

Tuesday Afternoon Poster Sessions, December 4, 2018

Plasma Processing

Room Naupaka Salon 1-3 - Session PS-TuP

Plasma Processing Poster Session

Moderator: Martin Nieto-Perez, CICATA Queretaro

PS-TuP-1 Synthesis of TiO₂/CuOx Thin Film Composites by the Simultaneous Ablation of Ti and Cu Metallic Targets, A. Valeria Garcia-Caraveo, Instituto Tecnológico y de Estudios Superiores de Occidente, Mexico; *E Camps, E Campos-Gonzalez*, Instituto Nacional de Investigaciones Nucleares, Mexico; *A Perez-Centeno*, Centro Universitario de Ciencias Exactas e Ingenierías, Universidad de Guadalajara, Mexico; *M Santana-Aranda*, Centro Universitario de Ciencias Exactas e Ingenierías, Universidad de Guadalajara; *G Gomez-Rosas, L Rivera*, Centro Universitario de Ciencias Exactas e Ingenierías, Universidad de Guadalajara, Mexico; *D Cardona*, Instituto Tecnológico y de Estudios Superiores de Occidente, Mexico; *J Quiñones-Galván*, Centro Universitario de Ciencias Exactas e Ingenierías, Universidad de Guadalajara, Mexico

Titanium Dioxide (TiO₂) is an n-type semiconductor material that has been used for different applications such as photo-catalysis, hydrogen separation and dye sensitized solar-cells. The band gap for TiO₂ anatase phase is 3.2 eV, which means that UV light is needed to generate a photo-catalytic reaction. In this work, we proposed to generate a combined system of TiO₂ with Copper Oxide (CuOx) to reduce the TiO₂ band gap so that the photo-catalytic process can be induced under UV and Visible light.

We present the results obtained for the synthesis of a combined system of TiO₂ and CuOx thin films deposited on glass substrates. We used a reactive Pulsed Laser Deposition (PLD) technique with a mixture of argon/oxygen. The produced plasma was diagnosed by means of the time of flight technique using a Langmuir planar probe; the voltage drop across a 20 Ohm resistance was measured and the current was calculated in order to estimate the ion density and its mean kinetic energy, with the aim of having a control on the deposition process.

A Nd:YAG pulsed laser with a wavelength of 1064nm was divided into two equal beams using a beam splitter. Each individual beam was focused on the surface of Titanium (Ti) and Copper (Cu) solid targets respectively for 15 minutes under an argon/oxygen pressure of 20 mTorr. Primarily we obtained an amorphous TiO₂ thin film and a CuO thin film, afterwards we deposited a multilayered thin film of TiO₂/CuO/TiO₂ and finally a combined system of TiO₂ and CuOx by ablating both Ti and Cu targets simultaneously.

The obtained thin films were characterized by X-Ray Photoelectron Spectroscopy (XPS), X-Ray Diffraction (XRD), Raman Spectroscopy, Ultraviolet-Visible spectroscopy (UV-Vis) in order to know the stoichiometry and oxidation phases and Transmission Electron Microscopy (TEM) was used to define the morphology.

Characterization reveals that a TiO₂ amorphous phase was deposited in any of the different experiments but Cu reacts in different ways depending on the plasma characteristics, it is shown that we can obtain different Cu oxidation states an even synthesize metallic Cu nanoparticles in a TiO₂ matrix.

PS-TuP-2 Study of Carbon Fiber Manufacturing Process by Plasma Oxidation/stabilization and Microwave assisted Carbonization, Seok-Kyun Song, B Kim, M Jung, Cheorwon Plasma Research Institute, Republic of Korea; *S Lee*, Korea Institute of Science and Technology, Republic of Korea
Carbon fiber has a carbon content of 90~95% or more, and its strength is ten times that of steel. The manufacturing process proceeds with stabilization (chlorination), carbonization of the PAN fiber. In particular, the stabilization process is long-term treatment at high temperature which high cost is incurred. In order to low cost manufacture for carbon fiber, that is need reduction of stabilization process time. For that, we researched the oxidation/stabilization process using atmospheric pressure plasma and e-beam technology.

The atmospheric pressure plasma system developed by CPRI team suggests possibility to reduce oxidation/stabilization process time (from 120 min to 30 min, from 120 min to 10 min by add e-beam technology) and cost. Plasma oxygen radical accelerates the progress of fiber cyclization. After plasma treatment, the surface of oxidized/stabilized fiber had no damage.

In the nitrogen atmosphere, oxidated/stabilized fiber was used for low temperature carbonization of 300 ~ 1000 degrees Celsius, and energy consumption was reduced by directly supplying energy to the heating

element near the fiber by the microwave assisted (MWA) method instead of the conventional resistance heating.

It has been suggested that energy can also be reduced by providing microwave assisted (MWA) energy directly to low temperature carbonized fiber for 1000 ~ 1600 degree Celsius high temperature carbonization.

PS-TuP-3 Design and Diagnosis of Atmospheric Microwave Plasma by Transmission Line Resonator, Jun Choi, Korea Institute of Industrial Technology (KITECH), Republic of Korea

Recently, atmospheric microwave plasmas (AMP) have attracted much attention as promising plasma sources for industrial applications such as material processing, surface treatment and biomedicine [1]. The AMP eliminates the need for a vacuum system and has a long operational time of the electrodes [2]. In this work, a plasma source using a two-parallel-wire transmission line resonator (TPWR) in atmospheric argon is described [3]. The E-field distribution and reflection coefficient of the resonator for the given frequency (~900MHz) are estimated by COMSOL Multiphysics software based on microwave theory to transfer maximum power from power supply to the resonator and enhance the power efficiency. The TPWR-AMP can sustain with low power less than 3 watts. The TPWR is fabricated to investigate the plasma characteristics such as excitation temperature, electron temperature and rotational temperature. The electron excitation temperature was measured by the Boltzmann plot and the rotational temperature was determined by comparing the spectra measurement and simulation of rotational lines of the OH band. The electron density was obtained by Stark broadening with the measurement of emission spectra. The characteristics of the TPWR-AMP shows that the device has the potential to be used effectively and widely in industry.

[1] J H Kim et al., Appl. Phys. Lett. **86** (2005)

[2] J Winter et al., Plasma Sources Sci. Technol. **24** (2015)

[3] J Choi et al., Phys. Plasmas **24** 093516 (2017)

PS-TuP-4 Nitridation of SiO₂ by using a VHF (162 MHz) Multi-tile Push-pull Plasma Source, You Jin Ji, K Kim, K Kim, J Byun, S Lee, Sungkyunkwan University, Republic of Korea; *A Ellingboe*, Dublin City University, Ireland; *G Yeom*, Sungkyunkwan University, Republic of Korea

Nitriding processes of SiO₂ thin film have been applied in various semiconductor device manufacturing. For example, in the fabrication of nanometer scale semiconductor devices, a nitriding process of SiO₂ for a nitride layer applied to gate insulator has become an important process to prevent the penetration of p-type dopant (boron) through the thin gate oxide. Typically, plasma and thermal nitridation methods are used to meet the requirement for the nitride layer. However, the thermal method has a bad influence on the device performance due to the high processing temperatures (600-1500 °C), and the plasma method tends to cause damage on the treated layer due to the ion bombardment and shows a low nitridation percentage in the film due to the difficulty in dissociating nitrogen molecules having a high electron-impact dissociation energy. Very high frequency (VHF; > 30 MHz) plasma is known to dissociate nitrogen molecules more effectively with a high dissociation rate at a low temperature due to a high electron energy tail in the electron energy distribution. Therefore, in this study, the nitridation of SiO₂ was performed to obtain a uniform silicon oxynitride (SiO_xN_y) layer using a VHF (162 MHz) multi-tile push-pull plasma source at room temperature. High nitrogen incorporation (~ 24.51 %) in the SiO_xN_y layer was confirmed by the X-ray photoelectron spectroscopy (XPS) analysis at the optimized nitridation condition. In addition, the EDS in TEM showed that a SiO_xN_y layer was uniformly formed after the nitridation of SiO₂ at the optimized condition. The leakage current of the MOS capacitor that has the SiO_xN_y layer formed by using the VHF (162 MHz) multi-tile push-pull plasma source was measured to be lower than that has the SiO_xN_y layer formed by the conventional CCP (60 MHz) plasma source.

PS-TuP-5 Fabrication of SnO Thin Films by Reducing Plasma on Atomic Layer Deposited SnO₂, Jaehong PARK, B PARK, H Kim, Yonsei University, Republic of Korea

Oxide semiconductors have been intensively investigated in emerging applications, such as thin film transistor (TFT), flexible electronics and solar cell materials, owing to the added functionality using great diversity of materials and structures. There have been numerous studies on n-type semiconductors such as ZnO, SnO₂, and In₂O₃, whereas the research on p-type semiconductors is still ongoing due to the lack of synthesis technology. SnO is a representative p-type oxide semiconductor with wide band-gap and high mobility, but the poor stability of SnO limits the synthesis method to physical vapor deposition (PVD). PVD is difficult to

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apply in future integrated circuit process with aggressive scaling down and 3D structuring. Atomic layer deposition (ALD) is a promising technique owing to atomic-scale thickness controllability and great conformality. In this study, we synthesized p-type SnO thin film using ALD and consequent plasma treatment for reduction. We investigated the crystal structure, morphology, and electrical properties of SnO by using x-ray diffraction (XRD) and atomic force microscope (AFM). In addition, we evaluated the thermal and chemical stability of SnO film. As a result, SnO film shows highly stable SnO phase even after annealing at 400 °C with oxidation/reduction environment. This study will contribute to apply oxide semiconductor in future application by implementing various device structure, such as p-n junction, complementary metal oxide semiconductor (CMOS), and p-channel TFT.

PS-TuP-6 Plasma-Surface Interactions in Atmospheric Pressure Plasmas: In situ Measurements of Electron Heating in Materials, *S Walton*, Naval Research Laboratory; *B Foley*, Pennsylvania State University; *J Tomko*, University of Virginia; *D Boris*, *E Gillman*, *S Hernandez*, Naval Research Laboratory; *A Giri*, University of Virginia; *T Petrova*, Naval Research Laboratory; *Patrick Hopkins*, University of Virginia

The energy flux to a surface during plasma exposure and the associated surface heating are of long standing interest as they contribute to the physico-chemical changes that occur during plasma-based materials synthesis and processing. Indeed, the energy delivered to the surface, via a flux of particles and photons, in concert with a flux of reactive species serves to chemically modify, etch, and/or deposit materials, with an efficacy that depends on the plasma processing environment. A unique feature of plasma synthesis and processing is that most of the delivered energy is absorbed at or very near the surface over short (picosecond) time scales. The dissipation of thermal energy proceeds through electron-electron and/or electron-phonon interactions as they propagate through the material, with relaxation time scales that can be orders of magnitude slower. Typically then, the surface is not in thermal equilibrium with the bulk material. Fast, surface-sensitive techniques are thus required to fully appreciate the dynamics of the plasma-surface interaction. In this work, we employ pump-probe Time-Domain Thermoreflectance, a surface sensitive technique typically used to measure thermal properties of thin films, to determine electron heating of thin metal films during exposure to an atmospheric pressure plasma jet. The results, in conjunction with current measurements, are used to develop a first order understanding of plasma jet-surface interactions. The results show that the energy delivered by the plasma jet causes a localized increase in electron energy within the thin film over an area commensurate with the plasma jet radius. More details of this work can be found in the following recently published paper: Walton, S.G., Foley, B.M., Tomko, J., Boris, D.R., Gillman, E.D., Hernandez, S.C., Giri, A., Petrova, Tz.B., Hopkins, P.E., "Plasma-surface interactions in atmospheric pressure plasmas: In situ measurements of electron heating in materials," *Journal of Applied Physics* **124**, 043301 (2018).

PS-TuP-7 Classification of Aluminum Alloys by an Inexpensive Laser Induced Breakdown Spectroscopy System, *Kevin Renato Maldonado Dominguez*, *R Sangines del Castro*, CNyN-UNAM, Mexico

Laser induced breakdown spectroscopy (LIBS) has been widely used for elemental analysis of solid, liquid and gaseous samples due to its portability and the practical null sample preparation. Due to the changing nature of the induced plasma the necessity of using a time-resolved spectroscopy system, consisting of a Echelle spectrograph and an ICCD camera, is usually mandatory; however, these systems are delicate and the portability could be difficult. Although field spectrometers with a CCD detector could have relatively high spectral resolution, their main drawback is the trouble to synchronize the laser beam trigger to the beginning of the spectral acquisition. If the trigger issue is resolved with a relatively low jitter, these spectrometers could be an attractive alternative for characterization of material composition via LIBS.

In this work, a CCD spectrometer (Avantes, AvaSpec) is tested to determine the composition of several aluminum alloys and is compared to the results of an Echelle spectrometer by identifying spectral lines from the NIST database and applying statistical tools. Different experimental parameters were studied and results are discussed in terms of the correct identification of the Al alloy.

PS-TuP-8 Optimizing Deposition Parameters for Reactive Magnetron Sputtering by Monitoring the Plasma Optical Emission Spectroscopy, *Genaro Soto-Valle Angulo*, *R Sangines*, CNyN-UNAM, Mexico

Thin films technology has grown during the last years due to its many applications. Extensive research has been focused on silicon oxides and nitrides for its excellent properties.

Although the wide variety of techniques for thin film deposition, reactive magnetron sputtering is still one of the preferred. However, one of the major drawbacks of this technique is the lack of repeatability of the film properties under the same deposition parameters as a result of the so-called target poisoning.

The aim of this work is to obtain the optimal deposition conditions for SiO₂ and Si₃N₄ thin films grown by reactive magnetron sputtering in order to guarantee specific film properties, particularly the refractive index for optical filters applications. Optical emission spectroscopy was used to monitor the deposition process in real time and to observe the species contained within the plasma. Information of the emission intensity was employed to obtain intensity ratios between certain emission transitions, and this information was then correlated with the film optical properties analyzed by spectroscopic-ellipsometry and XPS. Relation between these parameters can give valuable knowledge about the deposition rate and refractive index, which will allow to fabricate thin films with variable refractive index and with a high degree of repeatability.

PS-TuP-9 Origin of Plasma Damage during Sputtering of Ultrathin ITO Contact Layer on p-GaN for InGaN/GaN LEDs, *T Kim*, *Y Cha*, *Joon Seop Kwak*, Sunchon National University, Republic of Korea

We systematically examined the origin of plasma-induced damage on p-GaN surface during the sputtering of ITO transparent conductive electrodes (TCE) and its effects on the forward voltage and the light output power (LOP) of InGaN/GaN LEDs. Firstly, we investigated the effect of direct current (DC) power in radio frequency (rf) superimposed DC sputtering (RF+DC sputtering) of ITO on the forward voltage and LOP of InGaN/GaN LEDs and found that the plasma-induced damage was sensitive to the DC power. The forward voltages of the LEDs at 20 mA drastically decreased from over 5 V to 3 V and the LOP of the LEDs was greatly enhanced by more than 20% at 250 mA, when the DC power was changed from negative to positive values. Secondly, electron flux as well as ion flux during the RF+DC sputtering of ITO with the various DC power were calculated based upon the plasma discharge parameters measured by cutoff probe and Langmuir probe. Changing the DC to positive power drastically reduced the electron flux in plasma, suggesting that plasma electrons play an important role in plasma-induced damage of p-GaN surface. Furthermore, the significant increase in forward voltage of the LEDs was observed, when electron-beam irradiation on p-GaN surface was employed. This confirms that the plasma electrons, not ions, can cause the plasma-induced damage on p-GaN during the sputtering of ITO. Lastly, physical mechanism for the generation of plasma-induced damage on p-GaN by the plasma electrons was suggested. The plasma electrons can compensate the deep level defects (DLDs) in the p-GaN surface and reduce the density of DLDs, which increase the effective barrier height at the ITO/DLD band of p-GaN. Furthermore, the plasma electrons yielded the energetic ad-atoms of ITO on p-GaN during sputtering by energy transfer of the electrons to the ad-atoms and increased the plasma-induced damage on p-GaN. We successfully demonstrated the plasma-induced-damage-free ITO TCE on the InGaN/GaN LEDs by sputtering, which showed 20 % improved LOP of the LEDs with comparable forward voltage of 2.9 V at 20 mA to the LEDs with conventional e-beam-evaporated ITO.

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