MICRO ARC-PLASMA HYBRIDS FOR DETECTION OF VAPORS AT ATMOSPHERIC PRESSURE

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Abstract—**Chemical sensors based on microdischarges – both arcs and plasmas – provide rapid and simultaneous detection of vapors by using their characteristic emission spectra in the plasma glow. Generally, D.C, microplasmas operate in vacuum while arcs exist at higher pressures. Arcs are driven by high transient currents and exhibit wider spectral distribution than plasmas, which are driven by steady currents that are smaller in magnitude. In this paper we report the controlled combination of these discharges and the combined use in ambient air to detect low concentrations of organic vapors. It is found that as the discharge gap is scaled to <75 μm, it becomes possible for a microplasma to exist at 1 atm. With appropriate powering circuitry, an arc can also exist under the same conditions. The relative strengths of the arc and plasma components can be controlled by the adjusting the value of a single resistor. The emission spectra of combined discharges have the spectral characteristics of both arc and plasma spectra. The utility of such a system to vapor sensing is demonstrated by detecting 17 ppm of acetone in air ambient with a 65 ms detection time, and without any pre-concentration. This is accomplished using pulses that consume 60 mJ/discharge, on a device that has an active area of 300 μm x 300 μm .**

I. INTRODUCTION

Gas sensors based on discharge emission spectroscopy offer a compelling alternative to conventional gas sensors as they detect chemicals based on their structure rather than their chemical properties [1][2]. To this end, various plasmas, including inductively coupled plasmas [3], hollow cathode [4], R.F. [5], D.C., and pulsed microdischarges have been miniaturized. In all these devices, the gases and vapors in the ambient are broken down into fragments in the plasma, and these emit light having characteristic line spectra that can be used detect the presence of the gas. Since the line spectra correspond to the atomic or molecular structure of the chemical, every chemical has a unique spectral signature in the microdischarge glow. In addition, the detection is rapid, as the discharge can be created and sampled in a few milliseconds.

Past reports have described the use of both microplasmas [6] and pulsed micro arcs [7][8][9] for various applications. Pulsed and D.C. microdischarges are attractive, partly because unlike other kinds of microdischarges, they can be operated at atmospheric pressures in air ambient with the use of simple battery-operated circuitry [10]. These are requisite features for the practical use of microdischarge sensors in handheld instruments. In one application [7], the pulsed arcs were used to measure the concentration of metallic impurities in liquid samples by striking a discharge between a metal anode and liquid cathode. It is possible by this method, to detect low levels (few ppm) of impurities without any preconcentration. Subsequent work focused on the use of pulsed arcs for chemical sensing of organic vapors [8][9]. Pulsed arcs have a significant afterglow period, which constitutes a changing portion of the emission spectrum [8]. By strategically selecting the optical sampling delay and duration relative to the initiation of the discharge, the sensitivity and signal to noise ratio of the detection can be greatly enhanced. Subsequent efforts demonstrated that it is also possible to reduce the power on these devices significantly (~23 μJ/pulse) by using a three-electrode powering scheme with separate current and voltage electrodes [9].

Microplasmas are both similar and different from larger scale plasmas in important ways [6]. Unlike conventional plasmas, where the glow exists in the region between electrodes, the glow in microplasmas is confined to the proximity of the cathode. Due to the large surface to volume ratio and small discharge gap, secondary emission from the cathode becomes the dominant mechanism for powering the microplasma (instead of avalanche multiplication in the discharge gap.) In addition, very high electron energy that is allowed by the high power density results in a spectrum that is rich in the UV-blue region. The microplasmas studied in [6] had a discharge gap of few hundred microns and existed only at reduced pressures (<30 Torr). At 1 atm. the microdischarge took the form of an arc, which is quite different. Unlike the microplasmas which are driven by steady currents, micro-arc discharges have high transient current. These typically result in optical emission over a wide spectral range covering both UV and infrared portions of the spectrum. In contrast, microplasmas which have a narrower (and stronger) spectral distribution of energy.

In this effort we report the use of hybrid micro arc-plasma discharges operating at atmosphere. It is found that as a consequence of further scaling of the discharge gap, it is possible for a microplasma to exist at higher pressures. Further, at these pressures, micro-arcs can co-exist, and the relative contributions of both can be easily adjusted by

using appropriate circuitry. The use of these hybrids in chemical sensing is investigated.

II. Design and Fabrication

The device (Fig.1) consists of planar copper microelectrodes fabricated on a glass (Pyrex™ #7740) substrate. The anode-cathode gap spacing is 75 μm. The cathode is designed to have a large area (300 μm x 300 μm) as microdischarges are driven mainly by secondary emission from the cathode. Some designs had a tip at the anode to provide a consistent breakdown path for the arc. Devices without the tip at the anode (and same discharge gap) were also fabricated and showed identical results.

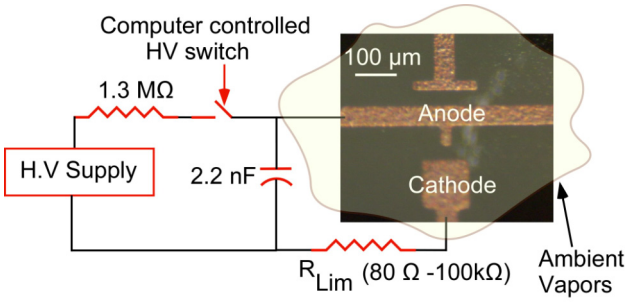


Fig. 1: Picture of the device and a simplified schematic of the circuit. The third electrode is not used. The anode-cathode gap is 75 μm.

To operate the device, a pulsed discharge is struck by discharging across the gap a 2.2nF capacitor that has been previously charged to a high voltage. A limiting resistor is connected between the cathode and ground to control the peak current. A computer-controlled circuit determines the timing of the discharge pulse and its synchronization with the spectrometer. In order to conserve power and increase lifetime, a one-shot detection scheme is used instead of a periodic scheme. All measurements are done at 810 Torr, with a pulse period of 45 ms.

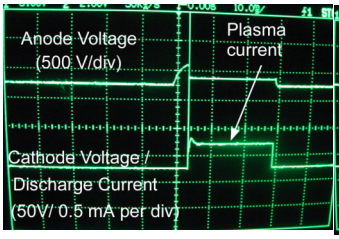
The device fabrication begins by sputtering a Ti/Cu (50 nm/100 nm) seed layer on the glass wafer. Then a 50 μm thick layer of photoresist (Clariant AZ 9260) is spun on and patterned to define the mold for electroplating. A layer of copper, 40 μm thick, is then electroplated to define the electrodes. The photoresist is removed, and the base layers etched away to form the device. Thick electroplated copper is used to increase the device lifetime, as some sputtering of the cathode is inevitable in the microplasma.

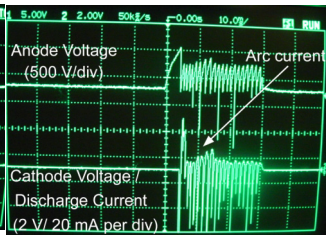
III. Experiments

A. Electrical Characterization

To evaluate the electrical characteristics of the hybrid discharge, an oscilloscope was used to examine the cathode and anode (through a 10 MΩ : 100 kΩ divider) waveforms. The anode waveform gives an indication of the breakdown voltage, while the cathode waveform indicates the discharge current (drop across the limiting resistor).

Figure 2a shows the captured waveforms when used with a 100 kΩ limiting resistor. This represents a plasma-like discharge, which has a steady discharge current. The breakdown voltage was 515 V and the sustaining voltage was 356 V. The discharge has a weak arc component as shown by a leading spike in the current step.





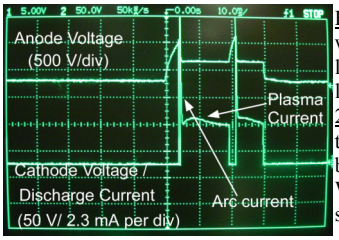


Fig 2a (top-left): Osilloscope waveforms when using a 100 kΩ limiting resistor, showing a plasma-like steady breakdown current. Fig. 2b (top): With 82 Ω limiting resistor the discharge is "arc-like", with bursts of current Fig. 2c (left): With 22 kΩ limiting resistor both steady and burst currents are seen.

Figure 2b shows the waveforms when using a 82 Ω limiting resistor. The breakdown voltage was 678 V and the sustain voltage was 415 V. This corresponds to an arc-like discharge, and is powered by high current spikes instead of a steady current. In this discharge, the capacitor repeatedly charges up to the breakdown voltage and dumps all the charge stored in surges of current. This behavior is clearly seen in the anode waveform.

Figure 2c shows the waveform for a hybrid arc-plasma discharge with the limiting resistor set to 22 kΩ. The breakdown voltage was 625V and the sustain voltage was 415V. The current waveform shows both a steady (plasma) and a spiky (arc) component. Note that the arc breakdown voltage is greater than the sustaining plasma voltage, and the capacitor charges up to the arc breakdown voltage after the plasma current shuts off.

If a capacitor is not used, the discharge takes the form of a plasma breakdown, even when small limiting resistors are used. This is because the high current required for the arc comes directly from charge stored in the capacitor, and the circuit presents high output impedance if a capacitor is not used. It is also necessary to minimize the inductance between the capacitor and the anode. When an inductor or long wires are used to connect to the anode, a plasma discharge is formed as the inductor reduces bursts in current. These results suggest that the capacitor and inductor values can be used to control the characteristics of the discharge.

B. Optical Characterization

Visual characterization is performed by observing the pulsed microdischarge with a video camera connected to a microscope. Figure 3a shows a photograph of the discharge at atmosphere, when a 100 kΩ limiting resistor is used. A bright glow is seen over the cathode and near the anode, with dark space in between. This is similar to D.C. microplasmas operating at reduced pressures [6], with the

exception that the pulsed microplasmas have a more pronounced anode glow.

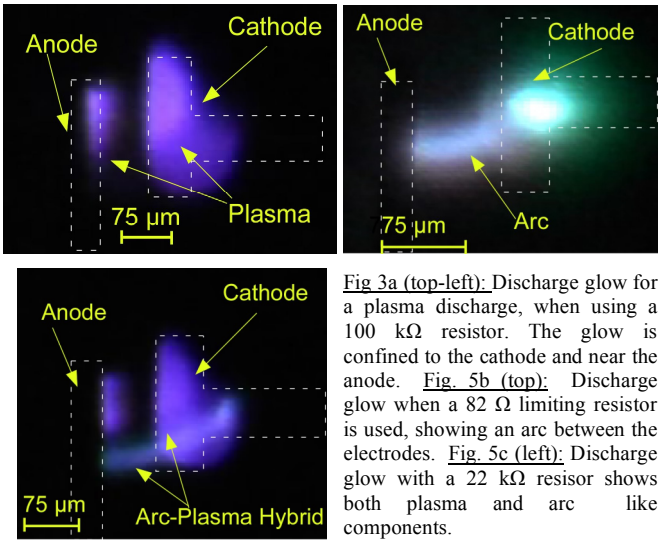


Fig 3a (top-left): Discharge glow for a plasma discharge, when using a 100 kΩ resistor. The glow is confined to the cathode and near the anode. Fig. 5b (top): Discharge glow when a 82 Ω limiting resistor is used, showing an arc between the electrodes. Fig. 5c (left): Discharge glow with a 22 kΩ resisor shows both plasma and arc like components.

Figure 3b shows the discharge glow when an 82 Ω limiting resistor is used. The bright glow is present in the gap between the electrodes rather than over the electrodes, and corresponds to a high temperature arc discharge.

Figure 3c shows a picture of the discharge glow when a 22 kΩ limiting resistor was used. A plasma-like glow over the electrodes and an arc like glow in the gap region, corresponding to the arc/plasma portions of the discharge respectively can be clearly seen.

A handheld fiber optic spectrometer (USB 2000 from ocean optics) was used to evaluate the spectral characteristics of the device. A trigger pulse, generated in synchronism with the microdischarge pulse, was used to capture the pulse discharge spectra. The integration time on the discharge was set to 65 ms.

Figure 4a shows the spectra obtained when a plasma-like discharge was initiated in air. For this case a 100 kΩ limiting resistor was chosen. The discharge shows a strong emission in the UV-blue region, but very little emission in other regions of the spectrum.

Figure 4b shows the spectrum when an arc-like discharge was initiated using a 82 Ω limiting resistor. Strong lines in the red-infrared region of the spectrum, which were not present in the plasma spectrum, become apparent now, but many of the lines present in the UV-blue region of the spectrum are absent.

Figure 4c shows the spectrum in air when a hybrid discharge was initiated when using a 22 kΩ limiting resistor. This spectrum contains both long and short wavelength lines corresponding to both arc-like and plasma-like components.

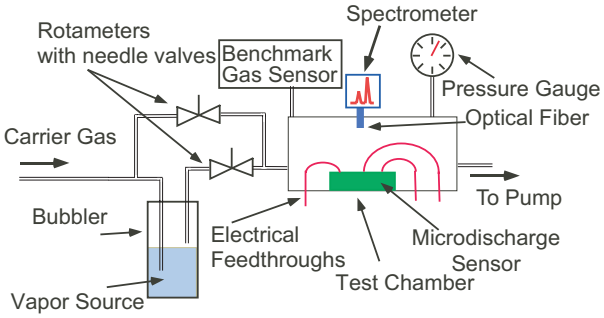


Fig. 5: Schematic of the test-setup for evaluating chemical sensor performance. The vapor concentration is adjusted by the flow rate through the vapor source relative to the overall flow rate. A MINIRAE 2000 vapor sensor is used as a benchmark.

C. *Chemical Characterization*

To evaluate the hybrid micro arc-plasma device as a vapors sensor, the device was placed in a chamber in which constant acetone concentration was maintained. Figure 5 shows a schematic of the setup. The vapor concentration could be adjusted by controlling the flow rate of the carrier gas through the vapor source relative to the total flow rate. Air was used as a carrier gas and the pressure inside the chamber was maintained at 810 torr. A vacuum pump was used to evacuate the chamber from impurities before the experiment, but was not used during the experiment. A commercial vapor sensor (MiniRAE 2000) was used as a benchmark.

The device was operated in hybrid mode with the limiting resistor set to 22 kΩ. A hybrid discharge was chosen as it had the strongest emission in the wavelength range of interest (350 nm-450 nm).

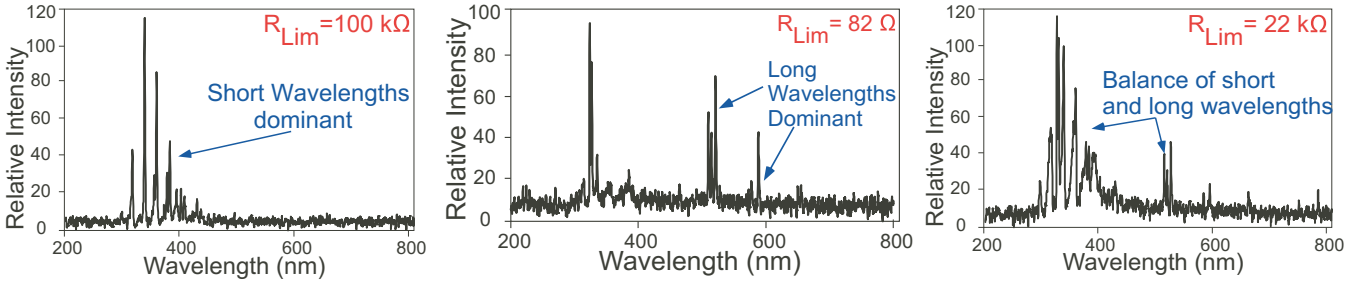


Fig 4a (left): Air spectrum with 100 kΩ limiting resistor, showing "plasma like" spectra with lines in the UV-blue region. Fig. 4b (mid): Emision spectrum with 82 Ω limiting resistor is "arc-like", with strong lines in the visible (green) region of the spectrum, but weaker lines in the UV-blue region. Fig. 4c (right): Emission spectrum with 22 kΩ limiting resistor is a mix of both plasma and arc-like spectra, having emission both in the UV-blue and green region.

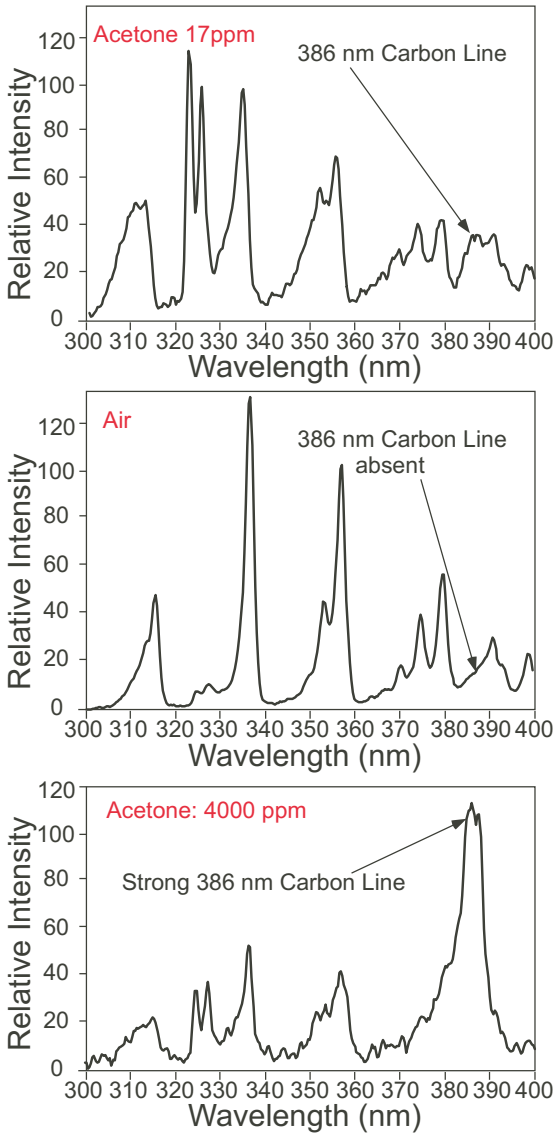


Fig. 6a (top): Spectrum of the microdischarge in 17 ppm acetone showing the presence of a line at 386 nm corresponding to carbon fragments from acetone. Fig. 6b (center): Emission spectrum of the microdischarge in air. The 386 nm line is absent. Fig. 6c (bottom): Emission spectrum in high concentration of acetone, showing pronounced 386 nm carbon line. Note that the spectra are normalized and on a relative scale. The nitrogen line intensity does not change, but the "carbon line" rises sharply compared to the spectrum in Fig. 6b.

Figure 6a shows a portion of the microdischarge spectrum (300 nm – 400 nm) taken in 17 ppm ambient of acetone. The spectrum shows a line at 386 nm that corresponds to carbon containing fragments produced from acetone. These lines are absent in the control spectrum taken in air (Fig. 6b). When the acetone concentration was increased to 4000 ppm, this line dwarfed all the other lines, which confirms that the line was due to fragments from the acetone (Fig. 6c). Note that the spectra are normalized to the strongest line. In the spectrum shown in Fig. 6c, the nitrogen lines seem diminished, not because the nitrogen emission is diminished, but because the carbon line is very intense due to the high vapor concentration.

IV. Conclusions

This work demonstrates that by tailoring the discharge gap, and drive circuitry, it is possible to control the balance between arc-like and plasma-like content of a microdischarge. At separations < 75 µm, micro-arcs and microplasmas can coexist. Micro-arcs have a wide spectral distribution in emission, while microplasmas have strong but narrow band emission mainly in the UV-blue region, when using air as the carrier gas. The micro-arc is driven by bursts of current while a steady but small current drives the microplasma. The relative contributions of both are adjusted by using a limiting resistor and carefully designed circuitry. The application of these discharges to chemical sensing is demonstrated by detecting 17 ppm of acetone in air ambient in a 65 ms detection time, without using any pre-concentration. This is accomplished using pulses of 60 mJ on a device with an active area of 300 µm x 300 µm and overall footprint of 10 mm x 10 mm.

Acknowledgments

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