

Plasma Science and Technology

Room 201 ABCD W - Session PS3+TF-FrM

Plasmas and PVD

Moderators: Erwin Kessels, Eindhoven University of Technology, the Netherlands; Scott Walton, Naval Research Laboratory

10:45am PS3+TF-FrM-11 Self-Regulating Electron Temperature in High-Power Impulse Magnetron Sputtering Discharges and Its Effect on the Metal Ion Escape, Kateryna Barynova, University of Iceland; Nils Brenning, KTH Royal Institute of Technology, Sweden; Swetha Suresh Babu, University of Iceland; Joel Fischer, Daniel Lundin, Linköping University, Sweden; Michael A. Raadu, KTH Royal Institute of Technology, Sweden; Jon Tomas Gudmundsson, University of Iceland; Martin Rudolph, Leibniz Institute of Surface Engineering (IOM), Germany

We analyze how the primary electron temperature in high-power impulse magnetron sputtering (HiPIMS) depends on the sputtered target. The analysis is based on the experimental discharge data for 7 different target materials, which were modeled using the Ionization Region Model (IRM), a semi-empirical global model for HiPIMS discharges. We observe that the electron heating and collisional cooling processes stabilize after some time into the pulse (20 - 40 μ s) reaching a steady state and leading to an almost constant electron temperature; the initial transients in the electron temperature are caused by only small discrepancies in these terms. The underlying mechanism that causes this self-regulation are the rate coefficients for electron impact ionization, which increase monotonically with electron temperature. This leads to a self-balancing mechanism in which an increase in the electron temperature increases the collisional losses of the kinetic energy of electrons because of the higher collisions rate with species in the ionization region. The opposite is true for the decreasing electron temperature. In addition, the steady-state electron temperature depends on the target material and inversely correlates with the self-sputter yield of the target. The species composition in the ionization region shifts from being composed of argon species to target species; and argon has a much higher ionization potential compared to all the studied target materials, so both the ionization and cooling rates substantially increase only at the higher electron temperatures compared to the ionization region composed of target species. This explains the experimentally observed low electron temperature in high self-sputter yield target discharges. Since the mean free path of the sputtered atoms, before being ionized, depends on the electron temperature, we can explain with the IRM results why in metal-rich discharges ionization occurs further away from the target, leading to higher chances of ionized sputtered species to escape to the substrate because the electric field is weaker there. The dominating species in the ionization region, which define the main collisional loss process and the electron temperature, are not identified only by the sputter yield of the target, but by a more complex recycling loop of argon and target species in the ionization region and the rarefaction of argon in front of the target.

11:00am PS3+TF-FrM-12 Comparison of Particle Size and Morphology of Graphene-Like Carbon Grown with and Without Substrate in Atmospheric Pressure Microwave Plasma, Parker Hays, Dhruval Patel, Dren Qerimi, University of Illinois at Urbana-Champaign; Michael Stowell, Lyten; David Ruzic, University of Illinois at Urbana-Champaign

Graphene-like carbon materials were synthesized on a substrate as well as free-standing using an atmospheric pressure microwave plasma (APP) system. Argon and nitrogen were utilized as carrier gases and methane as the carbon precursor. This study compares the morphological and structural differences of the materials formed under each growth condition.

Free-standing carbon material was collected from the APP using quickly inserted TEM grids at various distances from the microwave insertion point. Carbon was also grown on a temperature-controlled aluminum surface using the same APP system by placement of the aluminum substrate at different distances from the microwave insertion point.

Scanning Electron Microscopy (SEM) images were used to find the particle diameter distributions for each case, showing for both the free-standing carbon and the carbon grown on aluminum that mean primary particle size increased as a function of increased methane flow rate and distance from the microwave insertion point, and decreased with an increase in microwave forward power. Furthermore, the particle diameter distributions in the free-standing case showed minimal change past the bulk plasma

boundary, meaning most of the free-standing graphene growth occurred in the bulk plasma and at the boundary.

Raman spectroscopy was employed to evaluate the structural order and defect density of the carbon materials. The free-standing material exhibited a higher I_D/I_G intensity ratio, suggesting increased disorder and a more amorphous structure compared to substrate-grown samples, which displayed sharper G and 2D peaks indicative of more crystalline graphene. These findings suggest that while substrate-free growth at atmospheric pressure offers a more scalable and simple synthesis route, growth on substrate may yield higher structural quality in the resulting carbon materials.

11:15am PS3+TF-FrM-13 Mass Spectrometric Study of Ar-Diluted Ammonia Borane Plasma for h-BN 2d Film Formation, Takeshi Kitajima, Reiji Kawasaki, Toshiki Nakano, National Defense Academy, Japan

Ammonia borane is used as a relatively safe source of BN for the rapid synthesis of h-BN, an important insulating material¹ in the field of two-dimensional electronics². Ammonia borane plasma attracts attention when aiming at high-speed film formation, and analysis of active species in the plasma is necessary. In this study, active species generated from ammonia borane powder irradiated with Ar plasma were analyzed by mass spectrometry. Parallel plate type 100MHz driven capacitively coupled plasma generated in a high vacuum chamber is used. After placing 0.1 g of ammonia borane (BH_3NH_3) powder on the RF electrode and evacuating, a 10 W glow discharge was formed with an Ar gas flow rate of 30 sccm. A copper sample heated to 800°C was placed downstream, and when BN radicals were supplied at a pressure of 800 Pa, an h-BN atomic film was formed over 30 minutes as shown in the SEM image and Raman spectrum of Fig. 1(a,b). Radical analysis in the downstream was performed with a mass spectrometer at a pressure of 30 Pa. Figure 1(c) shows the difference in the mass spectrum when the plasma is turned on and off. $BNH_5(30)$ is increased by plasma lighting. $O_2(32)$ is produced by plasma irradiation to the chamber wall. Radicals generated from ammonia borane raw material leading to formation of h-BN atomic film are presumed to be BNH_5 generated by decomposition of BH_3NH_3 . Dangling bonds of BNH_5 are thought to generate chemical reaction activity on the substrate. Time dependence of mass signal is shown in Fig.1(d). The relation of OH and BNH_5 is shown in Fig.1(e). OH is linearly related to BNH_5 amount and presumed to the major source of production. Contrarily, O signal is nonlinear to the BNH_5 signal as shown in Fig.1(f). The consequence of the oxygen related radical exposure will be summarized in the presentation. 1 K.H. Lee, et.al. Nano Letters 12, 714 (2012). 2 L. Song, et.al. Nano Letters 10, 3209 (2010).

11:30am PS3+TF-FrM-14 Automated Deposition Chamber for Functional Dielectrics: Development and Implementation, Stanislav Udoenko, Ian Mercer, Susan Trolrier-McKinstry, Jon-Paul Maria, Darren Pagan, The Pennsylvania State University

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Automated Deposition Chamber for Functional Dielectrics: Development and Implementation

The dielectric and piezoelectric properties of ferroelectrics make them essential in the fabrication of multilayer ceramic capacitors (MLCCs) and various transducers (such as those used in medical ultrasound, naval sonar, and consumer electronics) [1–3]. Modern devices demand high-quality, reproducible material synthesis, especially in the fabrication of complex multilayer structures where the thickness of individual layers critically affects device performance. In this context, automating the material synthesis process becomes highly beneficial, as it reduces human error, increases repeatability, and improves overall efficiency. However, there is currently a disconnect between university-based materials design and synthesis which is primarily an analog process and large-scale automated manufacturing found in industry.

This project focuses on developing a framework for digitizing and automating functional ferroelectric synthesis in a university setting. Our demonstration case is the sputtering of ferroelectric films within a vacuum deposition chamber. In the initial stage, a data acquisition and controller system was designed and installed on deposition chamber automated for doped AlN. Next, LabVIEW-based software was developed to acquire data from all electronic units of the chamber—including sputter cathode power

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supplies, mass flow controllers, temperature controllers, and vacuum pumps. Following software development, control functionality was implemented, enabling the system to send control commands to all electronic units while simultaneously logging process parameters in real time. Additionally, Python scripts were developed to convert deposition recipes—originally created in Microsoft Excel—into system control routines, easing use by non-experts.

References:

- [1] Jaffe, B., Cook, W. R., & Jaffe, H. (1971). *Piezoelectric Ceramics*. Academic Press.
- [2] Setter, N., et al. (2006). "Ferroelectric thin films: Review of materials, properties, and applications." *Journal of Applied Physics*, 100(5), 051606.
- [3] Haertling, G. H. (1999). "Ferroelectric ceramics: History and technology." *Journal of the American Ceramic Society*, 82(4), 797–818.

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