Current-Voltage Characteristics of DC Discharge in Micro Gas Jet Injected into Vacuum Environment

K Matra, H Furuta and A Hatta
Department of Electronic and Photonic Systems Engineering, Kochi University of Technology, Kochi, 782-8502, Japan
E-mail: 148001e@gs.kochi-tech.ac.jp, hatta.akimitsu@kochi-tech.ac.jp

Abstract. A current-voltage characteristic of direct current (DC) gas discharge operated in a micro gas jet injected into a secondary electron microscope (SEM) chamber is presented. Ar gas was injected through a 30 $\mu$m orifice gas nozzle (OGN) and was evacuated by an additional pump to keep the high vacuum environment. Gas discharges were ignited between the OGN as anode and a counter electrode of Si wafer. The discharge was self-pulsating in most of the cases while it was stable at lower pressure, larger gap length, and larger time averaged current. The self-pulsating discharge was oscillated by the RC circuit consisting of a stray capacitor and a large ballast resistor. The real time plots of voltage and current during the pulsating was investigated using a discharge model.

1. Introduction
Nowadays, micro plasma jet has attracted much attention because of its various merits, such as its ability to confine micro scale local plasma, to be generated in atmospheric and sub-atmospheric pressure [1-2]. Moreover, its low operating cost, non-thermal plasma, low power consumption, and easy handling make it attractive in many plasma applications. Some of its applications are surface modifications, analytical chemistry, nanostructure growth and biomedical treatment [3].

DC micro plasma in scanning electron microscope (SEM) using small orifice gas nozzle (OGN) anode has been developed by our group [4-6]. The usage of orifice type gas nozzle consisting of a small hole on a thin wall is a key issue for injection of localized high pressure micro gas jet into vacuum environment. Otherwise, by using a micro capillary type gas nozzle, the pressure of injected gas becomes too small to operate gas discharge due to the poor conductance of long capillary tube.

Micro plasma processing in SEM is expected to be used for local restoration of micro electronics devices such as large scaled integrated circuits (LSIs), fabrication of micro mechanical system devices (MEMS) and nanostructured material growth with SEM observation. In addition, there are advantages of plasma generation in SEM that break down voltage of plasma ignition can be reduced by electron beam and that the profile of injected gas can be observed directly [6].

Because the injected gas rapidly disperses in the vacuum environment, the discharge gas locally exists at the exit of the OGN. The discharge space is limited in the dense gas jet area which profile is determined by the local high pressure gas injection and the rapid dispersion without any wall or any strict boundary. The micro gas jet in vacuum will bring some interesting features of not only the rapid gas flow but also the self-limited discharge space with a large gradient of pressure as compared to conventional gas discharges.
In this paper, electrical characteristic of DC gas discharge operated in a micro gas jet injected in the SEM chamber is presented. The current and voltage characteristics were studied with variation of supplied pressure, electrode gap distance and source voltage. Because the discharge current sometimes oscillates the self-pulsating due to a large ballast resistance and a stray capacitance in the discharge circuit, the temporal wave forms of voltage and current were investigated with monitoring the time averaged values.

2. Experimental apparatus and procedure

The experiments in this research were conducted inside an SEM (Hitachi S-3000N) chamber as shown in figure 1. The anode of an OGN was supported by a 3-D micro-manipulator in the chamber. As the cathode, a cut of mirror polished Si wafer of low resistivity was placed on another 3-D micro-manipulator vertically and counter to the OGN. The gap distance between the electrodes was adjusted to 100, 200, or 300 µm by monitoring the scale in the SEM image, as shown in figure 2a.

The OGN was a standard 1/8" SS-316 tubing closed at one end with a laser drilled hole 30 µm diameter (Lenox Laser). Ar gas was supplied to the OGN through a mass flow controller (MFC). The Ar pressure applied to the orifice, monitored by a pressure gauge, was proportional to the gas flow rate of MFC. The conductance of the OGN as the ratio of gas flow rate to the applied pressure was about 0.075 sccm/kPa. A turbo molecular pump (PFEIFFER VACUUM, TMU071YP) was installed on a service port on the side wall of the chamber in addition to the original vacuum system. With injection of micro gas jet at 15 sccm flow rate which was the maximum in the present experiments, the chamber pressure was kept below 1 Pa and the SEM worked in the normal operating conditions.

Figure 2a shows a typical SEM image of the gas jet between the OGN and Si cathode at 15 sccm flow rate, 210 kPa orifice gas pressure (OGP) and with 300 µm gap. The gas molecules were ionized by the scanning electron beam at 5 keV in energy, and the produced electrons were detected in the same way as the detection of secondary emission from solid surfaces. The brightness of the image depends on total accumulated number density of gas molecules along with the beam line passing through the gas jet. Because the conductance of the straight part of OGN (3 mm inner diameter, D) was sufficiently larger than that of the small end orifice (30 µm, d), as depicted in figure 2b, the pressures inside of OGN and at the exit of orifice would be close to the applied pressure. When the discharge was ignited, the SEM image became fully white due to sufficient electrons from the discharge plasma.

![Figure 1. A schematic diagram of the micro gas jet set up in SEM observation chamber.](image1)

![Figure 2. (a) A SEM image of the OGN anode and Si cathode with Ar micro gas jet and (b) a schematic drawing for OGN.](image2)
A positive voltage was applied to the OGN from a DC power source of 1 kV maximum through a 10 MΩ ballast resistor ($R_B$). The discharge voltage and current were monitored using a set of digital multi-meters (DMM). One was used for measuring the source voltage and the other was used for measuring the voltage drop across the ballast resistor $R_B$ which reflected the discharge current ($I_d$). The applied voltage to the OGN (discharge voltage) was obtained from the difference between the measured voltages. The discharge voltage and current sometimes pulsated due to oscillation in the circuit while the source voltage was kept constant. The monitored voltage and current by the multi-meters at a small sampling rate of 2 samples/s represented time averaged values when the discharge was automatically pulsating at frequencies around a several kHz. For measuring the real pulsating current, the counter electrode of Si substrate was grounded by a 50 Ω through terminator ($R_M$) inserted in BNC cable line, and for measuring the real discharge voltage of OGN, a high voltage probe was connected to the OGN. The current and voltage waveforms for the pulsating mode were stored in a digital oscilloscope (IWATSU-LeCroy, LT364).

3. Experimental results and discussion

3.1. Voltage-current characteristics of discharge in micro-gas jet in vacuum

Figure 3 shows the plots of voltage and current monitored by the multi-meters with variation of gap length ($G_D$) and the applied pressure (OGP). The obtained V-I curves look almost following the conventional characteristics of glow discharge, i.e., constant voltage with variation of current. By increasing the source voltage while the discharge voltage was almost constant, the discharge current limited by the ballast resistance gradually increased.

Figure 4 shows typical waveforms of voltage and current monitored by the oscilloscope at the experimental condition of 300 µm gap and 140 kPa pressure. There were two different modes of discharge observed, self-pulsating mode and continuous mode [7-10]. The discharge at 800 V (source voltage, $V_s$) was automatically pulsating even if the source voltage was kept constant. The discharge at 1,000 V was stable and continuous at the constant voltage and the constant current. In a previous work, it has been confirmed that the continuous discharge mode is suitable for local plasma processing like as micro sputter etching due to their stability [6].

![Figure 3](image-url)

Figure 3. Plots of time averaged voltage and current for discharge in micro gas jet in vacuum monitored by the multi-meters.

![Figure 4](image-url)

Figure 4. Waveforms of voltage and current monitored by the oscilloscope at 300 µm gap, 140 kPa pressure, and 800 V and 1,000V source voltages ($V_s$).
Figure 3 actually includes both the plots for the stable continuous discharge mode which represent real voltage and current, and also the hypothetical plots of time average for the pulsating discharge mode. The thick lines plotted in figure 3 show the results in which the discharge mode transited from the self-pulsating mode to the continuous mode when the monitored current exceeded critical current values as indicated. The minimum current to sustain continuous gas discharge ($I_{\text{min}}$) depended on the gas, pressure, electrode gap distance, electrode material and etc. All the plots on the thin lines show the apparent values on DMM for self-pulsating mode. The continuous mode was observed at the lower pressure (75 kPa) when the current exceeded 20 and 5.68 µA at the gap distance of 200 and 240 µm, respectively. At the medium pressure (140 kPa) the continuous mode appeared at the gap length 300 µm when the current exceeded 40 µA. At the higher pressures, 140 and 210 kPa, and at the shorter gap distance, the discharge was always pulsating even if the current was increased until the maximum source voltage.

As shown in figure 4, the current waveform at 800 V consists of sharp pulses with peak current ranging from 2-3 mA repeating with intervals about 0.75 ms. At 1,000 V, the discharge current was stable and continuous at about 70 µA as indicated in figure 3. The peak current in the pulsating mode was 30 to 40 times larger than that for the continuous mode. In the pulsating mode, the voltage dropped abruptly during the short pulses of discharge current and gradually increased during the intervals resulting in saw tooth wave. The pulsating frequency, mostly determined by the interval length, ranged in 0.5-5 kHz depending on the pressure, gap length, and also the source voltage.

3.2. Self-pulsating discharge oscillated in the circuit

As shown in figure 5, the current waveform at 800 V consists of sharp pulses with peak current ranging from 2-3 mA repeating with intervals about 0.75 ms. At 1,000 V, the discharge current was stable and continuous at about 70 µA as indicated in figure 3. The peak current in the pulsating mode was 30 to 40 times larger than that for the continuous mode. In the pulsating mode, the voltage dropped abruptly during the short pulses of discharge current and gradually increased during the intervals resulting in saw tooth wave. The pulsating frequency, mostly determined by the interval length, ranged in 0.5-5 kHz depending on the pressure, gap length, and also the source voltage.

The self-pulsating mode is characterized by two threshold voltages. One is sustained voltage or recovery voltage as the minimum voltage to sustain plasma ($V_{RE}$) and the other is breakdown voltage as the maximum ($V_{BR}$) [12]. For the self-pulsating mode, the discharge voltage oscillated between these two voltages as indicated in figure 5 and 6. The discharge current appeared in the period of voltage drop from $V_{BR}$ to $V_{RE}$. Here, the self-pulsating of the discharge can be divided into 3 phases as depicted in figure 5. The 1st phase is increasing voltage during the intervals of discharge, the 2nd phase is increasing of discharge current, and the 3rd phase is decreasing of discharge current with abrupt decrease of voltage to terminate until the voltage decreased to $V_{RE}$ [13].
In the 2nd phase, in the present result, the discharge current abruptly increased to 0.83 mA within the rising time of 4.8 µs and the voltage dropped from 472 V to 453 V. In the 3rd phase, the discharge current decreased from the peak to around 50 µA within 16 µs together with decreasing of voltage from 453 V to $V_{RE}$ (405V). After diminishing the discharge, the voltage increases to $V_{BR}$ (472 V) again. This process was periodically repeated resulting in the self-pulsating discharge.

In figure 6, it should be noticed that, even at the same condition for gas discharge, the observed breakdown voltage $V_{BR}$ and the minimum sustain voltage $V_{RE}$ varied with variation of the source voltage. It is necessary to explain the reason why the voltage source affected the discharge characteristics.

For the minimum voltage, $V_{RE}$, the observed value was not the minimum sustain voltage for the discharge but just the turning voltage from decreasing phase to the recovery phase. By carefully looking at the transition from the 3rd phase to the next 1st phase, i.e., at the termination of the discharge, the voltage has started to recover before the current disappears completely. Because the current was continuously supplied through the ballast resistance also during the discharge, the minimum voltage $V_{RE}$ appeared at the time when the discharge current decreased to the supplying current. When the discharge current was decreased to the supplying current, the actual voltage turned to increase before termination of discharge. When the source voltage was increased, due to increase of the supplying current through the ballast resistance, the decreasing of voltage was terminated faster and started to recover at the higher voltage.

For the breakdown voltage, it should be necessary to consider the pre-ionization before the distinctly observed current pulse. When the voltage approached the real breakdown voltage, ionization started to build up the plasma column and finally to ignite the main discharge. It will take a few µs for building up the plasma column. Due to the delay of building up the main discharge after the voltage became the breakdown voltage, the observed $V_{BR}$ was exceeded the real breakdown voltage. When the source voltage was increased, the working voltage was increased more rapidly during the delay time resulting in observation of higher maximum voltage at the ignition of main discharge.

Figure 7 shows the voltage and current waveforms with variation of source voltage at the same discharge condition of self-pulsating mode, 210 kPa pressure and 100 µm gap. The pulsating frequency increased with increasing the source voltage. Figure 8 shows a model for the electrical circuit of discharge including the stray capacitor.

During the interval of discharge, the stray capacitor was charged through the large ballast resistor until the gas breakdown voltage. When the voltage exceeded the breakdown voltage, the gas discharge started to flow the electrical current with discharging the stray capacitor. When the current supplied from the ballast resistor was sufficient to sustain the continuous discharge ($I_d \geq I_{min}$), the continuous mode of discharge started. If the current supply from the power source through the ballast resistor was insufficient for sustaining the continuous discharge ($I_d < I_{min}$), the discharge was automatically terminated and the stray capacitor was charged again until the breakdown voltage. Therefore, $I_{min}$, the minimum current to sustain continuous discharge, was the threshold between the self-pulsing mode and continuous mode.

When the source voltage ($V_s$) was increased, the supplied current through the ballast resistor increased resulting in rapid recovery of voltage for the next discharge of ignition and higher frequency of pulsating. When the ballast resistor was decreased, the supplied current increased with resulting in higher frequency [14]. The trends of charging the stray capacitor simply follow the RC oscillation circuit model. In the present experiments, the stray capacitor was about 65 pF. It has been confirmed that the most of stray capacitor existed in the coaxial high voltage cable between the ballast resistor and the electrode.

The self-pulsating discharge also could be controlled by varying ballast resistor and stray capacitor [9, 14-15]. Due to the extreme peak power together with controllable pulse frequency, the self-pulsating mode is also applicable for micro plasma processing [14-16].
Figure 7. Voltage and current waveforms with variation of the applied voltage at 210 kPa pressure and 100 µm gap.

Figure 8. Model for the voltage oscillation due to charging and discharging of the stray capacitance through the ballast resistance.

4. Conclusion
Characteristic of current and voltage of discharge operated in micro gas jet injected into vacuum environment was investigated using the orifice of 30 µm diameter for anode and the Si substrate for cathode equipped in the SEM. The discharge showed self-pulsating mode and continuous mode. The continuous discharge occurred at lower pressure gas jet with longer gap distance at larger current. The self-pulsating of voltage and current was investigated using a model of discharge circuit including stray capacitor.

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6. References
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