DESIGN OF AN INJECTION-MOLDED IMPEDANCE CELL FOR MEASURING THE DIELECTRIC CONSTANT AND CONDUCTIVITY OF LIQUIDS AND GASES ACROSS ADJUSTABLE NANOMETER ELECTRODE GAPS

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INTRODUCTION
Accurate measurement of dielectric constant and conductivity, also known as, Electrochemical Impedance Spectroscopy (EIS) is a simple, yet powerful technique for analyzing the properties of liquids and gases. EIS is currently used in a wide range of industrial applications including monitoring the purity of boiler water in power plants [1], as well as, the health of lubricants in heavy machinery [2]. Conductivity measurements can also be used as a chemical detector by using a conductivity assay to gain selectivity over specific ions or compounds.

Recently, the idea of portable chemical analysis systems has gathered considerable attention as a way to reduce cost and expand access of laboratory technologies [3]. EIS is very attractive chemical detection technology for such systems because it is inherently electrical and therefore, can be easily integrated within microelectronic systems. Moreover, unlike many optical detection techniques, EIS does not require the use of lasers or spectrometers which can be expensive and bulky.

The apparatus used for measuring dielectric constant and conductivity, known as an impedance cell, is a geometrical arrangement of electrodes that allow a sample liquid or gas to be measured using an impedance analyzer. Existing conductivity cells, however, are not practical for use with portable devices because in order carry out measurements of desirable accuracy, typically 1% or better [4], these impedance cells require extensive cleaning and calibration, which are difficult to apply outside of a laboratory environment.

This paper explores the vision of creating disposable impedance cells that enable accurate measurements of dielectric constant and conductivity without calibration. The single-use nature of such devices would eliminate problems with contamination and the need for cleaning.

THEORY
A key problem in many EIS measurement systems is the extraction of the intrinsic values of dielectric constant and conductivity, $\varepsilon$ and $\kappa$, from measurable quantities of capacitance and conductance, $C$ and $G$. These quantities are related by the geometrical factor of the cell constant, or $K_{\text{cell}}$, and the fringing field capacitance and conductance, $C_0$ and $G_0$, according to the following equations,

$$C = \frac{\varepsilon}{K_{\text{cell}}} + C_0$$
$$G = \frac{\kappa}{K_{\text{cell}}} + G_0$$

Since the values of $C_0$ and $G_0$ are artifacts of electrode geometry and manufacturing process, contributions from these terms cannot be easily predicted. As a result, it is very difficult to determine $\varepsilon$ and $\kappa$ from geometrical calculations of $K_{\text{cell}}$. This problem is exacerbated by absorption on electrode surface which tend to change the effective size of the impedance cell. Existing conductivity meters typically address this problem by periodically calibrating the measurement apparatus using standard solutions with known values of $\varepsilon$ and $\kappa$. This correction is of limited value since the contribution from $C_0$ and $G_0$ are typically dependent on the value of $\varepsilon$ and $\kappa$. As a result, this calibration is valid only if the $\varepsilon$ and $\kappa$ of the sample and calibration liquids are similar. Furthermore, as discuss in the introduction, the use of calibration liquids is undesirable since the
calibration liquid may irreversibly contaminate the impedance cell.

The need for calibration liquids can be avoided using differential measurements of an array of impedance cells where the cell constant is varied in a predictable way. Such designs confront a fundamental tradeoff between varying the cell constant over a sufficient range in order to make an accurate estimate of $\varepsilon$ and $\kappa$ and minimizing the variation of $C_0$ and $G_0$. For impedance cell arrays that vary the electrode separation, this tradeoff is optimized when electrode gap is minimized, since small modulations on the electrode gap results in large variations of the cell constant.

Small electrode gap structures between planar electrodes can be difficult to fabricate since small parallelism errors between the electrodes can produce large errors in the measured results. These types of errors could be avoided using spherical electrodes since the nearest points between two spheres are by definition tangent with one another.

The cell constant between closely spaced spherical electrodes can be obtained from the expression for capacitance [5],

$$C = \pi r \varepsilon_r \varepsilon_0 \log \left( \frac{2r}{x} \right) + C_0,$$

where $x$ is the electrode separation, $r$ is the radius of the electrode, $C_0$ is a constant, $\varepsilon_0$ is the vacuum permittivity, and $\varepsilon_r$ is the relative dielectric constant. This expression has the convenient property that the spatial derivative of $C$ is inversely proportional to the electrode separation such that,

$$\frac{x}{\varepsilon_r} = -\pi r \varepsilon_0 \left( \frac{dx}{dC} \right) = y.$$

Thus, the value of the relative dielectric constant can be determined from a linear regression of $y$ versus $x$, where the value of $dx/dC$ can be obtained using the secant approximation.

**DESIGN OF THE TEST APPARATUS**

A test apparatus that approximate the geometry of the molded cell with adjustable electrode has been designed in order to validate the concept of an impedance cell using closely spaced spherical electrodes and to determine parameter such as range and tolerance of the electrode separation. The test apparatus consists of a spherical electrode, mounting shafts, sample chamber, and a mechanism for adjusting the electrode separation.

The spherical electrodes are fabricated by depositing a thin platinum film on one hemisphere of a silicon-nitride sphere. The silicon-nitride spheres have a diameter of 9.52564 ±0.00013 mm (Cerbec Saint Gobain Ceramics: Grade 5 spheres). The electrode film consists of a 50 nm platinum film with a 5 nm chromium adhesion layer.

![FIGURE 1. Cross section of the test impedance cell](image)

The electrode spheres are mechanically constrained in the tip of a mounting shaft using a ring nut as shown in Figure 1. Dimples on the face of the ring nut allow it to be tightened. In doing so, the ring nut preloads the sphere against the shaft and makes electrical contact with the platinum film. Measurement probes from the impedance analyzer are connected the electrodes via the mounting shaft.

The sample chamber is designed to constrain the sample liquid between the electrodes while allowing small modulations of the electrode separation. The sample chamber is a cube with a central bore. The mounting shaft slides through the bore and the sample liquid is sealed via Kalrez (Dupont) o-rings, which offers similar solvent resistance as Teflon, but with significantly greater flexibility.

The sample chamber is electrically isolated from the mounting shafts. The chamber, made of ANSI 316 stainless steel, is connected to ground and act as a guard electrode. The sample liquid is sealed within the sample chamber and electrode shaft.
The electrode separation is modulated by precise adjustment of the mounting shaft using a monolithic flexural stage, shown in Figure 2. Both mounting shafts are clamped to the flexure mechanism, and are electrically isolated via a 50 µm layer of polyimid film. The displacement of both mounting shaft along its axial direction is measured using a capacitance probe (Lion Precision).

The monolithic flexural stage consists of three functional blocks: a coarse adjustor, a fine adjustor, and a metrology frame. The coarse adjustor is a manual lead screw that displaces the bottom mounting shaft. Assuming this screw can be adjusted in 1° increments, the resolution of the coarse adjustor is 0.9 µm.

The fine adjustor consists of an actuator and a flexural transmission. The actuator is a servo motor and lead screw assembly with 50 nm linear resolution (Newport: LTA-HL). The flexural transmission is a cantilever flexure that reduces the travel of the actuator by a factor of 190:1, thereby improving the actuator resolution from 50 nm to 0.25 nm. The overall range of the fine adjustor is 50 µm. The fine adjustor design is also optimized to minimize parasitic motion along axes perpendicular to the axis of measurement. This is done by using a symmetric dual beam flexure to constrain motion along a single axis while using a flexure wobble pin to transfer displacement from the cantilever beam to the symmetric dual beam flexure.

The electrode mounting shafts are clamped to the adjustment mechanism between two half cylinders surfaces. During installation, the top shaft is rigidly clamped first while the bottom shaft is adjusted by the coarse actuator. After the desired coarse position has been reached, the bottom shaft is then clamped and further adjustment of the electrode spacing is accomplished through the fine adjustor.

The metrology frame include two capacitance probes for measuring the position of the electrode mounting shafts. As shown in Figure 2, the bottom capacitance probe uses the same attachment with the coarse adjustor. This probe is designed to be added only after completing the coarse adjustment.

EXAMPLE RESULTS
The feasibility of this impedance cell has been confirmed via dielectric constant measurements of air and methanol. Figure 3 shows the capacitance data from air where the horizontal axis is the position of the electrode and the vertical axis is \( y \) as defined previously. The relative dielectric constant is the inverse of the slope. The data points are more distributed at larger electrode gaps since the differential value of \( C \) is smaller at larger gaps. The slope is determined using a weighted least squares (WLS) fit. The measured dielectric constant of air is within 1% of the known value of 1.00.

Figure 4 shows the measurement of the dielectric constant of methanol at 21.6°C. This data show considerably less noise than the air data because of the larger dielectric constant therefore, larger capacitance signal. Figure 5 shows the spread of results from 20 repeated measurements of the relative dielectric constant for methanol. The results fall well within a ±1% range of the known value of 33.26 for this temperature. The correct determination of the
relative dielectric constant for two drastically different media confirms that calibration is not necessary in this impedance cell design.

INJECTION-MOLDED IMPEDANCE CELL

After the concept of the design was verified using the test apparatus, the design of the injection molded impedance cell was been developed. As shown in Figure 6, the impedance cell consists of a planar substrate and molded plastic cover. The planar substrate is a silicon or glass wafer with a thin metal electrode layer. The metal film is patterned via standard photolithography techniques using a transparency mask. The molded plastic cover contains partial spheres that are offset from the bottom electrode at several depths. The mold surfaces of the partial spheres are diamond turned to obtain nanometer smooth surfaces. The plastic cover is made from a rigid, solvent resistant plastic, such as Teflon. The metal film is deposited by electron-beam deposition which covers the inside of the molded plastic cover almost entirely except for a few places where the top electrodes need to avoid the metal traces on the bottom substrate. Electrical contact to the top electrode film is accomplished via metal-metal contact with the traces on the bottom of the substrate. The molded plastic cover and bottom substrate is assembled together by wicking in a cyanoacrylate adhesive. Finally, the bottom substrate can be mounted on a standard printed circuit board to interface with impedance measurement circuitry.

CONCLUSIONS

The design of a disposable impedance cell for measuring dielectric constant and conductivity of liquids and gases without requiring the use of calibration standards has been developed. Preliminary experiments show 1% absolute accuracy can be achieved using this design. A patent is currently pending on this device.

REFERENCES