

GEC15-2015-000751

Abstract for an Invited Paper
for the GEC15 Meeting of
the American Physical Society

Diagnostics of Nano-Particle Formation in Process Plasmas

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The main sources of particle generation during plasma surface processing and the formation of nano-composite materials are (i) the formation of large molecules, mesoscopic clusters and particles in the plasma bulk by chemically reactive gases, and (ii) the formation and incorporation of particles at surfaces (target, substrate) by means of plasma-wall interaction. The plasma process promotes the particle formation by excitation, dissociation and reaction of the involved species in the gas phase. The different stages of the particle growth in the gas phase can be observed by various plasma diagnostics as mass spectrometry, laser induced evaporation, photo-detachment, IR absorption, microwave cavity measurements, Mie scattering and self-excited electron resonance spectroscopy (SEERS). Common diagnostics of particle formation also use the observation and analysis of harmonics and other discharge characteristics. Especially the early stages of the particle growth are not well investigated since they are experimentally inaccessible by standard methods as mentioned above. A novel collection method based on neutral drag was tested in order to get a better insight into the early stages of particle growth. The experiments were performed in an asymmetric, capacitively coupled rf-discharge, where multiple growth cycles can be obtained. Making use of the correlation between the particle growth cycles and the bias voltage as well as the phase angle between discharge current and voltage it was possible to monitor each growth process in-situ. This allowed to collect particles at any desired stage of the growth cycle via the neutral drag method. Size distributions of the nanoparticles at the different stages of the growth cycle were determined ex-situ by transmission electron microscopy. The observed correlations of particle size and bias voltage, which can be used for prediction of the particle growth, are qualitatively explained. Furthermore, the change of the electron density in the plasma during the growth cycles has been monitored by microwave interferometry and the nano-particle formation and deposition was observed in-situ by XPS and NEXAFS at a synchrotron beamline.

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