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# In situ plasma studies using a direct current microplasma in a scanning electron microscope

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Background incl. aims:
Plasmas, sometimes called the fourth state of matter and defined as an ionized gas, have applications in a variety of fields, including biomedicine, materials science, and gas conversion. A typical example of plasma technology is their use in the semiconductor industry, where they fulfill a critical role in the etching process of silicon wafers when fabricating nano-scale components that make up computer chips [1]. At the same time, plasmas are heavily researched to gain further fundamental insights, and to develop novel technologies and applications. These research areas include plasma catalysis, gas conversion, nanomaterial synthesis, and biological treatments [2].

Many of the relevant plasma processes take place at a microscopic scale. However, current diagnostics (e.g., optical techniques) are often limited to global measurements, naturally averaging out microscopic effects. Micro-scale investigations of plasma-treated materials are typically limited to ex situ studies, where post-mortem analyses are performed on samples after plasma treatment [3]. This limits the temporal resolution of current studies, while also exposing the sample to ambient conditions during the transfer from the plasma device to the diagnostic equipment.

Therefore, we aimed to develop a setup that enables the formation of a plasma inside a scanning electron microscope (SEM). Specifically, we sought to achieve true in situ SEM imaging during plasma operation and to investigate the effect of plasma on materials at high spatial and temporal resolution, while preserving the original state of the plasma-treated sample.

Methods:
We modified an environmental SEM to integrate the instrumentation for generating and monitoring a plasma within the chamber [4]. A custom flange was designed to provide a passthrough for electronics and gas supply. The gas supply is connected to a steel tube with a small orifice, which brings the gas close to the sample, and they are both placed in the field of view of the SEM. The sample is grounded, while the nozzle is connected to a DC-DC converter placed inside the chamber which is powered by a DC power supply outside of the microscope. This way, plasmas in a variety of gases were generated by supplying the nozzle with a voltage up to 2 kV. The current flowing through

the sample to the ground was measured using a shunt resistor, enabling real-time monitoring of the plasma discharge. While the plasma was on, the SEM could be used in its normal high-vacuum operating mode.

The in situ setup was tested for a range of plasma and imaging conditions. Different discharge gases were used to generate the plasma, including N2, CO2, and an O2/Ar mixture. Flow rates were varied between 2.5 and 7.5 sccm, which was monitored by a mass flow meter outside of the microscope. Further, the distance between the nozzle and the sample could be varied, typically between 75 and 150 µm. This setup offers high flexibility, though a plasma discharge was not possible for all conditions (e.g., large gap distance and low flow rate) at our maximum applied voltage of 2 kV. True in situ imaging was possible with both the secondary electron and backscattered electron mode of the Everhart-Thornley detector, while EDX measurements were performed between plasma treatments.

Results:
To characterize the plasma discharge, we acquired the voltage-current characteristics, where the plasma current was monitored while varying the applied voltage. These data were obtained for a number of gap distances and gas flow rates. These characteristics all exhibit a rising slope and the positions of the curves for varying conditions are indicative of a so-called obstructed abnormal glow discharge. This implies that a discharge should be sustainable at even lower voltages, if the gas density could be increased.

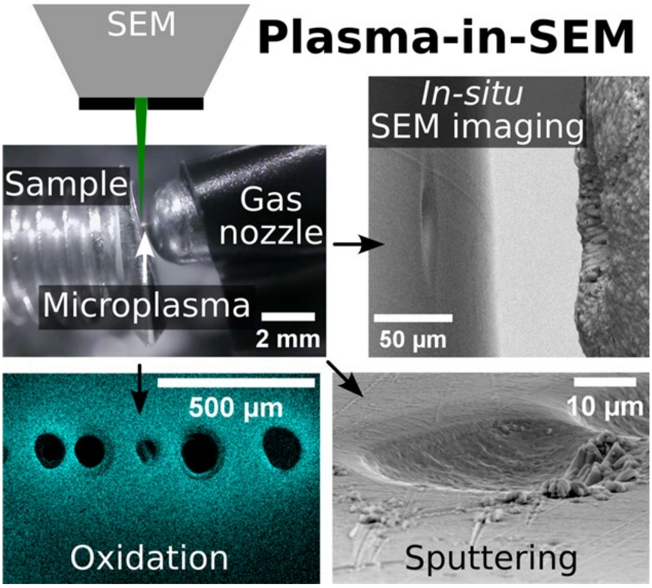
Next, we investigated how the plasma operation affected SEM imaging, showing that true in situ SEM imaging was possible during the plasma discharge. While the electron beam is deflected by the electric field needed to sustain the plasma, the presented setup is sufficiently stable to obtain high-quality SEM images. The deformation of the images by the deflected electron beam must be considered for quantitative image analysis, but regardless the images are suited for qualitative studies. In a proof-of-concept example, a video was captured where the sputtering of the sample could be monitored in real-time. The homogeneous sputtering of the sample also yielded microscopic conical structures, formed out of spherical particles or impurities that were present on the sample surface.

To further explore the SEM-plasma synergy, we employed elemental mapping to study the composition of the sample and how it is affected by various plasmas. Apart from the physical effects of the plasma (i.e., sputtering), chemical reactions were also observed. We showed that oxygen-containing plasmas, e.g., CO2, can oxidize the Cu sample outside of the sputtered area. For the latter, the sputtering effect was dominant and exposed the pristine Cu below the surface.

Finally, we explored the effect of the voltage polarity on the plasma properties, since a negatively biased electrode accelerates the positive ions to the nozzle rather than to the sample. This yielded sputtering of the nozzle instead of the sample and subsequent redeposition of nozzle material on the sample. At the same time, the sputtering of the sample was strongly limited while the oxidation in a CO2 plasma could be observed for a broad region.

Conclusions:
This work presents a dedicated setup to generate a plasma inside a scanning electron microscope. The setup enables true in situ SEM imaging, where electron-based images can be acquired while treating the sample with a plasma. The interaction of the plasma and the sample was studied by real-time imaging, where a pit being formed in the sample sheds light on the sputtering behavior of the setup. Moreover, microscopic conical structures were formed by bombarding spherical particles or impurities with ions. Finally, chemical effects of the plasma were observed, where a CO2 plasma was able to oxidize the Cu sample. This setup is a stepping stone for future research, both in the direction of materials science and toward more advanced plasma diagnostics.

**Graphic:**



**Keywords:**

SEM, Plasma, Sputtering, In-situ SEM

**Reference:**

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