

Characterization of Plasmas from a Pulsed Jet Discharge for Applications to VUV Spectroscopy and Micromechanics

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Abstract—Plasmas from a pulsed jet discharge have been characterized with respect to gas species and nozzle design. Spectral lines from the gas used in the pulsed nozzle are apparent in the visible region. The vacuum ultraviolet spectrum, in particular for heavier gases, is dominated by emission from species sputtered from the nozzle. The production of highly ionized and excited states from materials created by the sputtering of the nozzle has possible applications in VUV spectroscopy. By operating the pulsed jet discharge at a 50 Hz repetition rate with NF_3 to produce excited fluorine ions, etch rates in excess of $10 \mu\text{m}/\text{min}$ have been achieved in silicon which may have applications to micromechanics.

I. INTRODUCTION

RECENTLY, a high-voltage high-current discharge through a pulsed, supersonically-expanding gas has been reported as a potential VUV laser excitation mechanism [1]. These discharges are designed with an electrode immediately behind the orifice of a pulsed jet with a grounded wire mesh located about 1.5 cm below the nozzle orifice. Such an arrangement achieves high peak currents ($\sim 2 \text{ KA}$) and a very high current density ($\sim 1 \text{ MA}/\text{cm}^2$) since the discharge is confined to the small area of the nozzle orifice. This approach differs from other recent work [2]–[7] in that the discharge duration is short ($\sim 100 \text{ ns}$), and currents in excess of 1 KA are employed.

Although the initial design and operation of these discharges with Ar was outlined in [1], the characterization of such a device with respect to nozzle design, gas parameters, and discharge current needed further study. In this work the visible, ultraviolet, and vacuum ultraviolet (VUV) emission spectra resulting from the excitation of the gas in the pulsed nozzle as well as from the sputtered material from the nozzle itself are investigated.

In addition to these spectroscopic studies, fluorine component gases such as NF_3 have been used in the discharge in order to etch silicon. The results may have applications to micromechanics, where high etch rates are necessary to produce relatively large but deep feature sizes ($\sim 10\text{--}100 \mu\text{m}$) in contrast with microelectronics, where small feature sizes ($\leq 1 \mu\text{m}$) are required at relatively shallow depths.

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II. DESCRIPTION OF DISCHARGE APPARATUS

A schematic of the pulsed jet discharge system is shown in Fig. 1. A pulsed valve (General Valve) is located on the top of the nozzle assembly and sends a short ($\approx 1 \text{ ms}$) pulse of gas through the orifice of the nozzle into the vacuum chamber. While the gas expands through the nozzle, a discharge through the nozzle towards the grounded grid is initiated. The back pressure on the valve is maintained at about 3 atm. The gases studied included He, N_2 , Ne, Ar, Kr, Xe, NF_3 , and SF_6 . A turbo pump backed by a rotary vane roughing pump restores the background pressure in the cell to a few times 10^{-5} torr between pulses when the discharge is operated at low repetition rates (1–2 Hz). The tungsten electrode is situated about 1.5 mm above the nozzle orifice, and a stainless steel wire mesh located 1.5 cm below the orifice serves as the ground plane.

The high-voltage discharge circuit is shown in Fig. 2. The maximum voltage from the power supply is 20 KV. The capacitance of the charging capacitor ($C1$) is 5.6 nF, and that of the peaking capacitor ($C2$) is 4.2 nF. With a thyatron (EG&G, model HY-3202) serving as the high-voltage switch, the current pulse, measured by a commercial current transformer, has a FWHM of about 100 ns, with some ringing out to about $1 \mu\text{s}$. At 15 KV charging voltage, the peak current is 1.8 KA. For a $500 \mu\text{m}$ diameter orifice, this peak current corresponds to an average current density of $0.9 \text{ MA}/\text{cm}^2$ inside the orifice. The trigger output of the pulsed valve is used as the master clock, but a trigger delay of about 1 ms for the thyatron is required for a stable discharge; i.e., the discharge is initiated about 1 ms after the beginning of the jet expansion.

Fig. 3 shows typical voltage and current waveforms. Because of the finite inductance of the electrode, the voltage peaks approximately 80 ns before the current. Assuming the value of the peaking capacitor (4.2 nF) is fixed, the ringing period of the current indicates that the inductance of the discharge is about 290 nH. The decay of the current ringing reveals that the resistance of the discharge reaches a value of approximately 3–4 Ω . Integration of the power calculated using these waveforms gives an estimate of the energy delivered to the discharge of about 200 mJ during the initial current pulse. The small bump in the decay of the initial current pulse (at $\sim 250 \text{ ns}$ in Fig. 3) is apparently due to a surge of current from the outer circuit loop, which includes the storage capacitor of the thyatron.

At higher charging voltages, there seems to be significant energy dissipated during the ringing period of the discharge. From the current voltage waveforms, it can be seen that there is a

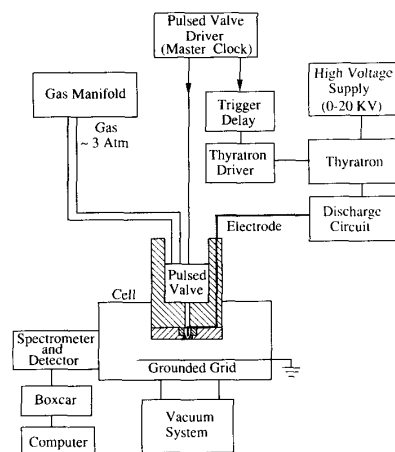


Fig. 1. Schematic of the pulsed jet discharge and the VUV detection apparatus.

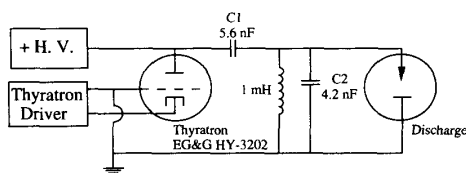


Fig. 2. Schematic of the high-voltage circuit.

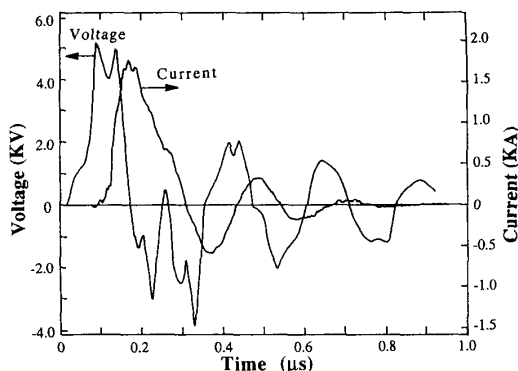


Fig. 3. Discharge voltage and current waveforms.

secondary peak of energy dissipation approximately 200 ns after the initial peak, with additional peaks occurring with a period of roughly 100 ns. This feature can also be seen in the temporal behavior of the fluorescence. Fig. 4 shows traces of the temporal response of the 207 nm emission from Si II, which is very bright in spectra taken with a Macor or glass nozzle using heavy gases in the pulsed jet. At a discharge voltage of 15 KV [Fig. 4(a)], the signal decays after about 100 ns, corresponding to the decay of the initial peak of the discharge current. At 20 KV [Fig. 4(b)], however, the intensity becomes very broad in time and shows a pronounced periodic structure in its decay. When the transit time of the photomultiplier (~ 30 ns) is taken into account, the second fluorescence peak is delayed about 200 ns from the initial peak of the discharge, and the period of later

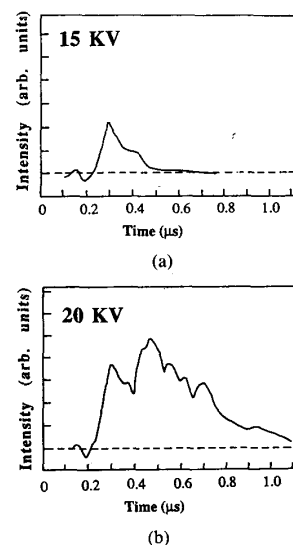


Fig. 4. Traces of the temporal behavior of the intensity of the 207 nm emission of Si II at (a) 15 KV and (b) 20 KV.

fluorescence peaks at about 80–100 ns, which is in good agreement with the analysis of the voltage and current waveforms.

The nozzle assembly is designed so that the nozzle is interchangeable, and therefore, a variety of orifice diameters and nozzle materials can be studied. Discharge operation with nozzle orifices ranging from 250 to 500 μ m in diameter has been characterized. Materials used for the nozzles include Macor, (a machinable ceramic), sapphire, glass, carbon, Delrin, and PVC. The nozzles are designed to give a supersonic expansion and to optimize the production and cooling of molecular radicals and ions [5]. All of the nozzles made from machinable materials and the sapphire nozzles have a 45° half angle above the nozzle orifice on the high-pressure side, and a 90° half angle on the vacuum side. The glass nozzles are made from glass tubing which is heated and tapered at approximately a 45° half angle until the orifice is about 500 μ m. Photographs of the discharge indicate the presence of a Mach disk about 1 cm below the nozzle.

Spectroscopic studies were performed with a scanning 0.2 m VUV spectrometer (Acton Research Corp.) using a photomultiplier with a scintillator as the detector with a temporal resolution of ~ 10 ns. The spectral resolution of the system was approximately 5 Å. The optical axis of the spectrometer was aligned to be approximately 2 mm below the nozzle orifice, corresponding to the point of brightest VUV fluorescence. This was determined experimentally by measuring the fluorescence intensity of several bright VUV lines as a function of the distance from the nozzle, with 1 mm resolution using a movable slit. The signal was averaged with a boxcar integrator, and the data were stored and processed in a computer. Temporal waveforms were obtained with a 400 MHz bandwidth oscilloscope.

III. SPECTROSCOPIC RESULTS

The emission spectra from the pulsed jet discharge were studied in the wavelength range between 35 and 550 nm using different gas species in the pulsed jet and a variety of nozzle materials. All of the spectra presented here have not been cor-

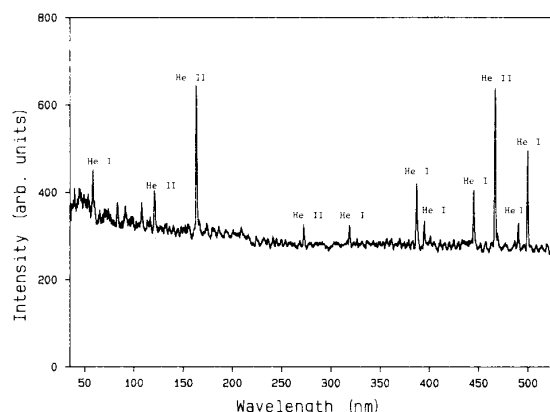


Fig. 5. Emission spectrum at 15 KV charging voltage, taken 250 ns after the peak of the current pulse, with He as the gas species and using a Macor nozzle.

rected for the spectral response of the detection system. With lighter gases such as He, spectra dominated by emission from the gas species in the pulsed jet were obtained. An example of a spectrum obtained with He and a nozzle made of Macor is shown in Fig. 5. All of the bright emission lines can be assigned to excited helium atoms and ions. To obtain this spectrum, the gate on the boxcar was set to a width of 75 ns and was delayed 250 ns after the peak of the current pulse. This time window for observation was chosen because the spectra measured at various time delays indicated that many spectral lines were more prominent during this interval.

Using Ar and other heavier gases in the pulsed nozzle, however, the spectra obtained are quite different. While the visible spectrum obtained with Ar is dominated by lines from Ar II and Ar III, the VUV spectrum is dominated by lines from the sputtered nozzle material. Fig. 6 shows the VUV spectrum obtained using Ar with a sapphire nozzle. The time interval for observation is identical to that used for the He spectrum. The dominant features are aluminum and oxygen lines originating from the sputtering of the sapphire (Al_2O_3) nozzle. Sputtering is due to the effective momentum transfer from the gas ions to the nozzle material. Typical ion kinetic energies inside the nozzle can be estimated to be 10 to several hundred eV. In this energy range efficient sputtering of the nozzle material by heavier rare gases is expected, whereas the sputtering efficiency of He is low [8].

To demonstrate that these spectral features are indeed almost independent of the gas medium, Fig. 7 compares a spectrum obtained with Kr in the pulsed jet discharge and a spectrum obtained using Ar. Both spectra were obtained under identical conditions using the same nozzle with a 300 ns wide gate delayed 200 ns from the peak of the current pulse. The more intense spectrum using Kr was taken first, and then the Ar spectrum. The intensity of the spectral features which are due to emission from the nozzle material tends to weaken the longer the nozzle is used due to the widening of the orifice by the discharge. Also, the sputtering efficiency of Kr is higher than for Ar due to their mass difference. The striking similarity between both spectra and the fact that all prominent features can be assigned to emission from aluminum shows that consideration of the nozzle material is indeed crucial for the interpretation of optical spectra from intense pulsed jet discharges.

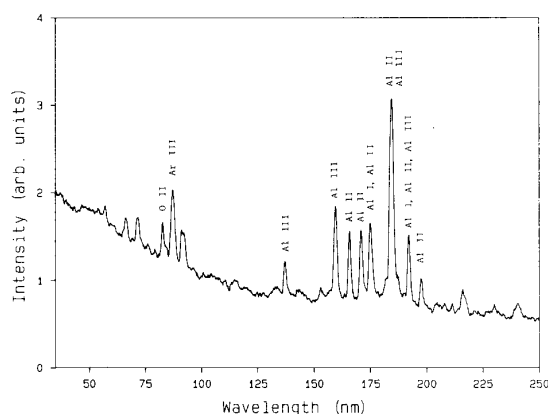


Fig. 6. Emission spectrum at 17.5 KV charging voltage, taken 250 ns after the peak of the current pulse, with Ar as the gas species and using a sapphire nozzle.

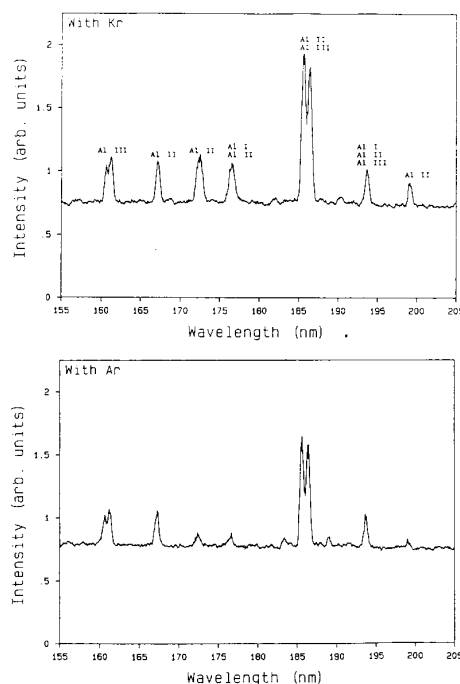


Fig. 7. Comparison of the emission spectra using a sapphire nozzle with Kr and Ar as the gas species. Both spectra were taken under identical conditions with the gate delayed 200 ns after the peak of the current pulse.

When Macor is used as the nozzle material, the VUV spectra with the heavier gases are dominated with Si and Al lines. An example with Ar as the gas species are shown in Fig. 8. The time interval for observation is again identical to that of the He spectrum in Fig. 5. Macor has a complicated chemical composition, but some of the major constituents are SiO_2 (46%) and Al_2O_3 (16%). Spectra obtained with glass nozzles show many of the same Si lines. Spectra taken with carbon, PVC, or Delrin nozzles exhibit strong carbon lines.

The high degree of excitation and ionization apparent in all of the spectra is a result of the initial confinement of the dis-

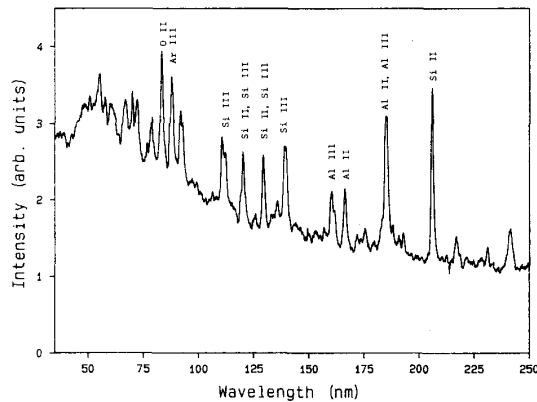


Fig. 8. Emission spectrum at 15 KV charging voltage, taken 250 ns after the peak of the current pulse, with Ar as the gas species and using a Macor nozzle.

charge to the small orifice allowing very high current densities ($\sim 1 \text{ MA/cm}^2$) to be achieved. Some of the spectral lines which have been identified as arising from the sputtering of the nozzle material indicate the formation of states as much as 45 eV above the ground state of the neutral species. Such pulsed jet discharges could therefore have applications in spectroscopic studies of highly excited states of materials that are difficult to evaporate, or in the development of incoherent VUV light sources for other spectroscopic investigations.

In [1], emission from the excimer Ar_2^* around 126 nm was reported using a pulsed jet discharge with similar discharge parameters. In the work reported here, the region around 126 nm was extensively studied with various nozzles and at various discharge voltages, but no evidence of emission from Ar_2^* was detected. Searches for other excimer emissions, such as He_2^* , Kr_2^* , ArF , KrF , and XeF , were also performed but no emission from these species in such a high-current pulsed jet discharge was observed.

IV. PLASMA ETCHING

Increased interest in micromechanics has created the need for faster etching mechanisms for a variety of materials. Recent work with continuous microwave discharges using a halogen containing gas has demonstrated a very high etching rate of silicon [9]. However, the limitations on the design of the nozzles for microwave discharges because of the need for impedance matching warrants exploration of other types of discharges which are more flexible in their design parameters to see if similar etch rates can be obtained. Chemical wet etching is either slow (typically about $1 \mu\text{m/min}$) and/or has problems with masking, since the mask is also etched [10]. Dry etching processes are often slower ($0.1 \mu\text{m/min}$) and rarely exceed $10 \mu\text{m/min}$ [9]. The ability of the pulsed jet discharge to create highly ionized species motivated the study of its feasibility as a high etch rate mechanism when used in conjunction with a halogen containing gas.

Fig. 9 shows the configuration used for etching. A stainless steel wire mesh was used as a test mask. The wire mesh was laid on top of the silicon wafer and mechanically held in place. The wafer was about 2 mm below the nozzle orifice. A discharge voltage of 10 KV was used as a compromise between etching speed and sharpness of the etching pattern. With lower

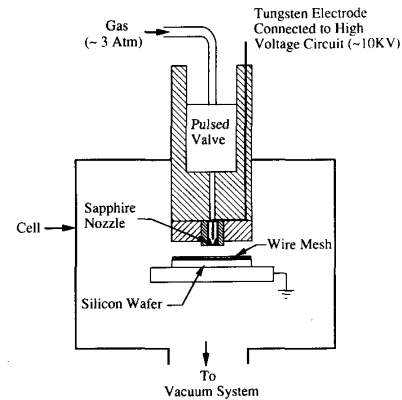


Fig. 9. Schematic of the pulsed jet discharge when used for plasma etching.

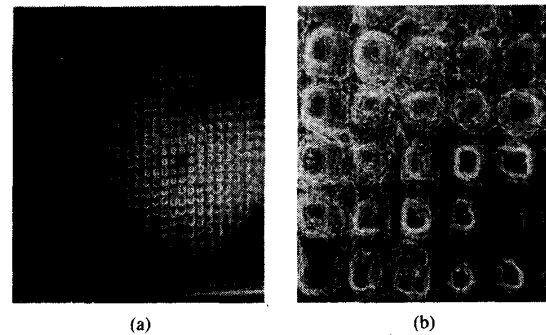


Fig. 10. Scanning electron micrographs of an etched silicon wafer. (a) View of the entire etched area, with a diameter of approximately $1/8$ in. (b) Detail of same wafer. The distance between the wires was approximately $35 \mu\text{m}$. The depth of the deepest etch point was about $50 \mu\text{m}$.

voltages, the etching rate was slower, and at voltage of less than 8 KV, the discharge was not stable when using NF_3 , as the gas species in the pulsed jet. At higher voltages, although the etch rate was somewhat larger, the etched pattern was less clear and some melting of the wire mesh occurred. The cleanest etching was observed using a sapphire nozzle, since this material was the most resistant against sputtering.

Scanning electron micrographs of an etched sample are shown in Fig. 10. The distance between the wires of the mesh was about $35 \mu\text{m}$ in this case. Optical microscopy indicated that the etched features were about $50 \mu\text{m}$ deep at the deepest point. Although further optimization of the discharge parameters will be necessary to obtain sharper features, it is encouraging that by optimizing only two parameters, the discharge voltage and the distance between the nozzle and the wafer, the wire mesh pattern could be replicated in the etched wafer.

Both NF_3 and SF_6 were used as the gas species for etching. Better results were obtained with NF_3 , possibly because the electron temperature is high enough that the dissociative attachment coefficient to NF_3 to form F^- exceeds that of SF_6 [11]. If Ar instead of a halogen-containing gas was used in the pulsed jet discharge, there was no observable etching; therefore, the etching is primarily chemical and not mechanical or thermal. The system was operated at 50 Hz, limited only by problems of heat dissipation in the nozzle. Fig. 11 shows the average of the

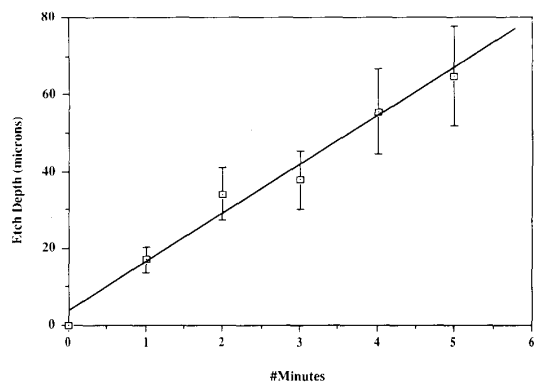


Fig. 11. Average over several runs of the deepest etch depth in a silicon wafer versus the time the system was operated at 50 Hz. NF_3 was used as the gas, and the discharge was operated at 10 KV.

deepest etch depth of several experiments as a function of the time with the discharge system operating at 50 Hz with a voltage of 10 KV. A least squares fit indicates an etch rate of approximately $12 \mu\text{m}/\text{min}$. With further improvements to the construction of the nozzles, so that the heat dissipation can be increased, it is anticipated the repetition rate of the discharge could be increased to further improve the etch rate to approximately $100 \mu\text{m}/\text{min}$, as reported in [9].

V. CONCLUSION

The characterization of a pulsed jet discharge has been performed and possible applications to VUV spectroscopy and micromechanics were explored.

The applications to VUV spectroscopy include the study of very highly excited states of atoms and ions of low vapor pressure materials such as Al and Si. These species are produced by the sputtering of the nozzle material in the high energy region of the nozzle orifice when heavy gases such as Ar or Kr are used.

The feasibility of using a pulsed jet discharge in conjunction with a halogen containing gas as a high etch rate mechanism for silicon has also been demonstrated. Etch rates of about $12 \mu\text{m}/\text{min}$ have been achieved using NF_3 .

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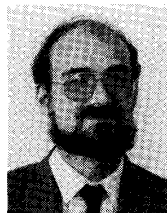
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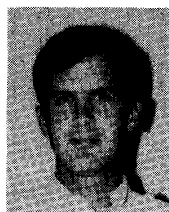


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