Operando Scanning Electron and Microwave Microscopies in Plasmas: A Comparative Analysis

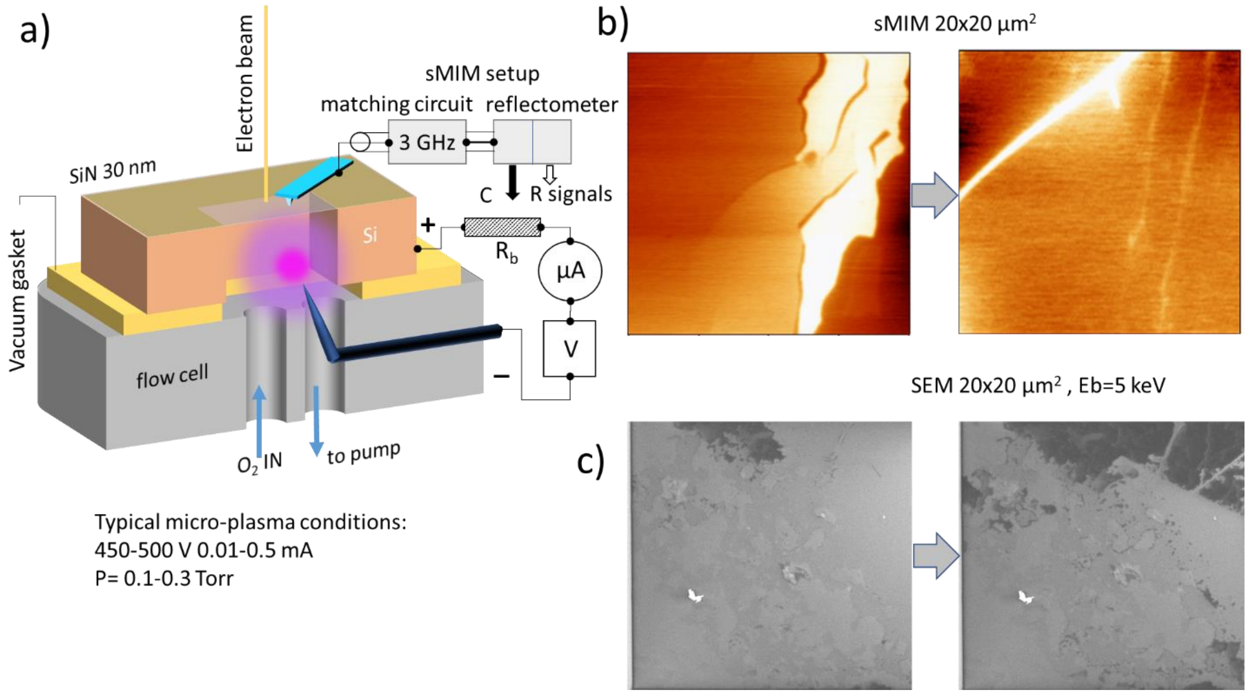
Andrei Kolmakov[1*] *and Alexander Tselev*[2]

[1] Physical Measurements Laboratory, National Institute of Standards and Technology, Gaithersburg, MD 20899, USA

[2] Department of Physics & CICECO–Aveiro Institute of Materials, University of Aveiro, 3810-193 Aveiro, Portugal

There exists a great need for an operando nanoscale characterization of evolution of surface composition and morphology during plasma-assisted processing. This includes sputter deposition, plasma-assisted etching, plasma-enhanced atomic layer deposition and other processes relevant for semiconductor and aerospace industries, environmental remediation, and biomedical technology. Recently, we proposed near-field scanning-probe-based microwave imaging as a tool to image surfaces immediately (a few seconds) after plasma processing with a sub-100 nm spatial resolution [1].

In this communication, we report on true operando near-field microwave imaging in plasma and its extension to imaging in SEM. The core of our approach is a microflow DC micro-discharge chamber equipped with a few-10s nm-thick SiN membrane transparent to a few keV electrons and microwave radiation (Figure 1a). This membrane isolates the probe of a scanning Microwave Impedance Microscope (sMIM) or an SEM column from plasma environment and thus enables real-time imaging of a surface of interest under plasma conditions. Using model systems, such as graphene, PMMA films, and polystyrene microparticles, we have comparatively explored performance of the SEM and sMIM in the same plasma-assisted processes. In particular, sensitivity, frame rate, spatial resolution, probing depth, and probe-induced effects were evaluated and compared. Figure 1b shows the process of etching of folded graphene by air DC plasma. The same process is depicted in the SEM snapshots in Figure 1c. The sMIM contrast in Figure 1b is due to graphene conductivity which degrades significantly after a few seconds of ca. 200 mW plasma treatment. Under optimal conditions, a resolution of ca. 50 nm can be achieved for metallic objects with commercially available probes. In turn, the contrast formation in SEM is due to local variations in the electron emission yield of the sample. This can be affected by presence of static charges in the SiN membrane. The spatial resolution can be as high as 10 nm with a frame rate on the order of 1 Hz. Interestingly, both the microscopies revealed formation of highly conductive filamentary structures of graphene during plasma etching—a phenomenon that is a subject of ongoing research.



Typical micro-plasma conditions:
450-500 V 0.01-0.5 mA
P= 0.1-0.3 Torr

Figure 1 (a) Experimental setup for operando sMIM and SEM studies under DC plasma; (b) sMIM (conductance channel) 20x20 μm^2 image of a graphene sample before (left panel) and after a few seconds (right panel) of application of DC air plasma (0.25 Torr, 500 V, 0.4 mA). The overall signal intensity drops (color scales are different in the images) due to local graphene etching, and filamentary structures are formed; (c) A similar sequence of 20x20 μm^2 images in SEM (5 keV) taken during DC (0.25 Torr, 450 V 0.2 mA) plasma treatment

References

[1] A. Tselev, J. Fagan, and A. Kolmakov, "In-situ near-field probe microscopy of plasma processing," *Applied Physics Letters*, vol. 113, no. 26, p. 263101, 2018.