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Physics Procedia 46 (2013) 3 – 11

Physics

Procedia

Nineteenth European Conference on Chemical Vapor Deposition, (EUROCV D 19)

High power pulsed plasma enhanced chemical vapor deposition: a brief overview of general concepts and early results

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Abstract

The general concepts of the emerging plasma enhanced chemical vapor deposition (PECVD) technique High Power Pulsed PECVD (HiPP-PECVD) are outlined; the main feature of HiPP-PECVD is the use of a power scheme characterized by high power pulses with a duty cycle of a few percent or less to generate a process plasma with a significantly higher plasma density compared to traditional PECVD. The higher plasma density leads to a more reactive plasma chemistry, which results in a higher rate of dissociation of the precursor molecules, i.e. a more efficient use of the source material. The high plasma density also leads to a higher degree of ionization of the growth species, enabling the possibility to guide the growth species to the substrate or applying an energetic bombardment of the growing film by applying a substrate bias. Early results on HiPP-PECVD have shown that HiPP-PECVD enables deposition of phase pure α -Al₂O₃ at substrate temperatures as low as 560 °C with mechanical properties comparable to standard thermal CVD grown material. Also, deposition of amorphous, copper containing carbon films at deposition rates higher than 30 μ m/h has been demonstrated together with results showing the more efficient plasma chemistry. It is suggested that HiPP-PECVD is a promising tool for low temperature deposition of films with tailored properties for e.g. the hard coatings industry.

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Selection and peer-review under responsibility of Organizing Committee of EUROCV D 19.

Keywords: PECVD; Pulsed plasma discharges; Substrate bias; Al₂O₃; Amorphous Carbon, HiPIMS

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1. Introduction

When a chemical vapor deposition (CVD) process is designed to use the energy from a plasma discharge, rather than thermal energy supplied by high process temperature, to activate the gas phase chemistry, it is denoted Plasma Enhanced CVD (PECVD) or alternatively Plasma Activated CVD (PACVD). It should be mentioned that also the name Plasma Induced CVD (PICVD) has been used, Veprek, 1985, but is very rarely seen in the literature today. In a PECVD process, the gas phase chemistry, i.e. the cracking of the precursor gases and subsequent gas phase reactions are mediated by energetic species in the plasma, predominantly through electron impact collisions, Hess and Graves, 1993, at a low overall process temperature. This means that precursors with low reactivity can be used together with low substrate temperatures during deposition, enabling deposition on temperature sensitive substrates. A PECVD system is classified as a direct plasma system, when the substrate is placed between the discharge electrodes (anode/cathode) and thus directly immersed in the plasma. A remote system is when the substrate is placed at some distance away from the zone of plasma generation, i.e. not directly subjected to the plasma discharge generated between the anode and cathode. Remote PECVD is generally regarded to be a more gentle technique, given the lower degree of particle bombardment, Alexandrov and Hitchman, 2009. This is also the configuration most frequently used when plasma activation of the precursor chemistry is employed in the type of CVD known as atomic layer deposition (ALD), Profijt et al., 2011.

The most common means of igniting a plasma in PECVD are by discharges between two capacitively coupled parallel plates or by inductive coupling between the plasma gas and an applied RF field, Martinu et al., 2010. Typical plasmas used in PECVD are only weakly ionized with electron densities in the order of 10^{14} - 10^{18} m^{-3} , Hess and Graves, 1993, and thus the collision reactions between precursor molecules and energetic species in the plasma, e.g. electrons, ions and excited atoms/molecular fragments have a rather low probability.

During the last 15-20 years, an adjacent field within plasma based thin film deposition techniques, ionized physical vapor deposition (iPVD), has grown rapidly, Helmersson et al., 2006. Especially by the introduction of the high power impulse magnetron sputtering (HiPIMS) technique, Kouznetsov et al., 1999, also known as high power pulsed magnetron sputtering (HPPMS), which uses high-power pulses at a low duty factor ($< 10\%$) and low frequency (< 10 kHz) leading to peak cathode power densities of the order of several kW cm^{-2} , which has been shown to generate a dense and highly ionized plasma Lundin and Sarakinos, 2012, and Sarakinos et al. 2010. The plasma in a HiPIMS process typical has an electron density in the range 10^{18} - 10^{19} m^{-3} . This leads to a strong increase of dissociative collisions between the energetic electrons in the plasma and the neutral precursor gas molecules resulting in highly reactive species known as radicals. Furthermore, the development of commercially available power supplies for HiPIMS, Gudmunsson et al. 2012, has opened up the possibility to use high power pulsed HiPIMS-like plasma discharges also in PECVD processes, which is the focus of the present work.

In this brief overview the general concepts of high power pulsed PECVD (HiPP-PECVD) are presented along with selected, early results obtained by different research groups. Furthermore, an outlook for HiPP-PECVD processes will also be presented.

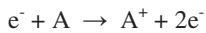
2. General concepts

2.1. High-power pulsed plasmas

There are a number of different pulsed power supplies that have been developed to generate HiPP discharges for HiPIMS. Currently there are several commercially available HiPIMS power supplies capable of

driving large area cathodes. The design of a HiPP power supply for a HiPIMS have previously been discussed by Christie et al., 2004, Sproul et al., 2004 and Kouznetsov, 2001. Early HiPIMS power supplies were built around an LC circuit switched by a thyristor. The control over the pulse and the pulse width was in this case limited. Modern HiPP power supplies are commonly based on a higher capacitance discharge giving an essentially square voltage pulse. The pulse is typically controlled by an IGBT switch with a pulse length set by the user.

The motivation for using a power scheme characterized by very high peak power densities ($\sim \text{kW cm}^{-2}$) delivered in short pulses (tens of microseconds) is the higher plasma density obtained leading to a dramatic increase in the degree of ionization of the species present in the plasma volume. Such pulsed plasma discharges can easily reach plasma densities, n_e , that are 2-3 orders of magnitude higher compared to for example direct current (DC) driven discharges, Lundin and Sarakinos, 2012, which effectively increases the probability of ionization of the deposition flux, through mainly electron impact ionization:



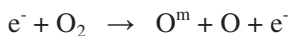
where e^- is an electron and A a neutral molecule or atom. As an example: for an electron density around 10^{19} m^{-3} commonly achieved in high-power pulsed plasmas, the ionization mean free path of a neutral atom is about 1 cm, whereas for an electron density of 10^{17} m^{-3} , as observed in a DC discharge, the ionization mean free path is 50 cm, Lundin and Sarakinos, 2012.

It should be noted that it is important to keep a low pulse duty factor so that the average applied power is kept at a level equivalent to DC discharge operation to allow for the cooling system to keep the cathode temperature below the melting point.

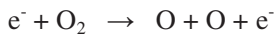
2.2. More reactive plasma chemistry

The film deposition chemistry in PECVD involves both gas phase and surface reaction. Often a surface, such as the surface of the growing film, is involved in order to conserve energy and momentum, as well as to provide longer interaction time between the reactants. In the case of N_2 (bond energy 9.8 eV) impact dissociation occurs when an energetic N_2^+ collides with a surface, which implies that electron impact ionization of N_2 in the plasma first must occur. Again, the concept of increasing the plasma density as described above will help to promote the required ionization, and ultimately a more efficient use of the precursor gas(es).

It should, however, be noted that a surface is not always required for dissociation of the precursor gas(es). Also electron-driven processes in the bulk plasma volume may play an important role depending on the probability for such reactions to occur. One example, which further highlights the importance of a high n_e , is electron impact dissociation of oxygen:



or



which occurs during the discharge pulse (high n_e), but not in between pulses (low n_e). Here O^m denotes an excited metastable oxygen atom.

2.3. Deposition from ionized species

The increase in ionized species through HiPP operation is also beneficial for the thin film growth process, since ions, unlike neutrals, can be guided towards the substrate by electric or magnetic fields, e.g. by a negative substrate bias. This can be used to enhance uniformity in film thickness when depositing films on complex shapes e.g. cutting tools and can also lead to a different surface chemistry in the process where ion bombardment can facilitate surface diffusion and desorption of byproducts. Ion bombardment has previously been shown to promote crystalline films over amorphous films, Carlson and Smith, 1982, preferred orientation, Greene and Barnett, 1982, and epitaxial growth, Pande and Seabaugh, 1984. Furthermore, by adjusting the substrate bias it is possible to tune the ion energy of the bombarding ions to provide significant amounts of energy to the surface. An energetic ion bombardment is regarded to be highly important to maintain the sp^3 -hybridization in deposition of sp^3 -hybridized films like cubic boron nitride, Konyashin et al., 1997, and diamond like carbon, Robertsson, 2008.

3. Early results

One of the few reports on HiPP-PECVD describes deposition of phase pure α -alumina at a deposition temperature as low as 560 °C using a parallel plate PECVD reactor, described in detail in Konstantinidis et al., 2011, equipped with a power supply capable of delivering unipolar voltage pulses up to 1.4 kV in 80-100 μ s long pulses at 5 kHz and a gas mixture of 82.6 % H_2 , 14.8 % Ar, 1.4 % O_2 and 1.2 % $AlCl_3$ at 175 Pa, Jiang et al., 2010. The plasma discharge voltage pulses were shown to have a direct effect on the crystal phase purity of the deposited alumina; at low discharge voltages of 900 V, a mixed phase of α and γ alumina was deposited. As the discharge voltage was increased, the intensity of the XRD peaks associated to the γ phase decreased and phase pure α -alumina was obtained by increasing the discharge voltage to 1.3 kV. Also the pulse length was found to have a small effect on the reduction of γ phase alumina; 100 μ s pulses yielded less γ phase compared to 80 μ s pulses.

The perhaps most encouraging result for the study, is that despite the low deposition temperature, the deposited films were shown to have similar elastic modulus and hardness as α -alumina deposited by “standard” thermal CVD at 1000 °C. These properties were explained by lower chlorine incorporation, leading to less porosity in the films. The lower chlorine incorporation was attributed both to a better cracking efficiency of the $AlCl_3$ precursor by the more reactive plasma chemistry obtained by the high power pulses, and to a more efficient surface chemistry due to increased energetic ion bombardment of the film during deposition, promoting chlorine desorption from the film surface. It was further estimated that as the discharge voltage, i.e. the cathode potential, increased from 0.9 kV to 1.4 kV, the average ion energy increased from about 140 to about 200 eV, leading to a more energetic ion bombardment during film growth.

Another approach to HiPP-PECVD was recently reported in Pedersen et al., 2012, where a hollow cathode discharge was used as plasma source. The hollow cathode approach has previously been utilized in PECVD processes for the deposition of amorphous carbon films and the process has been studied both by experiments and modeling, Bolt et al., 1998, and up-scaling by the use of an array of hollow cathodes has been demonstrated, Hellmich et al., 1998. Coating of the inside of metallic tubes with amorphous carbon has been done by using the tube itself as a hollow cathode and introducing the process gases in the tube, Lusk et al., 2009. Recently very high deposition rates, 6-60 μ m/h, for amorphous carbon films with moderate hardness, 18 GPa, were reported by using a magnetically enhanced hollow cathode discharge in a PECVD setup, Zimmermann et al., 2012.

In the hollow cathode HiPP-PECVD study, Pedersen et al., 2012, a Cu cathode mounted in the camber lid, was used to ignite an Ar plasma jet down towards the substrate. Acetylene gas was fed into the plasma from the side to deposit amorphous carbon films at high deposition rate. The high power pulses were superimposed on a conventional DC power by connecting a HiPP power supply and a DC power supply in series to facilitate the ignition of the plasma and give more stable operating conditions. A similar setup of power supplies has been shown to be beneficial also for magnetron sputtering, Samuelsson et al., 2012. HiPP-PECVD processes using a total applied average power of 50–200W_{in} DC or HiPP+DC mode were studied. HiPP pulses of approximately –450 V and 12 A were used with a pulse duration of 30 μs and a repetition frequency of 500 Hz. These discharge conditions resulted in an average HiPP power of 30W to the cathode and the total input (average) power was varied by adjusting the DC power.

The plasma in the hollow cathode HiPP-PECVD process was investigated in more detail by optical emission spectroscopy (OES), the changes in the degree of ionization between the different discharge configurations. Fig. 1 shows a comparison between optical emission spectra taken from a DC discharge and a HiPP + DC discharge. In Fig. 1a-b the total average discharge power was 150 W, while the HiPP fraction was increased in Fig 1c where the average power was decreased to 70 W, still keeping the HiPP contribution to 30 W. It should be noted that the vertical scale in Fig. 1 is arbitrary and independent of each other, which means that quantitative comparisons cannot be made. Significant qualitative differences between the different cases can be noted: 1) By using pure DC power for the plasma discharge, only neutral copper (Cu I) can be detected ($\lambda \sim 324 - 327$ nm, $\lambda \sim 510 - 523$ nm and $\lambda \sim 578$ nm), while when using HiPP + DC power, also emission from singly ionized copper (Cu II) is detected ($\lambda \sim 404 - 420$ nm), NIST, 2012. This is particularly striking in Fig. 1c, where the HiPP contribution to the overall discharge is considerably higher. 2) Only when using HiPP + DC power can emission of singly ionized argon (Ar II) be detected (Fig. 1c at $\lambda \sim 428 - 437$ nm and at $\lambda \sim 459 - 473$). 3) Emission of singly ionized carbon (C II) is only found in the HiPP + DC mode, which is most clearly seen at $\lambda \sim 658$ nm, but also at $\lambda \sim 426-427$ nm (Fig. 1c), NIST, 2012.

By using this HiPP-PECVD setup, amorphous carbon film containing copper were deposited from a gas mixture of Ar and C₂H₂ with growth rates in the 30 μm/h range. Very encouraging is the low hydrogen content of 4 at.% in the films, achieved at a process pressure of 450-550 mTorr without applied substrate heating. It was found that the growth rate increased with about 20% when using a combination of HiPP and DC power as compared to using pure DC power for the same average input power and flow of acetylene (Fig. 2). This was attributed to the more efficient plasma chemistry achieved in the more ionized plasma supplied by the HiPP discharge, as previously described in Section 2.2. It was further shown that the microstructure of the deposited film could be significantly altered by varying the AC substrate bias, i.e. by varying the kinetic energy of the surface bombarding species. A high substrate bias, leading to a very energetic ion bombardment, yielded a film with a very dense microstructure, while a lower substrate bias gave films with a less dense and even porous microstructure.

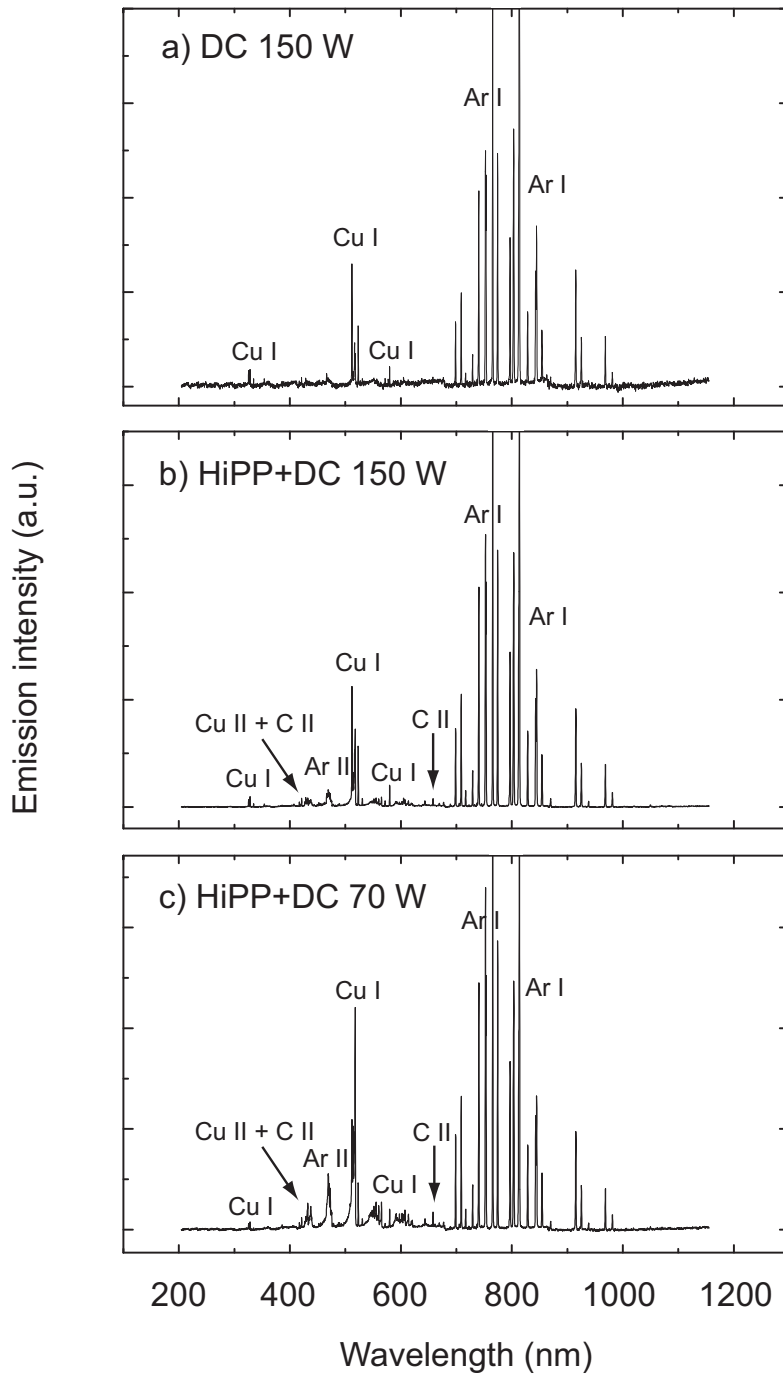


Figure 1: Optical emission spectra taken in an Ar plasma fed with acetylene with (a) a DC discharge at an average power of 150 W, (b) a HiPP + DC discharge at an average power of 150 W, and (c) a HiPP + DC at an average power of 70 W. The different regions indicating neutral and ionized species are given. Reproduced with permission from Pedersen et al., 2012, copyright 2012 Elsevier B.V.

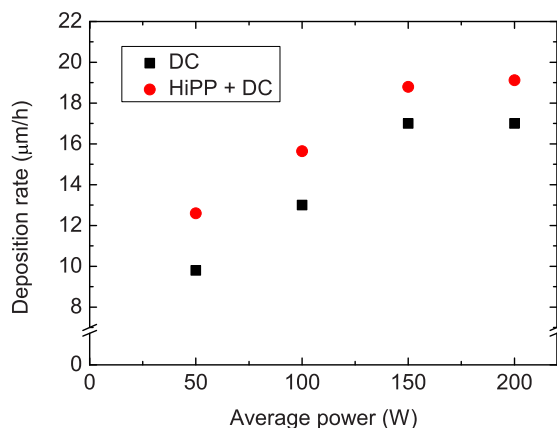


Figure 2: Deposition rate for different input power using DC power (squares) and HiPP + DC (circles) for a constant C_2H_2 flow of 3 sccm. Reproduced with permission from Pedersen, 2012, copyright 2012 Elsevier B.V.

4. Future outlook

One of the key aspects of HiPP-PECVD is the high degree of ionization of the species present in the plasma (Fig. 1). As described in the introduction, by applying a negative bias on the substrate, ionized species can be guided towards the growing film by a low substrate bias in order to increase film thickness uniformity on complex shapes, or be accelerated towards the growing film by a high substrate bias, to provide an energetic bombardment during film growth. One of the main motivations for an energetic bombardment in PECVD has been to maintain sp^3 -hybridization during deposition of diamond and cubic boron nitride, Konyashin et al., 1997 and Robertsson, 2008, although this remains to be investigated in the case of HiPP-PECVD.

Furthermore, it has also been shown by HiPIMS that ionic bombardment allows phase tailoring of both elemental and compound films, Lundin and Sarakinos, 2012. One example is given by Alami et al., 2007, for deposition of Ta films by HiPIMS. Ta forms both a low resistivity body-centered cubic crystal structure (also known as the α phase) at elevated temperatures as well as a metastable high resistivity tetragonal phase (β -Ta) at room temperatures, Clevenger et al., 1992. The great abundance of Ta ions in the HiPIMS discharge (up to 70 % of the neutral Ta was ionized) in combination with a substrate bias led to a more efficient momentum transfer to the growing film, which in turn allowed for controlling the magnitude of the internal stress in order to promote the α phase. There is no reason why the same approach should not work in HiPP-PECVD. In particular, since similar results have also been demonstrated by plasma enhanced ALD (PEALD), where an energetic bombardment by ions created in the plasma has been shown to alternate crystal structure of metal oxide films, Profijt et al., 2012 and Profijt et al., 2013. It was shown that the crystalline phase of titania deposited at 300 °C changed from anatase, when deposited without substrate bias, to rutile when -200 V substrate bias was employed.

These results show that an ion bombardment, obtained by using a properly designed substrate bias voltage scheme and the ionized species in the plasma, is a potentially very powerful process parameter that can be used for the tuning of film properties. Especially, tuning crystalline phase and even preferred growth

orientation of polycrystalline hard coatings of e.g. $\text{TiC}_x\text{N}_{1-x}$ and Al_2O_3 is of great interest to the hard coatings industry since it has been shown that the performance of hard coatings of TiC, Leonhardt and Wolf, 1996, and $\alpha\text{-Al}_2\text{O}_3$, Ruppi, 2008, can be optimized by depositing the coatings in the proper crystal orientation. Furthermore, also the ability to tailor the stress in films by ion bombardment should be of interest to the hard coatings industry since the stress modification in CVD grown α -alumina today is done by sand blasting post growth treatment, Barbatti et al., 2009.

It still remains to confirm that the plasma density in HiPP-PECVD is as high as can be expected from similar high power pulsed plasmas used in HiPIMS. It is well known from the field of HiPIMS that a high power pulsed plasma lead to a higher plasma density, and that the plasma density scales with the instantaneous power delivered to the plasma discharge. To our knowledge no plasma density measurements have yet been performed in the HiPP-PECVD devices, this due to the higher process pressure of the HiPP-PECVD processes (70-175 Pa) compared to HiPIMS processes (0.1-1 Pa), making conventional collisionless sheath approximation theory for Langmuir probe measurements unreliable, Demidov et al., 2002.

Given the possibilities to tailor the film properties by ion bombardment, HiPP-PECVD processes which allows for a significant ion bombardment and very efficient plasma chemistry, due to their higher plasma densities, should have great potential for realizing highly efficient deposition of films with tailored properties.

Acknowledgements

Petter Larsson is gratefully acknowledged for technical and scientific assistance in designing and constructing the HiPP-PECVD used by the authors. Financial support from the Swedish innovation agency (VINNOVA) and Ångpanneföreningens forskningsstiftelse (ÅForsk) is gratefully acknowledged. One of the authors (DL) also gratefully acknowledges the financial support provided by the Swedish Research Council (VR) through his postdoctoral fellowship.

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