymer. The formed iCVD polymers could also be grafted on various inorganic substrates with silane compounds. These crosslinking and grafting techniques give iCVD polymers chemical and mechanical stability, which allows iCVD polymers applicable to various click chemistry without any modification of reaction conditions. Pre-patterned iCVD polymer could be obtained via photolithography and an azido-functionalized dye molecule was also successfully attached on iCVD polymer via click chemistry. Moreover, pPA film demonstrated sensitivity to e-beam irradiation, which enabled clickable substrates having nanometer scale patterns without requiring the use of an additional e-beam resist. Direct e-beam exposure of this multifunctional iCVD layer, a 200 nm pattern, and QD particles were selectively conjugated on the substrates via click chemistry. Thus, iCVD pPA has showed dual functionality as of “clickable” e-beam sensitive material.

Saturday August 23

Applications 2

Chair: Harv Mahan

Day: **Saturday August 23** Time: **10:40**

10:40 – 11:20 Invited Lecture

Hot Wire Chemical Vapor Deposition of adherent diamond coatings on cemented tungsten carbides**R. Polini**¹ (polini@uniroma2.it), **M. Barletta**², **G. Cristofanilli**², **G. Rubino**²,
V. Valentini³¹ Dipartimento di Scienze e Tecnologie Chimiche, Università di Roma Tor Vergata, Via della Ricerca Scientifica 1, 00133, Roma, Italy² Dipartimento di Ingegneria Meccanica, Università di Roma Tor Vergata, Via del Politecnico 1, 00133, Roma, Italy³ CNR, Istituto dei Sistemi Complessi ISC, Area della Ricerca di Roma 1, Via Salaria km 29.300, C.P. 10, 00016 Monterotondo Scalo (RM), Italy (polini@uniroma2.it)

Surface-modified cutting tools and wear parts exhibit enhanced properties and lower wear rates. Significant improvements can be obtained by depositing a hard, tough, low friction and oxidation-resistant coating. Substrates currently used for surface-modified tools are based mainly on cemented tungsten carbide (WC-Co), high-speed steels or ceramic materials (SiAlON). Particularly cemented carbides provide a number of basic properties essential for the successful application of chemical vapor deposition (CVD) coatings. In fact, WC-Co withstands the many hours at high CVD temperatures without structural changes that could affect the performance of the final product.

CVD diamond exhibits outstanding properties, namely superhardness, chemical inertness and large thermal conductivity. The coating of 3-D shapes and the ease of scale-up are feasible with low pressure HWCVD at relatively low costs.

However, the direct deposition of diamond onto WC-Co substrates presents several problems. In fact, the interfacial graphitization of diamond by cobalt can reduce the adhesion strength substantially.

Several methods to improve the adhesion of CVD diamond films on WC-Co will be discussed. In particular, the use of CrN-base interlayers deposited by Physical Vapor Deposition (PVD) and of SiC interlayers deposited by HWCVD has proven to be a viable way to deposit adherent diamond coatings on WC-Co.

Saturday August 23

11:20 – 11:40

Initiated Chemical Vapor Deposition of Fluoropolymer Coatings for the Surface Modification of Complex Geometries**Malancha Gupta and Karen K. Gleason** (kkg@mit.edu)

Massachusetts Institute of Technology

This talk will present an in-depth mechanistic study of the initiated chemical vapor deposition (iCVD) deposition of low surface energy poly(1H,1H,2H,2H-perfluorodecyl acrylate) (PPFDA) thin films. PPFDA films have many uses due to their hydrophobic and oleophobic properties. Fourier transform infrared spectroscopy and x-ray photoelectron spectroscopy of the iCVD PPFDA films showed complete retention of the fluorine moieties. Deposition rates as high as 375 nm/min were achieved. It was found that the deposition rate and the molecular weight increases with decreasing substrate temperature and increasing monomer partial pressure. Quartz crystal microbalance measurements showed that these effects correlated with an increased monomer concentration at the surface.

The iCVD technique was used to functionalize electrospun fiber mats and fiber-on-end (FOE) polymeric membranes with PPFDA in order to make water-repellant, self-cleaning membranes. It was found that the iCVD process can be used to functionalize membranes with very high aspect ratio (~80) pores. X-ray photoelectron microscopy data confirmed the presence of the PPFDA coating on the topside and backside of the membranes and electron microprobe analysis confirmed the presence of the coating along the pore wall. Static and dynamic contact angle measurements (low hysteresis) showed that the coated membranes exist in the Cassie-Baxter state.

Saturday August 23

11:40 – 12:00

Precursor Cat-CVD a-Si films for formation of high-quality poly-Si films on glass substrates by flash lamp annealing

Keisuke Ohdaira (ohdaira@jaist.ac.jp), **Kazuhiro Shiba**, **Hiroyuki Takemoto**, **Tomoko Fujiwara**, **Yohei Endo**, **Shogo Nishizaki**, **Jang Young Rae**, and **Hideki Matsumura**

Japan Advanced Institute of Science and Technology (JAIST)
1-1 Asahidai, Nomi, Ishikawa 923-1292, Japan

Amorphous silicon (a-Si) films prepared by catalytic chemical vapor deposition (Cat-CVD) show high suitability for precursor films of polycrystalline Si (poly-Si) formed on glass substrates by flash lamp annealing (FLA). Intrinsically small amount of hydrogen contents of as low as 3% in the Cat-CVD a-Si films enables us to skip dehydrogenation processes prior to FLA, which process is in general necessary for crystallization of a-Si by excimer laser annealing in order to avoid peeling of Si films. With the help of Cr films inserted between Si films and glass substrates, a-Si films exceeding 4 μm in thickness can be crystallized without any serious peeling of Si films. These poly-Si films show high crystallinity close to unity and clear band-to-band photoluminescence, indicating excellent crystal quality. In addition, these films show long carrier lifetimes of over 10 μs after a defect termination process of high-pressure water vapor annealing, and indicate negligible diffusion of Cr atoms as well as of dopant B and P atoms during FLA. These properties are significantly important for realization of high-efficiency solar cells, and we have succeeded in demonstrating fabrication and actual operation of solar cells using these poly-Si films.

Saturday August 23

12:00 – 12:20

Reduction of oxide layer on lead-free solder material by high density ammonia radical

S. Kagata (g346417s@tobata.isc.kyutech.ac.jp), **T. Nakashima**, **A. Izumi**

Kyushu Institute of Technology

The development of lead-free solder is advanced along with the prohibition of sales of an electric equipment including lead by enforcing the RoHS (Restriction of Hazardous Substances) instruction. However, the solder is oxidized easily, diverse issues happen because of the oxidation, therefore the oxide removal technology is strongly required. In this work, we propose a novel oxide reduction technology of the solder raw material using the ammonia decomposed species generated by HW (hot-wire) method. It was confirmed that the tin oxide was able to be reduced by this treatment, and the re-oxidation was suppressed. Moreover, it was confirmed that the particle size of lead free material was controlled. On the other hand, we clarified that the activation energy on the reduction of tin oxide was almost zero by this processing which is showing the temperature dependence was low.

Saturday August 23

Nanostructures and Patterning

Chair: Malancha Gupta

Day: **Saturday August 23** Time: **13:40**

13:40 – 14:20 Invited Lecture:

Synthesis of Nanowires Using Hot Filament Chemical Vapor Deposition (HFCVD)**Sreeram Vaddiraju** (sreeramv@mit.edu)Department of Chemical Engineering
Massachusetts Institute of Technology, Bldg. 66-419, Cambridge, MA 02139

Novel methods, employing the aid of no foreign catalyst clusters or templates, for the synthesis of nanowires will be presented. Unlike catalyst-assisted vapor-liquid-solid schemes, these methods eliminate the adverse affects arising from the contamination of the nanowires.

The first method involves the synthesis of group III-nitride nanowires using low-melting metal pools in conjunction with hot filaments. Here, hot filaments were employed for the dissociation of either molecular nitrogen or ammonia into atomic nitrogen. Supersaturation of the molten metal droplets with nitrogen lead to the spontaneous nucleation and basal growth of nanowires in this approach.

The second method discusses the synthesis of transition metal and metal oxide nanowires using hot wire chemical vapor deposition. In contrast to the first method, hot filaments were used as the source of the metal in this method. The chemical vapor transport of metal oxides, generated from the filaments, onto substrates lead to the formation of either metal or metal oxide nanowires. Metal nanowires were obtained on substrate maintained above the decomposition of the respective metal oxide. Similarly, metal oxide nanowires were obtained on substrates maintained below the temperature of the metal oxide. The nucleation and growth steps involved in the formation of these nanowires will be discussed.

Saturday August 23

14:20 – 14:40

Silicon nanorods deposited by glancing angle Catalytic Chemical Vapor Deposition**Fengzhen Liu** (Liufz@gucas.ac.cn), **Yanhong Ma, Meifang Zhu, Zhenzhong Sun and Chaowei Li**College of Physical Sciences, Graduate University of Chinese Academy of Sciences,
PO Box 4588, Beijing 100049, China

Films formed by glancing angle deposition (GLAD) exhibit an isolated columnar nanostructure if the incident vapor flux arrives at an oblique angle with respect to the substrate surface.

Vertically oriented amorphous and microcrystalline Si nanorods were grown on c-Si (111) substrates by the combination technique of GLAD and Cat CVD. The incident angle of radical flux from silane through the heated filament reaches to the rotated substrate is 85°.

The orientation, diameter and density of Si nanorods varying with the deposition pressure, rotation rate and substrate were investigated. The structures of Si nanorods were characterized by scanning electron microscopy (SEM), spectroscopic ellipsometry (SE) and Raman scattering, etc. The SEM images show highly oriented Si nanorods in vertical posts. The diameters of Si nanorods are substrate dependent to be 47, 58 and 65 nm for nanorods on c-Si, stainless steel and glass, respectively, as fixing the source-substrate distance of 15cm. The void fractions of a-Si and μ c-Si nanorod films determined from SE are approximately 38% and 46%, respectively. The deposition rate can be modified easily. However, controlling the uniformity of nanorod diameter in growth direction, crystallization in situ and doping of the core/shell structure are important issues for the application of coaxial Si nanorods as solar cells.

Saturday August 23

14:40 – 15:00

Formation of Isolated Carbon Nanofibres with Hot Wire CVD using Nanosphere Lithography as Catalyst Preparation Technique

Z.S. Houweling (Z.S.Houweling@uu.nl), **V. Verlaan** and **R.E.I. Schropp**

Utrecht University, Faculty of Science, Debye Institute for Nanomaterials Science, SID – Physics of Devices, P.O. Box 80.000, 3508 TA Utrecht, the Netherlands

Isolated carbon nanofibres have been formed in an experimental hot wire chemical vapor deposition (HWCVD) reactor. Heated tungsten filaments are used to decompose a controlled mixture of ammonia, methane and hydrogen at a filament temperature of 2000° C. The structure of the deposited carbon nanofibres and tubes are mainly determined by nickel catalyst particles of nanometer dimensions on the substrate. To obtain a highly ordered matrix of these nanoparticles with excellent monodispersity, nanosphere lithography was used. Nickel is evaporated through a silica colloid monolayer mask, when the colloids are removed a successive thermal treatment allows the formation of nickel nanospheres. The dimensions of the catalyst particles and areal density are controlled by the diameter of the silica colloids and thickness of the nickel film.

These nickel nanoparticles are tested as catalyst in HWCVD depositions. Indeed, carbon nanofibers are deposited. The deposited carbon structures and catalyst containing substrates have been characterised with Scanning Electron Microscopy, Back Scatter Electrons analysis, Energy Dispersive X-ray spectroscopy, RAMAN spectroscopy and Rutherford Back Scattering measurements.

Saturday August 23

15:00 – 15:20

Resist-Free Patterning of Low Dielectric Constant Polymer Using iCVD

Nathan J. Trujillo (njt@mit.edu) and **Karen K. Gleason**

Department of Chemical Engineering, MIT

As the average feature size in integrated circuits continues to decrease, reducing the dielectric constant of the interconnect dielectric (ILD) becomes crucial to minimizing RC delay, power consumption and cross-talk noise. Selective deposition of patterned low-k materials is an off ITRS-roadmap approach to process step reduction, whose successful implementation is both economically and environmentally beneficial. Initiated Chemical Vapor Deposition (iCVD) is a low-energy, one step, solvent-free process and was used to create thin films from a novel low-k OSG precursor, 1,3,5,7-Tetravinyltetramethylcyclotetrasiloxane (V4D4), with a bulk dielectric constant of ~ 2.7. Large well-ordered patterns of iCVD V4D4 were successfully grafted onto silicon substrates using a monolayer assembly of colloidal nanoparticles to define the pattern; low-k features as small as 75 nm were reliably obtained. Other resist-free patterning methods have been demonstrated. 193 nm lithography was used to pattern vinyl monolayers, which are covalently tethered to Si substrates. The unexposed monolayer acts as an underlying foundation to covalently link the iCVD polymer; the exposed (patterned) regions are recovered by removing non-tethered polymer with an environmentally friendly solvent such as IPA.

Saturday August 23

Processing

Chair: Friedhelm Finger

Day: **Saturday August 23** Time: **15:40**

15:40 – 16:00

A novel patterning technique using super-hydrophobic PTFE thin films prepared by Cat-CVD method**Michihisa Takachi** (s0730048@jaist.ac.jp), **Hiroaki Yasuoka**, **Keisuke Ohdaira**, **Tatsuya Shimoda** and **Hideki Matsumura**JAIST (Japan Advanced Institute of Science & Technology)
Asahidai, Nomi-shi, Ishikawa-ken 923-1292, Japan

Following the pioneer work by K. Gleason et al., we have also succeeded in obtaining 10 nm-thick super-hydrophobic PTFE (poly-tetrafluoroethylene: “Teflon” in commercial name) films with water-repellent angles over 150° by Cat-CVD (catalytic chemical vapor deposition).

Here, we attempted to utilize this property as a new patterning method. If we can make patterns of non-water-repellent area in hydrophobic films, by dropping highly functional liquids such as liquid metals, liquid semiconductors and liquid insulators on them, we can easily make patterns of such materials. For instance, metal lines are formed by simply dipping samples with patterned PTFE films in functional solution. This may skip a lot of processes in formation of metal wiring to fabricate liquid crystal displays, for instance. As a first fundamental study, we have prepared hydrophobic line patterns with a width of 50 μm and water repellent areas on a glass substrate by deposition of PTFE films through a metal mask and have demonstrated completely selective placement of water drops only on the hydrophobic lines.

Saturday August 23

16:00-16:20

Continuous hot-wire chemical vapor deposition on moving glass substrates**A. Bink, M. Brinza, and R.E.I. Schropp** (r.e.i.schropp@uu.nl)

Utrecht University, Faculty of Science, Debye Institute for Nanomaterials Science, SID-Physics of Devices

In hot-wire chemical vapour deposition (HWCVD) the reactive radicals are created by the catalytic decomposition of source gases at transition metal wires. Thus, the radical source can be regarded as a linear source. If the (colder) ends of the linear source are sufficiently far from the outer edges of the substrate, the source is uniform over the entire width of the substrate. This makes HWCVD a very suitable technique for homogeneous deposition of thin films on continuously moving substrates in an in-line manufacturing system. This process is further aided by the fact that transport of insulating substrates (such as glass) can be easily achieved, as the substrate is not part of the decomposition mechanism as in plasma enhanced CVD. Rigorous grounding of the moving substrates is not required, and no special care needs to be taken to make shields or liners equipotential planes. Moreover, as the creation of dust particles in the gas phase can be avoided, deposition has been undertaken with the substrates facing *upward*, thus further simplifying the mounting of the substrates. At Utrecht Solar Energy Laboratory, the next step towards cheap semiconductor production is now taken by demonstrating uniform deposition on *moving* glass substrates. Amorphous silicon thin films with device-quality properties have been achieved. The dark and photoconductivity on moving substrates were $\sigma_d = 8 \cdot 10^{-12} \Omega^{-1} \text{cm}^{-1}$ and $\sigma_{ph} = 1.9 \cdot 10^{-6} \Omega^{-1} \text{cm}^{-1}$ and the dark activation energy was $E_a = 0.92 \text{eV}$, which indicates intrinsic properties for this 1.85 eV bandgap material. We will also show the deposition of microcrystalline silicon moving substrates.

Saturday August 23

16:20 – 16:40

Fabrication of thin film amorphous silicon solar cells by HWCVD on flexible substrates

P. Alpuim¹ (palpuim@fisica.uminho.pt), **G.M. Junior¹**, **F. Almeida¹**, **S.A. Filonovich²**, **J.E. Bourée³**

¹ Departamento de Física, Universidade do Minho, Campus de Azurem, 4800-058 Guimaraes, Portugal

² CENIMAT/I3N-CEMOP, Campus de FCT/UNL, 2829-516 Monte de Caparica, Portugal

³ Laboratoire de Physique des Interfaces et des Couches Minces, CNRS UMR 7647, Ecole Polytechnique, 91128 Palaiseau, France

Amorphous silicon solar cells were fabricated on two different flexible substrates: 125 μm -thick plastic film (polyethylene naphthalate, PEN) at a substrate temperature of 150°C, and 50 μm -thick stainless steel foil at a substrate temperature of 220°C. The precursor gases (silane diluted in hydrogen) were decomposed using a straight tantalum filament ($d=0.5$ mm, $l=14$ cm) heated up to 1750°C. Filament-to-substrate distance was 75 mm. The working gas pressure was 40 mTorr. The photosensitivity of the intrinsic amorphous layers has been measured around 10^5 . Raman spectroscopy revealed a small FWHM (~ 45 cm^{-1}) value for the transverse optical Si-Si vibration peak. The solar cells on plastic have the following p-i-n structure: PEN/ZnO:Al (10 nm)/p⁺-a-Si:H (20 nm)/buffer a-Si:H (8 nm)/i-a-Si:H (400 nm)/n⁺-a-Si:H (30 nm)/Al. The substrate solar cells on stainless steel use the same layer sequence but deposited in reverse order giving rise to an n-i-p device. The role of the wide-bandgap buffer layer is to limit the recombination current at the p-I interface. The AM1.5 efficiency obtained so far for cells on plastic attains 4%, which may be due to the lower structural disorder of the amorphous films as compared to the disorder of the films deposited by the conventional plasma-enhanced CVD.

Saturday August 23

16:40 – 17:00

Low Environmental Impact Processing of Sub-50 nm Interconnect Structures

Chia-Hua Lee, Wyatt Tenhaeff, Karen K. Gleason (kkg@mit.edu)

Department of Chemical Engineering, Massachusetts Institute of Technology, Cambridge, MA, 02139, USA

A novel method for processing of sub-50 nm structures by using carbon nanotube (CNT) masks and integrating quantum dots (QDs) on patterned polymer substrates has been established. Poly(styrene-alt-maleic anhydride) (PSMa) was prepared by the initiated chemical vapor deposition (iCVD) method, an alternative to spin-on deposition. The sub-50 nm PSMa polymer patterns were prepared by low energy oxygen plasma etching by using CNTs as the masks. The water soluble, amine-functionalized QDs underwent the nucleophilic acyl substitution reaction with the PSMa containing anhydride functional groups. This integration method for developing high performance QDs devices on inexpensive, lightweight flexible substrate avoids energy intensive fabrication of high purity silicon wafer.

Sunday August 24

Commercialization and Wrap-up

Chair: Ruud Schropp

Day: **Sunday August 24** Time: **09:00**

09:00 – 09:40 Invited Lecture:

Review of HWCVD Si materials for solar cell application

Qi Wang (qi_wang@nrel.gov)

National Renewable Energy Lab. Golden, CO 80401

Hydrogenated amorphous silicon (a-Si:H) thin films and its application to solar cells fabricated using the hot-wire chemical vapor deposition (HWCVD) or (CAT)-CVD will be reviewed. This review will focus on the comparison to the standard plasma enhance (PE) CVD in the terms of deposition technique, film properties, and solar cell performance. Try to answer the questions of what are advantages of using HWCVD for a-Si:H solar cell research? What are the criteria for industry to adapt this technique for mass production? What is the future of HWCVD?

Sunday August 24

09:40 – 10:20 Invited Lecture:

HWCVD of Polymers: Commercialization and Scale-Up

Hilton G. Pryce Lewis (hilton@gvdcorp.com), **Neeta P. Bansal,**
and **Aleksandr J. White**

GVD Corporation, Cambridge, MA USA

GVD Corporation specializes in process development and equipment design for the production of ultra-thin polymer coatings using hot wire chemical vapor deposition (HWCVD, also known as initiated chemical vapor deposition, iCVD). HWCVD allows many coating compositions to be produced, including fluorocarbon and silicone polymers, copolymers, and vinyl hydrocarbon polymers. It is especially valuable for creating ultrathin layers of insoluble, infusible polymers which are hard to process by conventional means, such as polytetrafluoroethylene (PTFE, Teflon®). HWCVD PTFE coatings are chemically robust, comprised of essentially 100% CF₂, resistant to solvents, conformal to complex surface geometry, and have excellent adhesion to a wide range of substrates. Since the part to be coated remains at room temperature, fragile materials like plastics and fabrics can be coated with ease. GVD has focused on scale-up of the process equipment and has developed several standard coating systems, which will be discussed in this paper. These include laboratory-scale batch coating systems, a medium sized production batch coating system, a large scale custom batch coater, and a pilot scale roll-to-roll web coater. All of GVD's systems are complete with a fully automated, computer based control systems and include options for an effluent monitors and an exhaust scrubber.

Sunday August 24

10:40 – 11:20 Invited Lecture:

Commercial Hot Wire Chemical Vapor Deposition (HWCVD) Equipment – Diamond History to Photovoltaic Future?

Jerry Zimmer¹ (jzimmer@sp3inc.com), **James Herlinger**¹, and **K. V. Ravi**²

1 sp3 Inc.
2 Crystal Solar

sp3 was incorporated with the goal of developing CVD equipment and processes to manufacture products that would exploit the unique properties of diamond. Meeting this goal required a deposition technology that would provide reliable and cost effective production scale manufacturing day after day over large areas. Hot Wire Chemical Vapor Deposition (HWCVD) technology was selected as the best route to achieve this goal.

Since 1993 sp3 has designed, built, and sold 5 generations of hot wire reactors and has performed over 10,000 HWCVD reactor runs in its own facility. The diamond deposition technology covers a range of substrate temperatures from below 350 C to as high as 900 C and can operate at even lower temperatures if required. With this capability the system can provide an excellent platform for researchers wanting to develop or modify the typical films required in the fabrication of photovoltaic devices.

This paper will address sp3's experience using HWCVD reactors as well as how the sp3 Hot Wire system could be used in the development of photovoltaic fabrication processes. Additionally possible scaling paths using HWCVD technology for the cost effective mass production of photovoltaics will be discussed.

Sunday August 24

11:20-12:00 Invited Lecture:

Plasma-assisted Cat-CVD growth of organic-inorganic hybrid materials for flexible film-based electronics

Hiroshi Nakayama^{1,2} (mdf@md-factory.com), **Asako Oka**¹ and **Hiroichi Ito**¹

1 Material Design Factory Co., Ltd.
2 Graduate School of Engineering, Osaka City University

Development of “flexible display devices” like flexible organic light-emitting diodes (OLED) is in progress rapidly. These flexible devices should be made from flexible materials to be matched with the properties of the film substrates. Organic-inorganic hybrid material (OIHM) is a candidate for such functional flexible materials. This paper presents our recent work to develop OIHM by using plasma-assisted catalytic chemical vapor deposition (Cat-CVD) method.

An application of OIHM is a water vapor and/or oxygen barrier film. We have developed barrier films with the alternate coatings of an amorphous SiCN grown by organic Cat-CVD (O-Cat-CVD) and plasma-synthesized Si-doped polyethylene. The water-vapor transmission rate is typically on the order of 0.001 g/m²/day with the total coating thickness of 300nm on polyethylene-naphthalate (PEN) film.

Another application of OIHM is insulating material like SiOC and SiCN. SiOC is so-called “low-k insulator” and is grown by O-Cat-CVD using tetraethoxysilane (TEOS) or dimethyldimethoxysilane (DMDMOS) at low substrate temperatures below 200 C. SiCN is also a quite promising material for water-resistive and high-k insulating materials for flexible devices like OLED and thin-film transistors (TFT). Our recent studies have shown the electric breakdown field higher than 6.5 MV/cm for SiCN film with the thickness of 200nm on glass substrate.

Poster Presentations

P1: Investigation of hydrogen radical density generated by various kinds of catalyzer

K. Abe, M. Ida, A. Izumi / Kyushu Institute Technology
S. Terashima, T. Sudo, Y. Watanabe, Y. Fukuda / Extreme Ultraviolet Lithography System Development Association (EUVA)
 g346403k@tobata.isc.kyutech.ac.jp

Atomic hydrogen cleaning is paid attention as a novel low-temperature cleaning method such as copper interconnects and Extreme Ultraviolet (EUV) exposure apparatus. In this work, we investigated the hydrogen radical density of various kinds of catalyzer such as W, Ta and 5%Re·W, etc. using a hydrogen radical monitor. The radical density of $3 \times 10^{11} \text{ cm}^{-3}$ was obtained for W catalyzer at the temperature of 1700°C. The Arrhenius plots of hydrogen radical density versus catalyzer temperature were obtained for the calculation of activation energy. The activation energy became 2.53 eV for W catalyzer.

P2: Amorphous silicon thin film solar cells deposited entirely by Hot-Wire Chemical Vapour Deposition at low temperature (<150°C)

Fernando Villar, Aldrin Antony (aldrinantony@ub.edu), **Jordi Escarré, Daniel Ibarz, Rubén Roldán, Marco Stella, Delfina Muñoz, José Miguel Asensi and Joan Bertomeu**

Departament de Física Aplicada i Òptica, Universitat de Barcelona, Martí i Franquès 1-11, E08028-Barcelona, Spain.

Amorphous silicon n-i-p solar cells have been fabricated entirely by Hot-Wire Chemical Vapour Deposition (HW-CVD) at low process temperature of 150°C. Textured Ag/ZnO back reflectors deposited on Corning 1737F were used as the substrates. The textured silver was deposited by rf magnetron sputtering at a substrate temperature of 450°C. Doped layers of very good conductivity and intrinsic a-Si:H layer of much less defect levels were used for the cell fabrication. A double n-layer ($\mu\text{-Si:H/a-Si:H}$ doped with phosphorus) and boron doped $\mu\text{-Si:H}$ p-layer were used for the cell. Indium tin oxide (ITO) layer of 80nm deposited at room temperature by rf magnetron sputtering was used as the top contact for the cell. The electrical and optical properties of the silicon layers were investigated by ellipsometry, Raman spectroscopy, X-ray diffraction, PDS and four probe conductivity measurements. In this paper, we report the characterization of all these layers deposited over glass and PEN substrates, and the integration of these layers to fabricate solar cell at low process temperature. An initial efficiency of 4.62% has been achieved for the n-i-p cells deposited over glass/Ag/ZnO textured back reflectors.

P3: Organic-inorganic multi-layer moisture diffusion barrier systems by means of a PE-CVD/i-CVD combined approach

G. Aresta (g.aresta@tue.nl), **M. Creatore, M.C.M. van de Sanden**

Plasma and Materials Processing group, Department of Applied Physics, Eindhoven University of Technology, 5600 MB Eindhoven, the Netherlands

Amongst the challenges and emerging technologies in the field of electronic displays/lighting and flexible electronics, the research on the “ultimate gas/vapor barrier performance” which allows long term stability devices is presently drawing considerable attention. The state-of-the-art is a μm thick multi-layer system, often consisting of an inorganic layer, which is considered the effective gas/vapor barrier, and an organic layer, which role is still under debate and not yet unravelled.

Our work aims to gain insight into the role of the organic interlayer in affecting the gas/vapor permeation mechanisms in the stack by investigating the effects of its chemistry, density/porosity and surface morphology on the final barrier performances. The chosen model chemistry in this project is a SiO_2 -based multi-layer system deposited by means of a combined initiated-CVD/ plasma-enhanced CVD set up, which couples the development of an organic layer according to a vacuum-compatible free radical-based polymerization process (initiated-CVD) with the plasma deposition of the inorganic barrier layer.

For these purposes, a new reactor is presently built up. The structure and the properties of both the organic and inorganic synthesized layers will be investigated by means of different techniques as well as the barrier effect (related to the organic/inorganic layer chemistry and thickness) of the multilayer films evaluated by means of the calcium test. Also the optical properties and the porosity of the films (related to the barrier performance) will be investigated by means of spectroscopic ellipsometry and ellipsometric porosimetry, respectively.

P4: A Kinetic Study of the Initiated Chemical Vapor Deposition (iCVD) of Poly(Ethylene Glycol)

Ranjita K. Bose and Kenneth K.S. Lau (klau@drexel.edu)

Department of Chemical and Biological Engineering, Drexel University
3141 Chestnut Street, Philadelphia, PA 19104

Initiated chemical vapor deposition (iCVD) is a low temperature variant of hot-wire CVD (HWCVD) which has been used to produce a wide variety of polymers like fluoropolymers, acrylic polymers and silicone polymers through a free radical polymerization mechanism. An initiator is activated in the gas phase by a resistively heated filament array. The radicals thus generated undergo surface polymerization on a cooled substrate by attaching to multiple monomer units.

In this work, iCVD has been extended to enable the cationic polymerization of ethylene oxide. Ethylene oxide, having a ring structure, is polymerized to poly(ethylene glycol) (PEG) using boron trifluoride as a cationic initiator. Fourier transform infrared (FTIR) and nuclear magnetic resonance (NMR) spectroscopies showed that the chemistry of PEG obtained by iCVD synthesis matched closely to PEG obtained from solution synthesis. Variable angle spectroscopic ellipsometry (VASE) gave a measure of the rate of polymerization. Gel permeation chromatography (GPC) was used to determine the kinetic chain length. The effect of substrate temperature, system pressure and reactant flowrates were studied to develop a kinetic model for the cationic iCVD of PEG. This work demonstrates that iCVD can directly apply liquid polymerization chemistries with great versatility.

P5: Nanocrystalline diamond coated WC drawing dies for Industrial Applications

Maneesh Chandran^{1,2}, C.R. Kumaran^{1,2}, S.S. Bhattacharya^{2,3}
and M.S. Ramachandra Rao^{1,2} (msrrao@iitm.ac.in)

¹Department of Physics, ²Nano Functional Materials Technology Centre and
³Department of Metallurgical and Materials Engineering
Indian Institute of Technology Madras, Chennai – 600 036, India

Nanocrystalline Diamond (NCD) films are considered to be the best candidates for wear resistant and other tribological coating applications because of the excellent combination of its high hardness (>100 GPa) and low friction coefficient (< 0.05) combined with high thermal conductivity (16 Wcm⁻¹K⁻¹) [1]. Hot Filament CVD (HFCVD) has an advantage over plasma CVD due to its excellent adaptability to product geometries, reproducibility, moderate activation temperature, and easiness to construct etc. Herein, we report on NCD coated tool for industrial applications. The nanocrystalline diamond films are grown on a Tungsten Carbide (WC) drawing die and on Si substrate using HFCVD at a temperature of ~ 900°C. Methane and Hydrogen are used as precursor gases. The gas flow dynamics have been varied in order to optimize the growth. The nanomechanical testing shows the hardness of ~ 54.5 GPa and a reduced modulus of ~ 439.5 GPa on the interior of the NCD coated drawing die for a contact depth of 76.6 nm. The Raman spectra (532 nm) at different points. This ensures the homogeneity of the coating. The working life time of the diamond coated drawing die can increase by a factor of 10-15 times as compared with that of WC drawing dies. NCD coated Si substrate was etched out to get a free standing diamond film. The optical transmittance study on the free standing diamond film showed a transmittance of 65 % in the visible range and 80% in the IR range. We thank Sp3 diamond inc. for providing us with NCD coated Si and for coating the WC die inserts; WC die inserts were provided by M.M. Murugappa Industries, Chennai.

[1] Tarun Sharda and Somanath Bhattacharyya, *Encyclopedia of Nanoscience and Nanotechnology*, 2, 337 (2004).

P6: Structural characterization of Nickel coated Carbon fibers

M.Fares¹, M.Y.Debili² (mydebili@yahoo.fr)

¹ Division of study and development of nuclear instrumentation, nuclear electronic laboratory, Research center of Birine B.P 180 Ain Oussera – Djelfa Algérie.

² LM2S, Physics department, Faculty of science, Badji-Mokhtar University BP 12 Annaba 23200 Algérie.

This study deals with structural characterization of nickel coated carbon fibers (Ni-C) prepared by CVD (gaseous decomposition of carbonyl Ni(Co4). They present good surface properties which intends them for applications as reinforcements or neutron guides. Average grain size estimation of as-produced fibers, from x-ray diffraction pattern using Scherer formula gives rise to relatively low values: $D_g(\text{carbon})=2.857\text{nm}$ $D_g(\text{nickel})=14.12\text{nm}$.

Heat treated specimen at 500°C are formed by carbon, nickel and hexagonal close packed (HCP) Ni₃C carbide (a=0.265nm, c=0.43nm). The carbon atoms are in interstitial position in hexagonal unit cell and this, probably in the grain boundaries.

Within the framework of this work, we were interested by oxidation at 500°C of nanocrystalline Ni-C fibers. The oxide formed is NiO (nickel protoxyde) FCC (a = 0.417nm) modifying the properties of surface of the Nickel coating. The fibres were analyzed before and after oxidation by x-ray diffraction, and scanning electron microscopy (SEM). We could show that the oxidation led to an increase in average grain size of both carbon and nickel regarding to as elaborated specimen and heat treated at 500°C under vacuum.

P7: Influence of the silane transport on the electrical properties of intrinsic microcrystalline silicon deposited by hot wire chemical vapor deposition

P.A. Frigeri (pfrigeri@ub.edu), O. Nos, C. Frevert, S. Bengoechea, M.Stella, J.M. Asensi, J. Bertomeu

Dept. de Física Aplicada i Òptica, Universitat de Barcelona
Barcelona-08028, Spain, Ph: +34934039221, fax: +34934039219

Intrinsic microcrystalline silicon is being more and more studied since its implementation as active layer in micro-morph solar cells. The Hot Wire Chemical Vapor Deposition (HW-CVD) technique is able to deposit electronic grade material and has the advantage over other deposition techniques of producing sharper interfaces between the layers due to the lack of ion bombardment. Hence, the subtle chemistry involved in the growth of the microcrystalline film is governed exclusively by the diffusion of the different radical species. However, the deposition chemistry is not yet completely clarified due to the wide range of possible reactions. Up to now, the quality of the deposited film was considered to depend only on the classical deposition parameters: pressure, substrate temperature, filament temperature, catalyzer to substrate distance and type of substrate. In our study we show that the way the chemical active region is supplied with silane and hydrogen influences the electrical properties of the microcrystalline material. A sequence ranging from microcrystalline to amorphous silicon films has been deposited fixing the above mentioned parameters but varying the gas flow path through the reactor.

P8: Role of catalyst size in growth of small and large diameter carbon nanotubes via PECVD: Surface and cohesive energy

S. Gupta¹ (guptas@missouri.edu), Y. Y. Wang² and R. J. Nemanich³

¹ECE Department, University of Missouri-Columbia, MO 65211

²Department of Mechanical Engineering, University of Texas, Austin, TX 72711

³Department of Physics, Arizona State University, Tempe, AZ 85281

Transition metal catalysts (Fe and Ni) are essential in the nucleation and subsequent growth of carbon nanotubes. The size of these catalytic particles determines the diameter of the carbon nanotubes ranging from single- double, oligo- to multiwalled. It is well known that metal catalysis is the key for the formation of carbon nanotubes, but the exact role played by the catalytic particle size in the growth process and formation of all small to large diameter nanotubes remains to be understood. Vertically aligned high areal density small and large diameter carbon nanotube films were deposited by microwave chemical vapor deposition using Fe as a catalyst. Experiments show that by continuous reduction in Fe thickness to from 80 nm to 0.5 nm, strictly multi-walled to single-walled carbon nanotubes can be achieved. Scanning electron and high-resolution transmission electron microscopy show that the diameter of carbon nanotubes ranged between 0.96–1.2 nm and 40-50 nm. Resonance Raman spectroscopy was used to further verify the diameter of nanotubes. A thicker Fe film of ~80 nm resulted larger catalyst islands and multi-walled and bamboo-like structures. In this context, a growth mechanism of tubules formation is proposed that incorporates surface and bulk diffusion components forming walls and bamboo-like internal structure [Wang, Gupta *et. al.* APL **85**(2004)] pointing at the role of surface and cohesive energy in small diameter catalyst particles.

P9: Study on low oxygen content SiCN film deposited by Hot-wire chemical vapor deposition method

S.K. Hasan (Syed_hasan_04@yahoo.com), K. Matsuo, T. Ikuta, A. Izumi

Kyushu Institute of Technology

SiCN film can be deposited by HWCVD method using only Hexamethyldisilazane (HMDS). A large amount of oxygen contains in the SiCN film prepared by the condition. It is found that the amount of oxygen can be decreased by flowing NH₃ with HMDS. It is also found when the flow rate of HMDS is 1.5 sccm and that of NH₃ is 100 sccm, the amount of oxygen becomes the lowest that suggests the formation of low oxygen content SiCN film.

P10: Preparation of n-type nanocrystalline 3C-SiC films by hot-wire CVD using N₂ as doping gas

Yoshiki Hoshide (y_hoside@echo.nuee.nagoya-u.ac.jp), Akimori Tabata

Department of Electrical Engineering and Computer Science, Nagoya University, C3-1 (631), Chikusa, Nagoya, 464-8603, Japan

N-type nanocrystalline 3C-SiC films were prepared by hot-wire CVD from SiH₄/CH₄/H₂ and N₂ as a doping gas. The gas flow rates of SiH₄, CH₄ and H₂ were 1, 1 and 200 sccm, respectively. As the N₂ gas flow rate was increased from 0 to 10 sccm, the conductivity and the activation energy improved from 0.05 to 0.3 S/cm and from 45 to 28 meV, respectively. This results from an increase in the amount of N as a dopant. On the other hand, at the N₂ gas flow rate between 10 and 50 sccm, the conductivity and the activation energy remained unchanged. The crystallinity, estimated from the Si-C stretching mode, deteriorated with increasing the N₂ gas flow rate from 10 sccm. This gave rise to the unchangeable electronic properties in spite of the increase in the N₂ gas flow rate.

P11: Importance of H₂ gas for growth of hot-wire CVD nanocrystalline 3C-SiC from SiH₄/CH₄/H₂

Yoshiki Hoshide (y_hoside@echo.nuee.nagoya-u.ac.jp), Yusuke Komura^a, Akimori Tabata

Department of Electrical Engineering and Computer Science, Nagoya University, C3-1 (631), Chikusa, Nagoya, 464-8603, Japan

^a present address: Mitsubishi Electric Corporation

We prepared silicon carbide (SiC) thin films by hot-wire chemical vapor deposition from SiH₄/CH₄/H₂ and investigated the dependence of film properties on H₂ gas flow rate. The SiH₄ gas flow rate was 1 sccm. In the case of CH₄ gas flow rate ($F(\text{CH}_4)$) of 1 sccm, nanocrystalline cubic SiC (nc-3C-SiC) grew even without H₂. On the other hand, when $F(\text{CH}_4)=2$ sccm, amorphous SiC grew without H₂ and nc-3C-SiC grew above 50 sccm of H₂ gas flow rate. As the H₂ gas flow rate was increased, the crystallinity, estimated from Si-C stretching infrared peak, was improved and the mean crystalline size decreased at $F(\text{CH}_4)=1$ sccm and increased at $F(\text{CH}_4)=2$ sccm. We discuss a growth mechanism of nc-3C-SiC.

P12: Generation of Crystalline Silicon Nanoparticles during Hot Wire Chemical Vapor Deposition

Chan-Soo Kim (damulkim@snu.ac.kr), Woong-Kyu Youn, Yung-Bin Chung and Nong-Moon Hwang

National Research Laboratory of Charged Nanoparticles, Department of Material Science and Engineering, Seoul National University, Sillim-dong, Gwanak-gu, Seoul, 151-742, Korea

Although deposition of crystalline silicon films at low temperature below 400°C has been intensively studied using hot wire, the mechanism has not been clearly understood. As a mechanism of low temperature deposition of crystalline silicon, we suggest a two-step growth mechanism, where crystalline silicon nanoparticles are generated in the gas phase with their subsequent incorporation into a film during hot-wire chemical vapor deposition (HWCVD). The purpose of this study is to confirm experimentally the existence of such crystalline silicon nanoparticles in the gas phase during silicon HWCVD. In order to capture the hypothetical crystalline silicon nanoparticles formed in the gas phase, the hot-wire reactor was divided into three by two-stage orifices. The charged nanoparticles that were predicted to form in the first chamber of a hot wire reactor were extracted through the first and the second orifices into the third chamber, where the amorphous carbon membrane of the transmission electron microscopy (TEM) grid was placed for capturing these nanoparticles. The gas phase nuclei of crystalline silicon were shown to exist in the silicon HWCVD. The size of crystalline silicon nanoparticles decreases with decreasing SiH₄ concentration.

P13: The growth of GaN films by alternate source gas supply hot-mesh CVD method

Yasuaki Komae¹ (yasuaki@stn.nagaokaut.ac.jp), Takeshi Saitou¹, Maki Suemitsu², Takashi Ito², Tetsuro Endou³, Hideki Nakazawa⁴, Yuzuru Narita⁵, Kanji Yasui¹, Masasuke Takata¹, Tadashi Akahane¹

1 Nagaoka University of Technology, Nagaoka 940-2188, Japan

2 Center of Interdisciplinary Research, Tohoku University, Sendai 980-8578, Japan

3 Research Institute of Electrical Communication, Tohoku University, Sendai 980-8577, Japan

4 Faculty of Science & Technology, Hirosaki University, Hirosaki 036-8561, Japan

5 Faculty of Engineering, Yamagata University, Yonezawa 992-8510, Japan

GaN is a direct transition semiconductor with a wide bandgap, which is used for optoelectronic devices operating in short wavelength and at high temperatures. In our previous study, hot-mesh CVD method using catalytic reaction on a ruthenium (Ru) coated tungsten (W)-mesh was investigated for the growth of hexagonal GaN films on AlN/SiC/Si substrates. The GaN films grown using the Ru coated W-mesh of 1100 °C showed good crystallinity and a strong near-bandedge-emission without yellow luminescence derived from defect levels. An alternating source gas supply or a intermittent supply of one of the source gases during the film growth are expected to be effective for the suppression of gas phase reaction and for the migration enhancement of the precursors on the substrate surface. In order to further improve the film quality of GaN films, we investigated the hot-mesh CVD by alternate gas supply during the growth of GaN film and AlN buffer layer. The characteristics of the GaN films grown by some gas supply patterns, that is, alternating supply of trimethylgallium and ammonia gases, an intermittent supply of one of those gases, were compared. The improvement of the film quality, however, could not be confirmed by the alternating supply of the source gases and the intermittent gas supply during the GaN film growth.

P14: N₂ decomposition by hot wire and N₂ post-deposition treatment on hydrogenated microcrystalline silicon thin films

Koji Mazaki (k_mazaki@echo.nuee.nagoya-u.ac.jp) and **Akimori Tabata**

Department of Electrical Engineering and Computer Science, Nagoya University,
C3-1(631) Chikusa, Nagoya 464-8603, Japan

Hydrogenated microcrystalline silicon ($\mu\text{-Si:H}$) thin films were prepared by hot-wire chemical vapor deposition and the post-deposition treatment of the $\mu\text{-Si:H}$ was carried out in atmospheres of N₂, H₂ or N₂/H₂. For the $\mu\text{-Si:H}$ treated in N₂ at the filament temperature (T_f) of 1600 °C, the N-Si₃ (asymmetric) stretching (N-Si₃(st)) peak was observed. It increased drastically as the T_f was increased from 1900 to 2000 °C. These findings indicate that N₂ decomposition occurs at $T_f=1600$ °C and is facilitated at $T_f=2000$ °C. The N-Si₃(st) peak of the $\mu\text{-Si:H}$ treated in N₂/H₂ was lower than that in N₂ and was almost the same as that in H₂ even when the T_f was 2000 °C. This indicates that H₂ prevents N₂ decomposition by the heated filament.

P15: Properties of hydrogenated microcrystalline silicon thin films prepared by hot-wire CVD at high gas pressure conditions

Koji Mazaki (k_mazaki@echo.nuee.nagoya-u.ac.jp) and **Akimori Tabata**

Department of Electrical Engineering and Computer Science, Nagoya University,
C3-1(631) Chikusa, Nagoya 464-8603, Japan

Hydrogenated microcrystalline silicon ($\mu\text{-Si:H}$) thin films were prepared by hot-wire chemical vapor deposition at high gas pressures of 4-12 Torr. As the gas pressure was increased, the crystalline volume fraction increased and the full width at half maximum of Raman peak due to crystalline silicon decreased, although the mean crystallite size remained unchanged. These findings indicate that the increase in the gas pressure improves the crystallinity of the $\mu\text{-Si:H}$. The deposition rate decreased and the film structure became porous. These result from the enhancement of the etching of amorphous phase by H radicals.

P16: Towards the development of CIGSSe-based photovoltaic on flexible substrates by thermal chemical vapor deposition

Saibal Mitra (SaibalMitra@MissouriState.edu)

Department of Physics, Department of Astronomy and Materials Science Department,
Missouri State University, Springfield, MO 65804

CuIn_(1-x)Ga_x[S_(1-y)Se_y]₂ (CIGSSe) are attractive absorber material because its electronic bandgap can be engineered by controlling the stoichiometry of the material. Conversion efficiencies of ~20% have been achieved in thermally evaporated material. The challenge remains to grow high quality material with processing temperatures low enough so that photovoltaic cells can be fabricated on flexible plastic substrates.

In this paper we present results where we have deposited CuInS₂ thin films by thermal chemical vapor deposition (TCVD) using single source precursors like copper indium triethylene xanthate dissolved in methyl dichloride. The time of deposition was also varied from 30 mins. to several hours and temperatures ranging from a low of 150°C to a high of 500°C. Samples were deposited on a variety of substrates including indium tin oxides (ITO) and then characterized using secondary electron microscopy (SEM), x-ray diffraction (XRD) and so on. We report the growth of both mixed phase and primarily (112) oriented stoichiometric thin films. The results will be presented in detail.

P17: Low temperature growth of SiC on SOI substrate with ultrathin top Si layer by hot-mesh CVD method

Hitoshi Miura¹, Atsushi Masuda², Yuichiro Kuroki¹, Hiroshi Nishiyama³, Kanji Yasui¹ (kyasui@vos.nagaokaut.ac.jp), **Masasuke Takata¹, Yasunobu Inoue³ and Tadashi Akahane¹**

¹ Department of Electrical Engineering, Nagaoka University of Technology, 1603-1 Kamitomioka-cho, Nagaoka 940-2188, Japan

² National Institute of Advanced Industrial Science & Technology (AIST), Central 2, Umezono 1-1-1, Tsukuba, Ibaraki 305-8568, Japan

³ Analysis Center, Nagaoka University of Technology, 1603-1 Kamitomioka-cho, Nagaoka 940-2188, Japan

Silicon carbide (SiC) is a wide bandgap semiconductor that is expected to be used in electronic devices for high frequency, high power and high temperature applications. Among its many polytypes, cubic SiC that possesses a zincblende structure, can be grown at low temperatures and epitaxially grown on Si substrates. In recent years, Si-on-insulator (SOI) structures have been found to be suitable for fabricating high-speed, low-power electronic devices. The SiC-on-insulator (SiCOI) structures are also valuable for fabricating SiC devices and micro-electro-mechanical systems (MEMS) that exhibit the excellent characteristics in harsh environments [1].

In this study, the fabrication of the SiCOI structure was investigated using hot-mesh (HM-) CVD method, which utilizes the high-decomposition rate of the H₂ gas by heated tungsten wires arranged in a mesh. The SiC films were grown on SOI substrates with ultrathin top-Si layer less than 100nm in thickness. During the growth of SiC films, catalyzer temperature was maintained at 1600°C. The substrate temperature was set in the range from 700 to 800 °C. The SiC growth at substrate temperature of 750°C did not induce the formation of voids in the top-Si layer. And the SiC epitaxial films were successfully grown on the SOI substrates with top-Si layer less than 10nm in thickness. These results elucidate the effectiveness of the HMCVD method for the fabrication of the SiCOI structure substrates.

[1] M. Mehregany, C. A. Zorman, N. Rajan, and C. H. Wu, Proc. IEEE, **86**, No.8 (1998) 1594.

P18: Texturization of c-Si by anisotropic KOH etching for the fabrication of heterojunction solar cells by Hot-Wire CVD

D. Muñoz¹ (delfina.munoz@ub.edu), **P. Carreras¹, J. Escarré¹, D. Ibarz¹, J. M. Asensi¹, J. Bertomeu¹, C. Voz²**

¹XaRMAE -Universitat de Barcelona, Departament de Física Aplicada i Òptica, Martí i Franquès 1, Barcelona 08028, Spain

²Universitat Politècnica de Catalunya, Grup de Recerca en Micro i Nanotecnologies, Jordi Girona 1-3, Barcelona 08034, Spain

Solar cells based on amorphous/crystalline silicon (c-Si) heterojunctions have attracted lots of attention due to their high-efficiency and cost-effective fabrication process. Sanyo has reported efficiencies over 19% for mass produced solar cells with the so-called HIT structure. Although they fabricate the devices by Plasma-Enhanced Chemical Vapor Deposition, the Hot-Wire Chemical Vapor Deposition technique (HWCVD) has also demonstrated to be a good alternative.

During the last years, our group has obtained very promising results on heterojunction solar cells by HWCVD with efficiencies up to 15.4%. In this work, we focus on increasing the short-circuit current of the device by introducing textured substrates. Concretely, we have studied the texturization process of (100) c-Si wafers by using a KOH solution, a simple and scalable process to obtain good quality textured wafers suitable for heterojunction devices. The optimization of the etching process has allowed obtaining random but uniform pyramidal structures. Then, heterojunction emitters were deposited by HWCVD onto these substrates and characterized by the Quasi-Steady-State PhotoConductance technique. Preliminary results have evidenced only a moderate reduction in the passivation quality with a reduction around 25 mV in the implicit open-circuit voltage. Nevertheless, these are encouraging results for fabrication of heterojunction solar cells on textured substrates.

P19: Hot wire chemical vapor deposition: limits and opportunities of protecting the tungsten catalyzer from silicide with a cavity

P.A. Frigeri, O. Nos (oriol_nos@ub.edu), **S. Bengoechea, C. Frevert, J.M. Asensi, J. Bertomeu**

Dept. de Física Aplicada i Òptica, Universitat de Barcelona
Barcelona-08028, Spain, Ph: +34934039221, fax: +34934039219

Hot Wire Chemical Vapor Deposition (HW-CVD) is one of the most promising techniques for depositing the intrinsic microcrystalline silicon layer for the production of micro-morph solar cells. However, the silicide formation at the colder ends of the tungsten wire drastically reduces the lifetime of the catalyzer, limiting its industrial exploitation. A simple but interesting strategy to decrease the silicide formation is to hide the electrical contacts of the catalyzer in a narrow but long cavity, which reduces the probability of the silane molecules to reach the colder ends of the wire. In this paper, we use a simple statistical model based on the assumption of molecular flow to study the limits and the opportunities of this simple mechanism to preserve the catalyzer. Experimental results such as the wire lifetime of the wire and the thickness profile of the silicon deposited in the internal walls of the cavity are compared with those predicted by the model.

P20: Low temperature decomposition as large molecule of TMA using catalyzer with resistance to oxidation in Catalytic CVD

Yoh-Ichiro Ogita (ogita@ele.kanagawa-it.ac.jp), **Tsugutomo Kudoh**

Kanagawa Institute of Technology
1030 Shimo-Ogino, Atsugi, Kanagawa Japan 243-0292

Alumina films are needed as an insulator of semiconductor devices and a high integrity film for permeation of oxygen. Cat-CVD (Catalytic chemical vapor deposition) technique is expected to deposit high quality films due to non-plasma damage. One of the authors has tried to deposit an alumina film on a Si wafer using TMA (Tri-methyl aluminum) and O₂ using Ir and W catalyzers in Cat-CVD and showed that the film was high quality films with stoichiometry. However, Ir catalyzer is so expensive and W catalyzer is short lifetime due to the oxidation. In this paper, we examined the catalytic decomposition of TMA for various metal catalyzers such as Mo, Ni, Kanthal, Ni-Chrom, Chromel, Inconel 600, SUS 304, and Al Chrom-O wires, of which resistance to oxidation had been confirmed. The catalytic decomposition was identified by QMS (quadrupole mass spectrometer). We have found that Chromel, SUS 304, and Alchrom-O catalyzers decomposed TMA to Al and CH_x at 200°C lower by 300°C compared to Ir and W catalyzers. In addition, CH_x was not decomposed into smaller molecules and atoms, which implies that these catalyzers make it possible to create alumina films not included carbon atoms, which means very low leakage current in the alumina films.

P21: Growth and properties of amorphous SiCN grown by organic Cat-CVD

Asako Oka¹, Hiromichi Ito¹ and Hiroshi Nakayama^{1,2} (mdf@md-factory.com)

¹Material Design Factory Co., Ltd.

²Graduate School of Engineering, Osaka City University

SiCN is known as an insulating and mechanical ceramics and therefore the previous studies were devoted to the mechanical properties, which are comparable to those of diamond, cubic BN and CN. Present work is the first study which is concentrated on the physical properties and the diffusion barrier characteristic of SiCN. In this work, SiCN amorphous alloy films were prepared using IMS/NH₃ gas mixture. The compositions of SiCN were controlled by the gas-flow ratio. At the low gas-flow ratio, NH₃/IMS, the concentration ratios, N/Si and C/Si, increase with increasing the gas-flow ratio, NH₃/IMS. Beyond the gas-flow ratio, NH₃/IMS, 20, the C content decreases rapidly. However the content ratio, N/Si, saturates at around 0.65. The value, (N/Si)+(C/Si), is about 0.9-1.0, which is smaller than N/Si=1.33 in Si₃N₄ compounds. The refractive index is analyzed to be about 2.06 and the extinction coefficient is about 0.0005 in the visible light range. On the basis of these features of SiNC film, we have developed H₂O/O₂ permeation barrier film for flexible organic LED. A tri-layer, with the total thickness of 300 nm, of O-Cat-CVD grown SiNC sandwiched by the Si-doped hybrid polymer grown on PEN film showed the H₂O permeation rate less than 0.01 g/m²/day.

P22: Kinetic Studies of Initiated Chemical Vapor Deposition of Vinyl Monomers and Divinyl Crosslinkers

Gozde Ozaydin-Ince (gozde@mit.edu), Karen K. Gleason

Department of Chemical Engineering, Massachusetts Institute of Technology, Cambridge, MA 02139, USA

In this work, systematic kinetic studies of the initiated chemical vapor deposition of vinyl monomers and divinyl crosslinkers, using tert-butyl peroxide as the initiator, are performed and the effects of process parameters on deposition are investigated.

For gas phase FTIR (Induct 3100 Process Analyzer from MKS) measurements, concentrations are calculated by calibrating the carbonyl peaks of the monomers with known concentration values in ppm. Detection limit for the vinyl monomers is approximately .1 ppm.

Residence time studies are performed at different reactor pressures and an optimal operating pressure of approximately 200 mTorr for a plug flow is determined. Deposition rates obtained at this operating pressure range from 5 nm/min to 70 nm/min for different process parameters.

Studies performed at different filament temperatures, ranging from 180°C to 360°C, clearly show the kinetic and mass transfer limited regimes from which the apparent activation energies are extracted. The transition to a mass transfer limited regime is typically observed in the range of 230°C to 270°C. The separate studies of deposition rate dependence on the initiator and the monomer concentrations enable us to determine the reaction rate constant and the order of reaction.

In the final set of experiments, the effects of substrate temperature variations, from 15°C to 45°C, on the deposition rate are studied and the adsorption kinetics are discussed.

Keywords: Initiated chemical vapor deposition, Silicon, polymer thin film, gas phase FTIR

P23: Post deposition annealing temperature effects on Si quantum dots embedded in Silicon nitride matrix prepared by HWCVD

A.K. Panchal¹, P. Narwnakar², S.S. Major³, C.S. Solanki⁴ (chetanss@iitb.ac.in)

^{1,4} Dept. Energy Science & Engineering, IIT Bombay, Powai, Mumbai 400076, India

³ Physics Dept., IIT Bombay, Powai, Mumbai 400076, India

² Applied Materials (USA), Bangalore, India

The optical and electrical properties of Si can be changed by controlling its band gap. Si band gap can be increased than bulk material by reducing Si size smaller than its Bohr's exciton diameter (4.9 nm) due to Si quantum confinement effects.

Si quantization effects in form of ultra thin superlattices for solar cell applications were used since early 80s. Boron doped a-Si embedded in SiC and SiN, and Boron doped SiC embedded in SiN superlattices were used as p-layer in p-i-n structure of a solar cell. More recently, use of a phosphorous doped Si-QD/SiO₂ multilayer in p-n junction is reported. It has also been proposed to use different size of Si-QD embedded in Si compounds (SiO₂, Si₃N₄ and SiC) matrix for multi-junction solar cell.

The effect of post deposition annealing on the properties of Si-QD in SiN multilayer prepared in a single SiN chamber of HWCVD is presented in this paper. The thin film prepared by HWCVD offers several advantages over conventional PECVD; (i) high deposition rate, (ii) 80% SiH₄ usage, and (iii) deposition uniformity over large area.

Twenty period alternate a-Si and SiN layers (each having thickness 10 nm) was prepared by cracking SiH₄ and SiH₄+NH₃ respectively. The as-deposited samples were post annealed in range of 800-950°C. The as-deposited and annealed samples were analyzed by FTIR, Micro RAMAN and AFM techniques. Fig.1 shows the Raman spectra of the as deposited (top) and annealed samples (bottom). The as-deposited sample has Raman line at 475 cm⁻¹, showing the presence of amorphous phase of Si. Lower frequency asymmetry in the Raman spectra of annealed sample shows the presence of crystalline Si-QD. The Analysis shows the Si-QD sizes in the range of 2 to 5 nm. Details will be discussed in the full length paper.

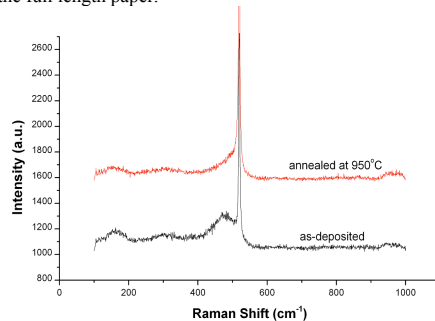


Fig.1 Micro Raman spectra of Si-QD/SiN multilayer prepared by HWCVD (a) as deposited and (b) post deposition annealed film at 950°C

P24: Effect of pressure on the structure of silicon nanoparticles synthesized by HWCVD

M.R. Scriba^{1,2} (mrscriba@csir.co.za), M. Härting² and D.T. Britton²

¹ CSIR, National Centre for Nano-Structured Materials, P O Box 395, Pretoria 0001 South Africa.

² Dept. of Physics, University of Cape Town, Rondebosch 7701 South Africa

The effect of pressure and wire temperature on the internal structure of silicon nanoparticles, synthesized by HWCVD is investigated. A light brown powder is produced in a stainless steel reactor, from 100% silane at pressures between 2 and 500 mbar with a tungsten filament, at temperatures between 1500 and 1900 °C and a flow rate of 50 sccm. Rutherford Backscatter Spectroscopy on the as produced powder confirms the composition of the particles as intrinsic silicon and using scanning electron microscopy the average particle size is measured to be below 100nm. The internal structure of the silicon nanoparticles, as analyzed by X-ray diffraction and Raman spectroscopy, and supported by high resolution transmission electron microscopy, clearly shows an increase in order with rising synthesis pressure.

P25: Small-Angle X-ray Scattering from nano-Si embedded a-SiC:H deposited by hot-wire chemical vapor deposition: correlation with microstructure and the fractal regimes

Bibhu P. Swain (swain@snu.ac.kr), Nong M. Hwang

National Research Laboratory of Charged Nanoparticles
School of Materials Science and Engineering
Seoul National University, Seoul South Korea
Tel: 8228809152, Fax: 8228809152

Nano-Si embedded a-SiC:H films were deposited by hot wire chemical vapor deposition (HWCVD) using SiH₄, CH₄ and H₂ gas precursors. Small-angle X-ray scattering (SAXS) and Raman analysis of a-SiC:H films indicates scattering from mass fractal aggregates of amorphous and nanocrystalline domain of nano-Si. We show that the derivative of I(q) vs q SAXS data demonstrates a crossover point q_c between surface scattering by primary particles and scattering by mass fractal aggregates. This crossover point is related to the mean diameter and the size distribution of the nano-Si.

P26: Plasmon loss and valence band structure of silicon-based alloys deposited by hotwire chemical vapor deposition (HWCVD)

Bibhu P. Swain* (swain@snu.ac.kr), **Sung H. Park, Nong M. Hwang**

National Research Laboratory of Charged Nanoparticles
School of Materials Science and Engineering
Seoul National University, Seoul, South Korea
Tel: 8228809152, Fax-822889152

X-ray photoelectron spectroscopy (XPS), Raman and XRD of amorphous silicon alloys (a-Si:H, a-SiC:H and a-SiN:H) and nc-Si embedded silicon alloys (a-Si:H, a-SiC:H and a-SiN:H) were deposited by hot wire chemical deposition (HWCVD). After determining alloy contributions to the plasmon loss at the surface, we observed one plasmon loss peak for amorphous films and two peaks for nc-Si embedded silicon alloys films. This analysis has been applied to a Si(2p) spectrum, giving a total intrinsic plasmon contribution of 18-26 % relative to the main peak intensity. Full Width at Half Maxima (FWHM) of valence band of alloy increases with increase of carbon and nitrogen content.

P27: Effect of H₂ dilution on a-CN: H films deposited by hot-wire chemical vapor deposition

Bibhu P. Swain (swain@snu.ac.kr), **Young-Bin Chung, Nong M. Hwang**

National Research Laboratory of Charged Nanoparticles
School of Materials Science and Engineering
Seoul National University, South Korea
Tel: 8228809152, Fax. 8228809152

Hydrogenated amorphous carbon nitride (a-CN:H) thin films were deposited by hot filament chemical vapor deposition (HWCVD) using the gas mixture of CH₄, NH₃ and H₂. The effects of the H₂ dilution on structural and electronic environments of carbon have been discussed by Raman and XPS spectroscopy. Full width at half maximum (FWHM) of C(1s) peak decreases and peak position shifts to high binding energy with increase of H₂ dilution. With increasing H₂ flow rate, 2p orbital intensity decreases and sp and 2s orbital intensity increase. The composition ratio of sp² to sp³ carbon increases from 1.47 to 1.8 with increasing H₂ flow rate, although the nitrogen content in the films is almost the same as 0.1.

P28: Growth of silicon carbide thin films by hot-wire chemical vapor deposition from SiH₄/CH₄/H₂

Akimori Tabata (tabata@nuee.nagoya-u.ac.jp), **Yusuke Komura***

Department of Electrical Engineering and Computer Science, Nagoya University
C3-1(631), Chikusa, Nagoya 46408693, Japan

* present address: Mitsubishi Electric Corporation

We prepared silicon carbide (SiC) thin films by hot-wire chemical vapor deposition from SiH₄/CH₄/H₂. In the case of 2 Torr, Si-crystallite-embedded amorphous SiC (a-SiC:H) grew at filament temperatures (T_f) below 1600 °C and nanocrystalline cubic SiC (nc-3C-SiC) did above T_f =1700 °C. On the other hand, in the case of 4 Torr, a-SiC:H grew at T_f =1400 °C and nc-3C-SiC did above T_f =1600 °C. When the amounts of Si and C atoms incorporated into film per unit time are almost the same and H radicals with a high density are created, which takes place at high T_f , nc-3C-SiC grows. On the other hand, at low T_f the amount of Si atoms incorporated is larger than that of C atoms and, consequently, a-SiC:H or Si-crystallite-embedded a-SiC:H grow, which is dependent on the amount of generated H radicals.

P29: Development of Hot-Wire Chemical Vapor Deposition (HWCVD) System and Radical Treatment System

Tadashi Takahashi (toyko@universalsystems.co.jp)

Universal Systems Co., Ltd.
4-17-23 Hiyoshi-Cho,
Kokubunji-City,
Tokyo 185-0032 Japan
Tel: +81-42-322-8726
Fax: +81-42-322-8736

Compact and reasonable SiCN-deposition system has been developed. In this work, we will demonstrate not only result of SiCN but also powder treatment using H₂ or NH₃.

P30: Properties of high-quality doped and undoped epitaxial silicon by hot-wire chemical vapor deposition

Charles W. Teplin, Ina T. Martin (Ina_Martin@nrel.gov), **David L. Young, Kim M. Jones, Manuel J. Romero, Robert C. Reedy, Paul Stradins and Howard M. Branz**

National Center for Photovoltaics, National Renewable Energy Laboratory, Golden, CO 80401

We describe growth and characterization of n- and p-type epitaxial layers deposited by hot-wire chemical vapor deposition (HWCVD) over a wide range of doping densities and report optoelectronic properties of the films. We are optimizing silicon epitaxy growth for thin crystal silicon solar cell applications. Epitaxy on highly crystalline Si seed layers on low-cost substrates such as borosilicate glass will require scalable growth below about 700°C. We have grown epitaxial films on (100) silicon wafers over 10 μm thick and have demonstrated growth rates over 200 nm/min by HWCVD from SiH₄ gas. Dislocation densities $\leq 10^5$ cm⁻² have been achieved by optimizing surface preparation. Measurements include carrier density, lifetime, Hall mobility and secondary ion mass spectrometry of dopant and impurity densities. A phosphorus-doped film with an electron density of 7.8×10^{16} cm⁻³ has a Hall mobility approaching the impurity-scattering limit of 786 cm²/V-s.

P31: Hot-Wire Assisted Synthesis and Post Process Modification of Metal Oxide Nanowires

J. Thangala, V. Chakrapani, Z. Q. Chen,^a S. Vaddiraju,^b S. Malhotra and M. K. Sunkara* (mahendra@louisville.edu)

Department of Chemical Engineering, University of Louisville
Louisville, KY 40245

Present addresses: ^aMonsanto Corporation, St. Louis, MO.
^bMassachusetts Institute of Technology, Cambridge, MA.

ABSTRACT

Highly crystalline one dimensional metal oxide nanowire arrays are of great interest due to their high surface area and fast charge transport characteristics. Such nanowire arrays are of significant interest for applications like electrochromic devices, solar cells, photoelectrochemical water splitting and field emission devices. These advantages motivated us to develop a synthesis concept for making nanowire arrays over large areas and on different substrates.

A novel and versatile method for the large-scale synthesis of metal oxide nanowire arrays and mat-like thin films on different substrates such as amorphous quartz, fluorinated tin oxide (FTO), tungsten and titanium will be presented. The synthesis concept involves chemical vapor transport of metal oxide vapor phase species using air or oxygen flow over hot filaments onto substrates kept at a distance.^{1,2} A thermodynamic model showing the species responsible for the nucleation and subsequent growth of the nanowires will be presented. Also, our recent experiments on doping of the NW arrays with different elements such as nitrogen and other metals during synthesis show promise. In addition, post process doping of metal oxide nanowire arrays using different gas phase sources will be presented.

The post process modification of metal oxide nanowire arrays with exposure to different gases resulted in complete transformation to the respective nitride and metal NW arrays.³ The crystallinity and the morphology of the nanowires is maintained after the transformation process. These results with post-process modification suggest that one can use the hot-wire assisted process to make oxide NW arrays and transform them to their respective metal, metal nitrides, sulfides and carbides.

Electrochromic devices made with the tungsten trioxide nanowire array substrates showed a highly reversible transmission modulation of over 70% with almost 0% transmission in the colored state, at a wavelength of 700 nm.⁴ Gas sensing measurements using tungsten trioxide nanowire networked films showed two orders of magnitude higher response when exposed to N₂O gas.⁵

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P32: Mechanism of Gas-phase Reactions with 1,1,3,3-tetramethyl-1,3-disilacyclobutane in Hot-wire Chemical Vapor Deposition Process

L. Tong and Y.J. Shi (shiy@ucalgary.ca)

Department of Chemistry, University of Calgary, Calgary, Alberta, Canada

Semiconductor thin film growth using hot wire chemical vapor deposition (HWCVD) is induced by precursors produced from primary decomposition and secondary gas-phase reactions in the reactor. To understand the gas-phase reaction chemistry in HWCVD process when using 1,1,3,3-tetramethyl-1,3-disilacyclobutane (TMDSCB) as a source gas for silicon carbide film growth, the reaction products produced from the HWCVD reactor were investigated using vacuum ultraviolet laser single-photon ionization coupled with time-of-flight mass spectrometry. It was found that TMDSCB decomposes on the tungsten filament to methyl and 1,1,3-trimethyl-1,3-disilacyclobutane radicals. Subsequent H abstraction reactions from the parent molecule by methyl radicals, and biradical combination reactions result in the formation of alkyl-substituted disilacyclobutanes ($m/z = 158, 172, 186$) and silyl-substituted alkanes ($m/z = 272, 286$). The formation of dimethylsilene ($m/z = 72$) was indirectly confirmed by the observation of its trimer, 1,1,3,3,5,5-hexamethyl-1,3,5-trisilacyclohexane ($m/z = 216$). The observation of 1,3-dimethyl-1,3-disilacyclobutane ($m/z = 116$) indicates that dimethylsilylene was also produced from HWCVD, which isomerized to methylsilene through 1,2-H migration. The decomposition and gas-phase reactions of TMDSCB in HWCVD produces reactive silene and silylene intermediates, which contain direct Si-C bond connection, therefore TMDSCB is expected to be a potentially useful source gas for SiC thin film deposition.

P33: Comparison of SiN_x deposition by HWCVD and PECVD in terms of relative atomic densities and their influence on microstructure and optical properties

V. Verlaan (a) (V.Verlaan@UU.nl), W.M. Arnoldbik (a), C.H.M. van der Werf (a), R. Bakker (a), Z.S. Houweling (a), A.D. Verkerk (a), I.G. Romijn (b), D.M. Borsa (b), A.W. Weeber (b), S.L. Luxembourg (c), H.F.W. Dekkers (d), R.E.I. Schropp (a)

- (a) Utrecht University, Faculty of Science, Debye Institute for Nanomaterials Science, SID – Physics of Devices
- (b) ECN Solar Energy, Petten, the Netherlands
- (c) Technical University of Delft, the Netherlands
- (d) IMEC vzw, Leuven, Belgium

SiN_x thin films have many commercial applications and a large variety of deposition techniques are available. We have compared the composition, microstructure and optical properties of SiN_x films deposited using RF PECVD, MW PECVD and LF PECVD with those of HWCVD SiN_x. The composition of these SiN_x films obtained by the four different deposition techniques is characterised by RBS, ERD, and FTIR. Reflection/transmission or ellipsometry measurements are applied to obtain the optical properties. The obtained composition is studied in combination with the infrared Si-H absorption peak and shape using an extended *chemical induction mode*[1], which provides detailed information about the microstructure. The atomic densities are correlated to the polarizability and refractive index using the *Lorentz-Lorenz* equation.

Despite the fundamental difference between the deposition methods, the bulk properties of the films appear to be determined by the atomic densities in the films only. Since the relative atomic density does differ for each deposition method, this can cause significant differences in bulk properties and thus device performance. Especially the N volume density was found to have a large influence on the bulk properties of the as-deposited SiN_x films. The variations in atom densities are discussed in relation with the specific deposition mechanisms of each method.

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P34: Surface roughness and morphology analyses of boron-doped diamond films using power spectral density and autocorrelation approaches

G. P. Vierkant¹ and S. Gupta³ (guptas@missouri.edu)

¹Alcatel-Lucent Technologies, Murray Hill, NJ 07974

²Institute for Materials Research, Hasselt University and IMEC, Wetenschapspark 1, B-3590 Diepenbeek, Belgium

³ECE Department, University of Missouri-Columbia, MO 65211

The surface roughness evolution of growing thin films provides much information about their growth mechanism. While some systems show stages of nucleation, coalescence and growth, the others exhibit varying microstructures with process conditions. In view of this classification, we report detailed analyses from atomic force microscopy (AFM) characterization to extract surface roughness and growth exponents for boron-doped diamond (BDD) films by utilizing Power Spectral Density (PSD) and Autocorrelation Function (ACF) mathematical tools. The machining industry has applied PSD for the tool design, wear and machined surface quality. We present similar analysis at nanoscale to study the surface morphology of microwave chemical vapor deposited BDD films. We found that films with relatively higher boron yielded higher amplitudes in the longer wavelength power spectral lines with amplitudes decreasing in an exponential fashion towards the shorter wavelengths determining correlation length. The ACF function was applied to 1D (x- and y- axes) AFM topography data that identify any spatial periodicity *via* repetitive ACF peaks. Periodicity at shorter spatial wavelengths was observed for undoped and low doping levels, while small correlation was observed for the higher concentration. These quantitative spatial analyses may prove useful in comparing synthesis techniques and films with varying compositional makeup with an insight into the growth kinetics in an inverse manner.

P35: HFCVD Coated Graphite and its Application as Electrode in Aluminum Electrolysis

Shanshan Wang^{a,b} (kesalina@ust.hk), Guohua Chen^a (kechengh@ust.hk) and Fenglin Yang^b

a) Department of Chemical Engineering, The Hong Kong University of Science and Technology, Clear Water Bay, Kowloon, Hong Kong, China

b) School of Life Science and Environmental Engineering, Dalian University of Technology, Dalian, China

The Hall-Heroult process of aluminum production is featured by the release of CO₂ as a result of the sacrificing graphite anode. Finding a proper inert anode active in oxygen evolution has been a research topic for electrochemists to have a green process of aluminum electrolysis.

A thin layer of diamond coating of the existing graphite anodes has shown some promise. If this electrode works, it would have minimum disturbance of the current practice. The challenges for the project are twofold, fabricating stable and active diamond-graphite composite electrode, and controlling proper electrolysis condition so as not to degrade diamond thin film. In this paper, we are going to report the recent findings from our lab dealing with both challenges.

P36: Transformation of HWCVD silicon films by laser-induced crystallization

Dong-Sing Wu^a (dsw@dragon.nchu.edu.tw), Bing-Rui Wu^a, Kuan-Chieh Huang^a, Chieh-Chia Chang^b, Hsin-Yuan Mao^a, Ray-Hua Horng^c, Chung-Yuan Kung^b

^a Department of Materials Science and Engineering

^b Graduate Institute of Optoelectronic Engineering

^c Institute of Precision Engineering

National Chung Hsing University, 250 Kuo Kuang Road, Taichung 40227, Taiwan

Hot-wire chemical vapor deposition is a promising method to deposit polycrystalline silicon at a high deposition rate, low substrate temperature and low hydrogen content. In this paper, low hydrogen content (< 2 at.%) silicon film was deposited as precursor films for the laser-induced crystallization process. Silicon films with low hydrogen content can be used for laser treatment without a dehydrogenation step. Laser-induced crystallization was performed using a Q-switched Nd-YAG laser with a wavelength of 355 nm (10 Hz). Structural analyses of silicon films after laser annealing were carried out by X-ray diffraction measurement, Raman scattering spectroscopy and transmission electron microscopy. After the laser-induced crystallization process, the grain size of silicon film with 100 nm-thick was grown larger than 100 nm. The Raman spectrum confirms that the present laser annealing process can further enhance the crystallinity of the silicon films. The Hall electron mobility values are observed to begin at ~1 cm²/V-sec for the as-deposited, increasing to >100 cm²/V-sec after laser treatment. This is very encouraging result for future fabrication of low temperature poly-silicon films in all kinds of applications.

P37: Back contacted silicon heterojunction solar cells prepared by HWCVD

Bing-Rui Wu^a, Dong-Sing Wu^a(dsw@dragon.nchu.edu.tw), Kuan-Chieh Huang^a, Hsin-Yuan Mao^a, Ray-Hua Horng^b

^a Department of Materials Science and Engineering

^b Institute of Precision Engineering

National Chung Hsing University, 250 Kuo Kuang Road, Taichung 40227, Taiwan

Interdigitated back contacted microcrystalline/monocrystalline silicon heterojunction solar cells were prepared by hot-wire chemical vapor deposition technique. This cell structure eliminates the front contact shading in conventional cells by interdigitating both contacts on the back side of the cell. In our study, the interdigitated p/n microcrystalline silicon films were deposited on the rear side of monocrystalline silicon wafers with thickness of 250 μm . The n- and p-type $\mu\text{c-Si}$ films deposited with different hydrogen dilution ratios from 0 to 0.9 at substrate temperature T_s of 250°C and filament temperature T_f of 1700°C on the working pressure P of 100 mtorr were discussed to obtain the optimum manufacture condition. The crystallinity of the p/n layer was improved with increasing the hydrogen dilution ratio due to the hydrogen-induced crystallization. Textured front surface was passivated by intrinsic amorphous silicon film of 20 nm thick and a silicon nitride anti-reflection film of 80 nm were deposited by plasma-enhanced chemical vapor deposition at 200°C. The experimental I-V characteristic has been measured at room temperature, 100 mW/cm² light source with AM 1.5-like spectrum. Typical efficiencies greater than 10% can be achieved for these back contacted silicon heterojunction solar cells.

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