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NIST Technical Note 1294

Two-Fluid Measurements on Thin Films

Frederick I. Mopsik

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[1] At Boulder, CO 80303.

[2] Some elements at Boulder, CO 80303.

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ERRATA

The enclosed **NIST Technical Note 1294**, **Two-Fluid Measurements on Thin Films**, has been found to contain two errors, as follows:

Page 9, line 16

Ten3dtwopi = 1.59155E-04

should read

Ten3dtwopi = 1.59155E+02

Page 10, lines 23 -26

D1 = G1 * Ten3dtwopi
D2 = G2 * Ten3dtwopi
D3 = G3 * Ten3dtwopi
D4 = G4 * Ten3dtwopi

should read

D1 = G1 * Ten3dtwopi/(Freq*C1)
D2 = G2 * Ten3dtwopi/(Freq*c2)
D3 = G3 * Ten3dtwopi/(Freq*C3)
D4 = G4 * Ten3dtwopi/(Freq*C4)

PLEASE NOTE THIS CORRECTION

NIST Technical Note 1294

Two-Fluid Measurements on Thin Films

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TWO-FLUID MEASUREMENTS ON THIN FILMS

INTRODUCTION

The problem of determining the dielectric constant, ϵ, of a solid introduces a dimensional problem not present when the dielectric constant of a fluid is required. For a fluid, a cell can be constructed such that the entire space between the measuring electrodes is filled first with air and then with the desired fluid. A simple ratio then determines the dielectric constant. This is clearly not a possible procedure for a solid.

For solids, other techniques are required. Even if the contributions from fringing fields are removed through the use of a guarded electrode, an estimate of the sample thickness is usually required. Since capacitance ratios can be determined to 0.001 percent with conventional transformer bridges, this thickness problem often is the limit to the measurement accuracy. It was to avoid this problem that the two-fluid method was developed[1],[2]. While the method was originally applied to samples several mm thick, it is even more useful for films that can be only 10 μm thick and, therefore, where uncertainty in the thickness is that much greater[3].

DEFINING RELATIONSHIPS

Consider the experimental setup shown in figure 1. A pair of electrodes is made such that the guard gap on the guarded electrode is sufficiently small so that the field lines between the electrodes remain parallel except just inside the gap. Then the effective electrical area of the guarded electrode, A, as determined by approximately the midpoint of the guard gap, defines the measurement area and is independent of electrode separation or any dielectric inside the electrodes. If a fluid is between the electrodes, then the capacitance is given by the customary equation

$$C = \epsilon_l \, \epsilon_0 \, \frac{A}{d} \, , \tag{1}$$

where ϵ_l is the dielectric constant of the medium filling the electrodes, ϵ_0 is the permittivity of free space, 0.088541 pF cm^{-1}, and d is the separation between the electrodes.

If a slab of dielectric material of dielectric constant ϵ and thickness t is inserted between the electrodes, and if the slab is sufficiently flat and parallel to the electrodes, as in figure 2, then the effective area of the electrodes is the same for both the inserted slab and the fluid, as the field lines remain parallel and the combination can be considered equivalent to two capacitors in series, whose total thickness is the original electrode spacing.

$$C = \frac{C_a C_b}{C_a + C_b} ,$$

$$C_a = \epsilon\, \epsilon_0 \frac{A}{t} , \tag{2}$$

$$C_b = \epsilon_l\, \epsilon_0 \frac{A}{d-t} ,$$

where C_a and C_b are the capacitances of the portions of space filled with the solid sample and the liquid respectively.

If four measurements are now made, C_1, only air, C_2, air plus sample, C_3, second fluid with dielectric constant ϵ_l, and C_4, second fluid plus sample, it will be found that there are sufficient conditions, knowing the value of the dielectric constant of air, ϵ_{air}, to solve for both ϵ for the sample and, if the electrical area is known, the sample thickness. The solution is

$$\epsilon = \frac{\epsilon_l\, C_4 \Delta C_2 - C_2 \Delta C_4}{C_4 \Delta C_2 - C_2 \Delta C_4} , \tag{3}$$

$$t = \frac{\epsilon_l\, C_4 \Delta C_2 - C_2 \Delta C_4}{C_2 C_4 (\epsilon_l - 1)} \cdot \frac{\epsilon_0 A}{C_1} , \tag{4}$$

where

$$\Delta C_2 = C_2 - C_1 ,$$

$$\Delta C_4 = C_4 - C_3 , \tag{5}$$

$$\epsilon_l = \epsilon_{air} C_3 / C_1 .$$

As a cross-check, the cell spacing can be computed from C_1 and the area of the cell. The only possible additional measurements are those necessary to estimate the dielectric constant of air: pressure, temperature and humidity. For the measurements made in this laboratory, the dielectric constant of air was computed from the measured barometric pressure and relative humidity in the laboratory. This value was then adjusted to the cell temperature. In these computations, dry air and water vapor are assumed to be ideal gases with constant molar polarizabilities using the Debye equation. For normal laboratory conditions at the National Institute of Standards and Technology (NIST), this gave a value of approximately 1.0006 for ϵ_{air}.

The above equations are derived with the assumption of lossless dielectrics. If loss is present, then the above equations are still valid except that the capacitances, C_i, must be replaced by the complex quantities $C^*_i = C_i(1 - iD)$. If the loss is small, so that $D < 0.1$, the above equations, using just the measured capacitances, are still valid and the loss can be estimated from the relation

$$\tan\delta = \frac{(\epsilon_l - 1)C_1C_2C_4}{C_4\Delta C_2 - C_2\Delta C_4}\left(\frac{D_2}{C_2} - \frac{D_1}{C_1}\right) , \qquad (6)$$

where D_1 and D_2 are the dissipation factors corresponding to the capacitances C_1 and C_2. An equivalent relation can be derived using the losses from measurements 3 and 4.

In practice, it is usually found that the two results using measurements 1 and 2 and 3 and 4 are not quite the same, due to probable surface leakage losses. In this laboratory when the loss measurements could be cross-checked on samples that had guarded, contacting electrodes, the loss using the air gap was found to be more reliable. For critical loss measurements, two fluid measurements should be combined with guarded, metallized samples, where the loss can be accurately determined but where the actual value of ϵ cannot.

MEASUREMENT REQUIREMENTS

For plane parallel, guarded electrodes with a small guard gap, the above equations assume a plane parallel sample and an inert second fluid. No parameter can change while the measurement takes place. For the cell or electrode set, which is described later, this implies good mechanical stability to mechanical stresses, the presence of any fluid, and any possible temperature fluctuations. The requirements will be discussed in turn.

Second Fluid

If many different materials are being measured, the choice of a second fluid can become a problem. Ideally, for best measurement sensitivity, the second fluid should come close to matching the dielectric constant of the material. This can be seen in eq (3) where ΔC_4 vanishes if the second fluid matches the sample and now ϵ/ϵ_{air} is simply the ratio of capacitances C_3/C_1.

Further illustration is shown in figures 3-5. The error multiplier to estimate the relative error in ϵ and t from the relative error of a capacitance measurement for both the thickness, t, and dielectric constant, ϵ, for a sample is plotted as a function of both the dielectric constant of the second fluid and the relative thickness of the sample. These values were computed numerically assuming a uniform random error for each of the four capacitance measurements. The clear minimum in the error multiplier for ϵ at match is easily seen. While the multiplier can get quite large for a high ϵ material in a fluid with $\epsilon_1 = 2$ where the gap is large compared to the sample, it should be remembered that with good measurement practice, the relative error in a capacitance measurement can be much better than 0.01 percent.

Even more importantly, the fluid must be stable and low-loss over time. Unfortunately this requirement usually eliminates most high dielectric constant liquids, because they absorb water vapor from the air and develop large losses as well as drift much too quickly over time. All the measurements made in this laboratory have used either heptane or a fluorinated liquid since they are quite stable and usually do not affect the measured film. While their low dielectric constants near 2 can increase the uncertainty in ϵ for high ϵ films, there is usually sufficient bridge accuracy so that 1 percent measurements can be made.

A strong requirement on the second fluid is that it not swell the film to be measured. Swelling not only physically changes the dimensions of the film at the time of measurement but can lead to permanent changes in the physical state of the film. Heptane not only can swell polypropylene, but can irreversibly change the orientation and dimensions of the film. Swelling can also lead to extraction of any additives found in the film, changing not only the film but the liquid itself. Swelling has usually been readily observed when the film is clamped at opposite ends in a U-frame by buckling or wrinkling. For all the materials measured in this laboratory to date, if heptane was not suitable, then a fluorinated fluid was usually found satisfactory.

Finally, the fluid must be compatible with the materials used to construct the cell. The cell holder described below uses epoxy resin to position the electrodes. Chlorinated solvents can attack the resin leading to mechanical instabilities as well as greatly increased conductivity in the fluid. A revised construction in which any adhesive is shielded from the fluid with an inert gasket would be an improvement.

Film Holder

The two-fluid measurement assumes a uniform film with reproducible properties. Any real film will have appreciable variations in thickness and dielectric properties as it is scanned along its surface. These variations will degrade the measurement by introducing additional experimental scatter. To counteract this error source, all films are measured in a holder that holds the film flat with the same area inserted in the cell. The holder consists of two matching aluminum plates in the form shown in figure 6. The film is clamped between the plates using small screws to hold the plates together. The holder fits in a shallow slot on the inside of the container centered between the cell electrodes with the depth controlled by the ears on the holder plates. Except for some angular tilt in the slot, the film has adequate reproducibility for the measurement. Repeated measurements on a polypropylene film using air as the fluid showed reproducibility to 0.01 percent in the capacitance.

Film Variations

The use of a film holder to reproducibly position the film does raise the question of the interpretation of the data for a film that cannot be assumed plane and parallel. To first order, any deviation from the film being parallel to the electrodes should be proportional to $1 / \cos \alpha$, where α is the angle of the film normal to the cell axis. For almost any allowable angle ($\alpha \leq 6°$) in a narrow gap, this error should be smaller than 1 percent and of no concern.

For variations in thickness across the film, the answer is more difficult since the presence of the film can affect the location of the field lines. If there is not much distortion of the field from that of the empty electrodes then the measured values would be averaged over the the area between the active electrodes. From eq (1), the measured thickness would be the mean of 1/d for the film being measured. Similarly, variations in the dielectric constant should be the simple arithmetic mean of the sample. For a given area, the result should be the properly weighted average for use in any electrical test.

MEASURING CELL

The key item in performing a two-fluid measurement is the cell. It must be rigid with a small guard gap so that the electrical area is well-defined, and once the spacing is set the capacitance must be stable. To minimize propagation of errors, the electrode spacing should be not much larger than 10 times the film thickness. For thin films, where a narrow gap is required, there is a stringent requirement for cell rigidity.

The cell design used in this laboratory was originally designed and built as a joint research project at NIST with the FMC Corporation by William P. Harris and Glen Oneal, Jr., and modified from the original published design[3]. A cross-section of the cell is shown in figure 7. It features guarded electrodes mounted in a cylindrical pipe that acts as the container for the dielectric fluid. The electrode spacing is adjustable and set by the use of shims between three lands in the low electrode, next to the assembly screws, and the mounting pipe. The entire assembly forms a well-shielded, three-terminal capacitor of good rigidity and convenient size. The only measurable capacitance is that between the guarded electrode and high, with an effective area that is essentially constant as the spacing is changed over the normal working range.

The cell is constructed entirely of stainless steel except for the ring used to dam the epoxy cement mixture between the guard and low electrodes, which is polytetrafluoroethylene. The low electrode assembly is held together by an epoxy mixture consisting of 85 pph 100 mesh silica and 15 pph Bliss Bond 100 [4] in the space from behind the dam to the top edge of the center electrode[*]. The center electrode originally had a thin step that just fit inside the guard gap. It was removed when the measuring face was ground and lapped flat after assembly. In the cell used by NIST, the nominal electrode diameter was 25.4 mm with a guard gap of 0.125 mm.

The outer electrode is held in place in the outer pipe using the same epoxy mixture. During assembly, a polytetrafluoroethylene ring between the high and low electrodes is used as a dam so that pressure can be applied to keep the high electrode flush with the low electrode when no shims are in place while the resin is cured. While this epoxy mixture is difficult to work with, due to the low resin content, it is designed to match the coefficient of expansion of the stainless steel.

Seepage of any fluid past the low electrode is prevented by a Polytetrafluoroethylene coated O-ring between the pipe and low electrode. This seal allows sufficient motion so that the opening between the electrodes can be easily adjusted by using hardened steel shims. Typical electrode separations can range from 0.1 to 0.3 mm. The minimum separation is set by the rigidity and friction characteristics of the film being inserted.

The only calibration that has to be done is to establish the effective electrode area[5] in order to define a dimensional scale. This can be done by using a set of shims in the range of 0.075 to 0.3 mm and any method that can measure the total extension of the cell, such as a calibrator and gage blocks. The capacitance is then measured at constant temperature as a function of change in separation. The data is fit with least squares, assuming that the capacitance times true separation is a constant, through the equation

$$C_1(t_1 - t_0) = \epsilon_0 A \ , \tag{7}$$

where C and t_1 are the measured values, t_0 is the computed effective origin of distance for the computed area, A.

MEASUREMENT PROCEDURE

The procedure for performing a measurement is quite simple but must be carried out in a proper order to minimize errors. The following sequence will assume that the film is not significantly swelled by the second fluid. For all measurements, it is assumed that the cell is at constant temperature. In this laboratory, this was achieved by inserting a container large

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[*] The mention of any commercial product does not constitute either an endorsement or a recommendation by NIST. It is only for information and to completely specify the procedures used in this report.

enough to hold the cell in a constant temperature bath set at 25 °C that regulated to 0.01 °C. The cell was allowed to equilibrate for several hours after any handling.

The first measurement is the capacitance and loss of the empty cell. At this time, the air temperature pressure and relative humidity are also recorded. Then the sample, mounted in a holder as shown in figure 6, is inserted in the cell and the second capacitance and loss measurement is recorded. At this time, another replicate set of measurements one and two can be made, or another set with another sample can be made if other samples are available.

After all the air measurements are recorded, the cell is filled with the second fluid. After the fluid reaches equilibrium temperature as demonstrated by constancy of the cell capacitance, measurements on all the desired films are taken in the order: sample in second fluid, second fluid. In this manner, if there is any contamination of the fluid by the film, the proper values for the fluid are obtained. After the last determination, the fluid may be discarded or stored for reuse if not much contamination is found.

After all the data are recorded, the data are entered in a computer program for reduction. The air temperature, pressure and relative humidity allow the dielectric constant of air to be computed[6]. The program has equations in it to compute the corrections to a brass scale barometer as well as the vapor pressure of water over a temperature range of 0 to 100 °C[7].

The cell can then be reused after all the residual fluid has evaporated. If the second fluid is not volatile, then a volatile rinse compatible with the epoxy cement should be used. With proper cell construction, the air capacitance should be highly stable to better than 0.001 pF for a 0.0010 cm spacing.

For reference, a copy of the program written in FORTRAN 77 is appended to this report. If the loss becomes large, the computation for ϵ should be redone as a complex computation by recasting the appropriate variables and the separate computation for loss can be omitted.

REFERENCES

1. Harris, W.P. and Scott, A.H., Precise measurement of dielectric constant by the two-fluid technique, 1962 annual report, conference on electrical insulation, NAS-NRC 1963.

2. Methods of test for AC loss characteristics and dielectric constant (permittivity) of solid electrical insulating materials, D-150, American Society for Testing and Materials, Philadelphia, PA 19103.

3. Oneal, G. Jr., Harris, W. P., Three-terminal cell for thin film dielectric measurements, 1969 annual report, conference on electrical insulation and dielectric phenomena, NAS-NRC 1970.

4. E. W. Bliss Co., 101 Chester Rd., Swarthmore, PA. The mixture requires pressure even after mixing to form a coherent, pore-free mass.

5. Harris, W.P., Precise determination of the area of guarded electrodes for accurate dielectric measurements on solid-disk specimens, 1963 annual report, conference on electrical insulation, NAS-NRC 1964.

6. Maryott, A. A., Buckley, F., Table of dielectric constants and electrical dipole moments of substances in the gaseous state, Nat. Bur. of Stand. (U. S.) Circular 537, (1953).

7. Wexler, A. and Greenspan, L., Vapor Pressure equation for water in the range 0 to 100 °C, J Res. Nat. Bur. of Stand. (U. S.) **75A**, 213 (1971).

APPENDIX A - DATA REDUCTION

```
C   Program to compute the dielectric constant, loss tangent and thickness
C   of a sample using the two fluid method.  The dielectric constant of air
C   is computed from the laboratory temperature, pressure and humidity.
C   The barometric pressure is corrected for a brass scale.
C   The cell is not assumed to be at laboratory temperature.
C   The loss is assumed to be low so that capacitance can be computed directly.

      CHARACTER FF*1, QUERY0*12, ANS*1, UPCANS*1, Title*80, LOSS*1
      FF = CHAR(12)
      M = 0
      OPEN (UNIT = 3, FILE = 'lpt1')
      Query0 = "Enter C and "
      Ten3dtwopi = 1.59155E-04

C  Check for which cell used to select proper electrode area

      WRITE (*,*) "Electrode [B] or [T]hin sample cell? "
      READ (*,*) ANS

C   Subroutine UPC converts to upper case

      CALL UPC(Ans, UPCANS)
      Ans = UPCANS
      Area = 0
      IF (ANS .EQ."T")  Area = 4.5228
      IF (ANS .EQ. "B") Area = 11.318 * .885418
100   CONTINUE
        M = M + 1
        WRITE (*,*) "Enter title: "
        READ (*,8001) Title
        WRITE (*,*) "Enter freq. in kHz: "
        READ (*,*) Freq
        WRITE (*,*) "Enter temp. of Cell: "
        READ (*,*) Tempcell
        WRITE (*,*)
     &    "Enter Temp. of air, Barometer, and Rel Humid in %: "
        READ (*,*) Tempair, Pressb, Relhumid

C  Correct pressure for brass scale using least squares fit to standard
C  table
```

```
  Press = PRESSB-PRESSB*(0.0001625+1.75E-10*TEMPAIR+
&    3.512E-11*(TEMPAIR**2))*TEMPAIR
  CALL EPSAIR(EPSA, Tempcell, Tempair, Press, Relhumid)
  WRITE (*,*) "D or G? "
  READ (*,*) ANS
  CALL UPC(Ans, UPCANS)
  Ans = UPCANS
  WRITE (*,*) Query0 // ANS // " for air: "
  READ (*,*) C1, G1
  WRITE (*,*) Query0 // ANS // " for sample + air: "
  READ (*,*) C2, G2
  WRITE (*,*) Query0 // ANS // " for liquid: "
  READ (*,*) C3, G3
  WRITE (*,*) Query0 // ANS // " sample + liq.: "
  READ (*,*) C4, G4
  Loss = ANS
  IF (Loss .EQ. "D") THEN
     D1 = G1
     D2 = G2
     D3 = G3
     D4 = G4
  ELSE
     D1 = G1 * Ten3dtwopi
     D2 = G2 * Ten3dtwopi
     D3 = G3 * Ten3dtwopi
     D4 = G4 * Ten3dtwopi
  ENDIF
  E0 = C3 / C1
  C5 = C2 - C1
  C6 = C4 - C3
  E1 = E0 * C4 * C5 - C2 * C6
  E1 = E1 / (C4 * C5 - C2 * C6)
  E1 = E1 * EPSA
  C9 = (E0 - 1) * C1 * C2 * C4 / ((C4 * C5) - (C2 * C6))
  D8 = C9 * (D2 / C2 - D1 / C1)
  T8 = EPSA * Area / C1
  T = (C2 - C1) / C2 * E1 / (E1 - EPSA) * T8
  WRITE (3, 8000) Title
  WRITE (3, 8000)
  WRITE (3, 8010)  Tempcell,  Freq, Pressb
  WRITE (3, 8000)
  WRITE (3, 8020) Tempair,  Press,  Relhumid
  WRITE (3, 8000)
  WRITE (3,8040) loss, loss
```

```
      WRITE (3,8050) C1, G1,  C2, G2
      WRITE (3,8000)
      WRITE (3,8060) Loss, Loss
      WRITE (3,8050) C3, G3, C4, G4
      WRITE (3,8000)
      WRITE (3,8070)
      WRITE (3,8050) E1, D8, T
      WRITE (3,8000)
      WRITE (3,8000)
      WRITE (3,8000)
      WRITE (*,*)  "New sample? "
      READ (*,*) ANS
      CALL UPC(Ans, UPCANS)
      Ans = UPCANS
      IF (ANS .NE. "N" )THEN
         M = MOD (M, 3)
         IF (M .EQ. 0) THEN
            WRITE (3,8002)  FF
         ENDIF
         GO TO  100
      ENDIF
     WRITE (3,8002)  FF
8000  FORMAT(1XA)
8001  FORMAT(A)
8002  FORMAT('&',A)
8010  FORMAT (1X"Temp = ",F6.2, 5X"Frequency = ", F6.3, " kHz ",
   &     5x"Barometer =", F5.1 )
8020  FORMAT (1X"Air Temp = ", F6.2, 5X"Press. = ", F5.1,
   &     5X"Humidity = ", F4.1, "%")
8040  FORMAT(6X "C(air)", T20, A1, T34, "C(air + sam)", T49, A1)
8050  FORMAT(1X4G15.6)
8060  FORMAT (6X"C(liq)", T20, A1, T34, "C(liq + sam)", T49, A1)
8070  FORMAT( 6x"Epsilon", T19," Tan d", T34," Thick(mm)")
      END


      SUBroutine EPSAIR (EPS, Tempcell, Tempair, Press, Relhumid)

      DATA F0, F1, F2, F3/ -7246.58, 77.6412, 5.74471E-03, -8.24704/
      T9 = Tempair + 273.15

C  Vapor pressure eq (18a),  A. Wexler and L. Greenspan
C   J . RES. NBS  75A, 213 (1971)
```

```
     V0 = F0 / T9 + F1 + F2 * T9 + F3 * LOG(T9)
     V0 = EXP(V0)
     V0 = V0 * 760 * Relhumid / 1.01325E+07
     B1 = 4.31601E-03
     B0 = 20.847 / (Tempcell + 273.15) + .0039
     V1 = Press - V0

C  Constants for molar polarizabilities derived from  NBS Circular 537 (1953)
C  Values taken are for dry air and water.

     P9 = B1 / 24.3009 * V1 / 760 * 296.15 / (Tempcell + 273.15)
     P8 = B0 / 24.3009 * V0 / 760 * 296.15 / (Tempcell + 273.15)
     P9 = P9 + P8
     EPS = 3 * P9 / (1 - P9)
     EPS = EPS + 1
   RETURN
    END
```

APPENDIX B – FIGURES

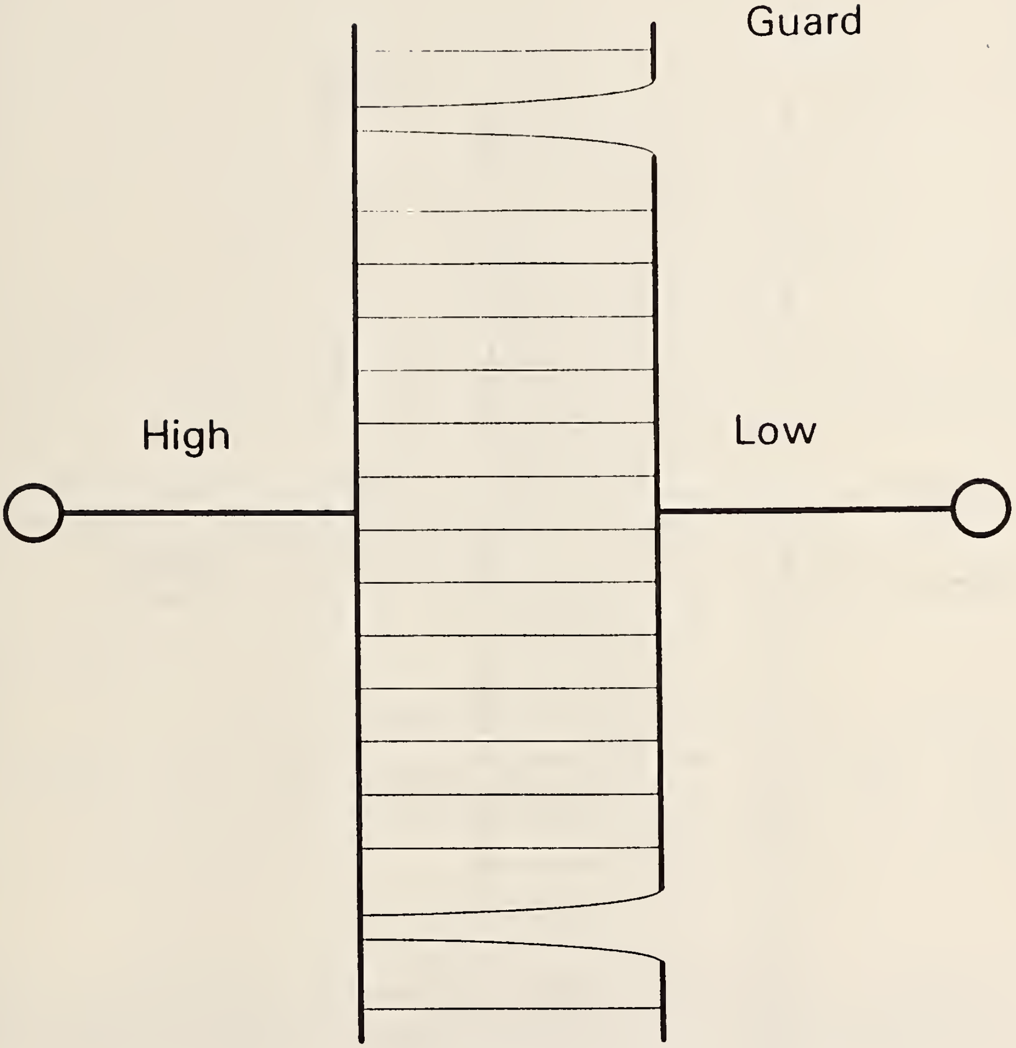

Figure 1. The guarded electrode configuration. The thin lines connecting the electrodes represent the flux lines. For a small guard gap they remain nearly parallel and evenly spaced as if the guarded electrode were continuous.

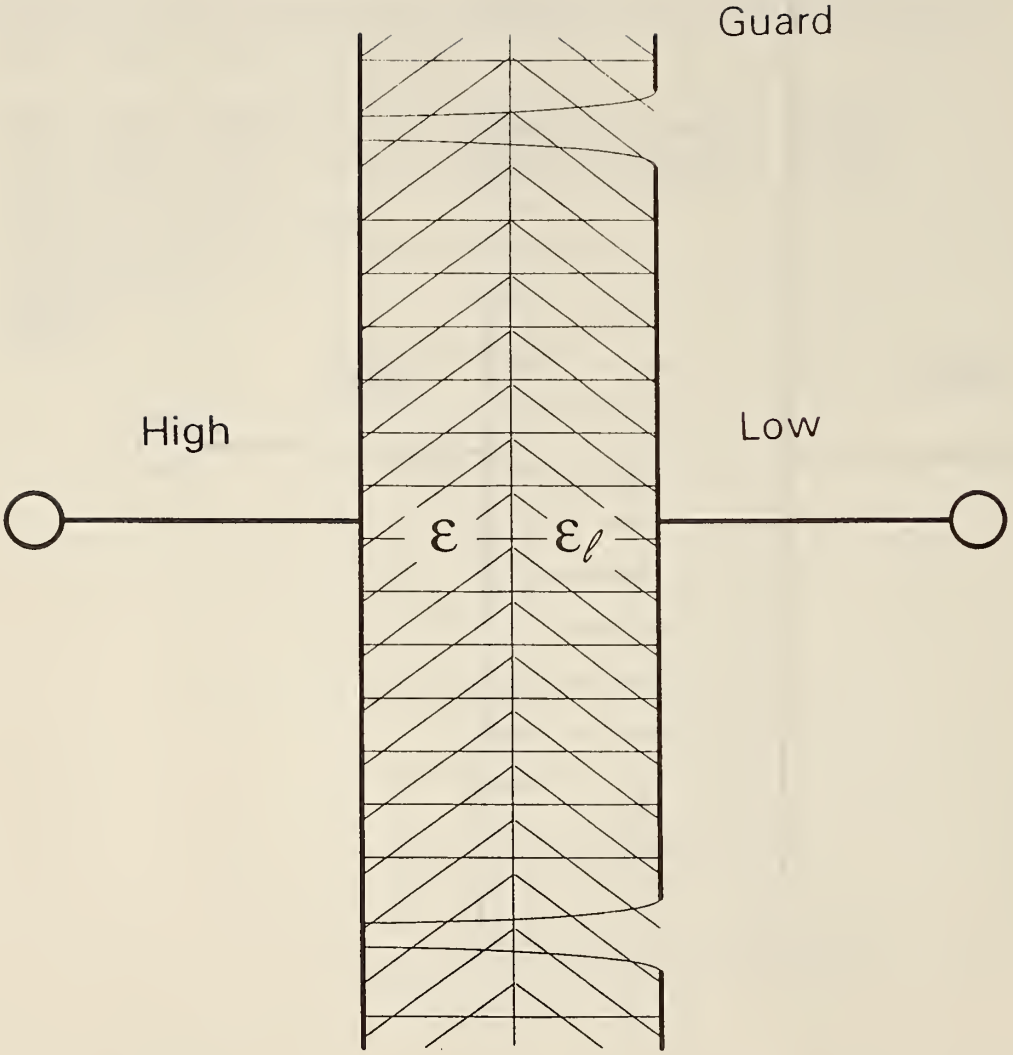

Figure 2. The guarded electrode with a solid sample, ϵ, in a fluid, ϵ_l, inserted. The flux lines shown remain nearly unchanged as they must if they are perpendicular to the interface.

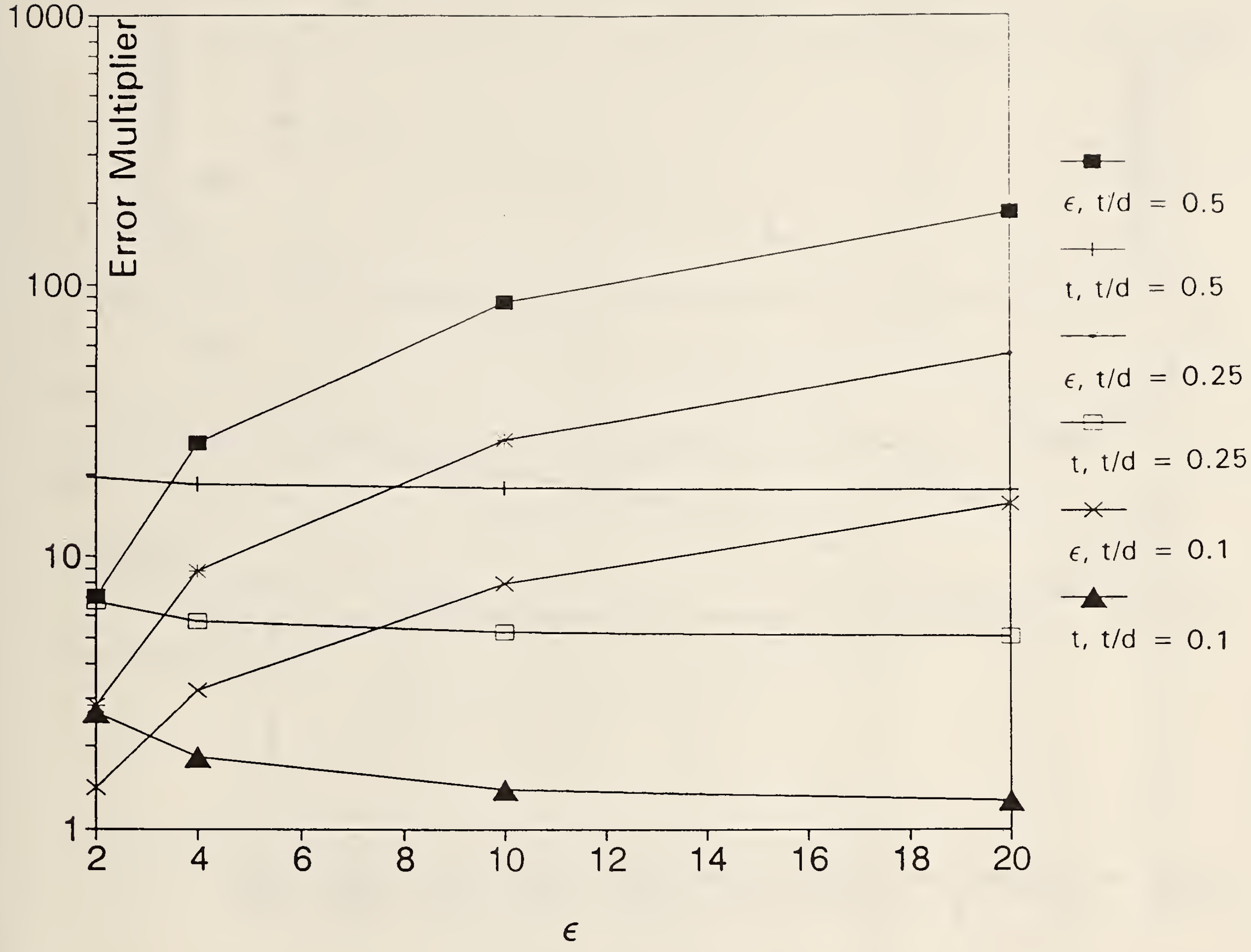

Figure 3. The relative error multiplier assuming constant error in the four capacitance measurements as a function of sample dielectric constant, ε, and sample thickness, t, relative to the electrode separation, d. The second fluid is assumed to have a dielectric constant of 2.

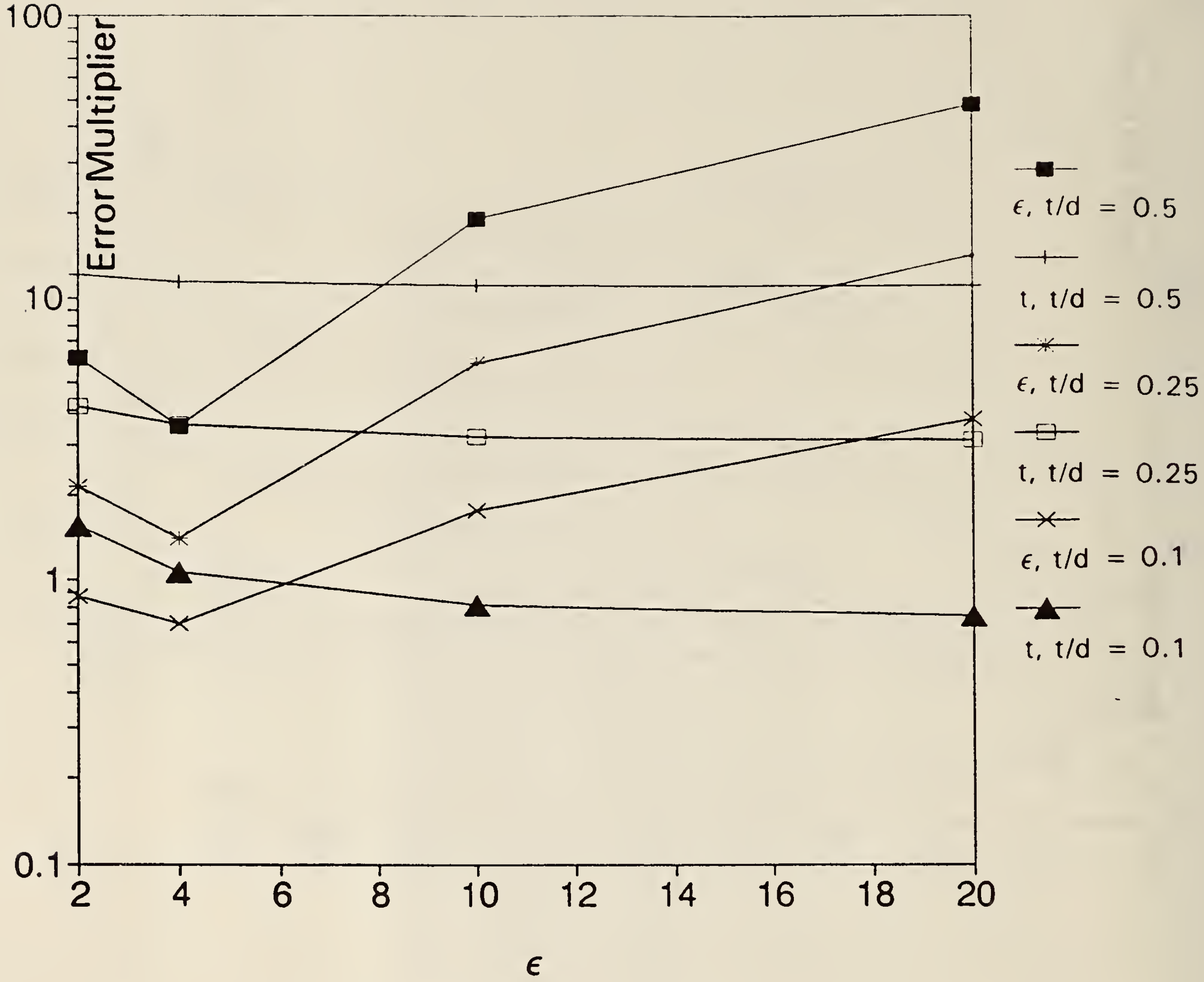

Figure 4. The same computation as in figure 3 with a second fluid dielectric constant of 4.

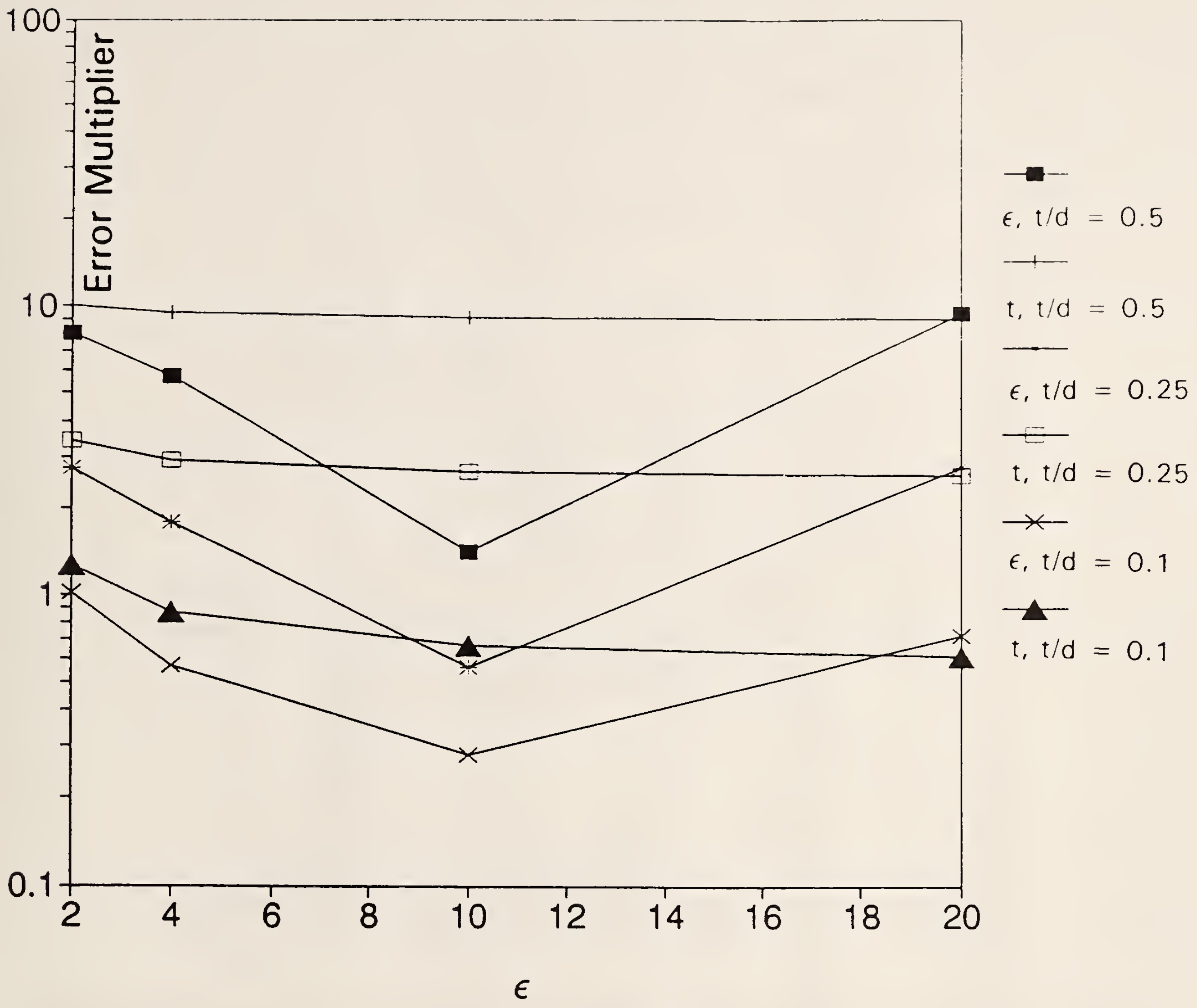

Figure 5. The same computation as in figure 3 with a second fluid dielectric constant of 10.

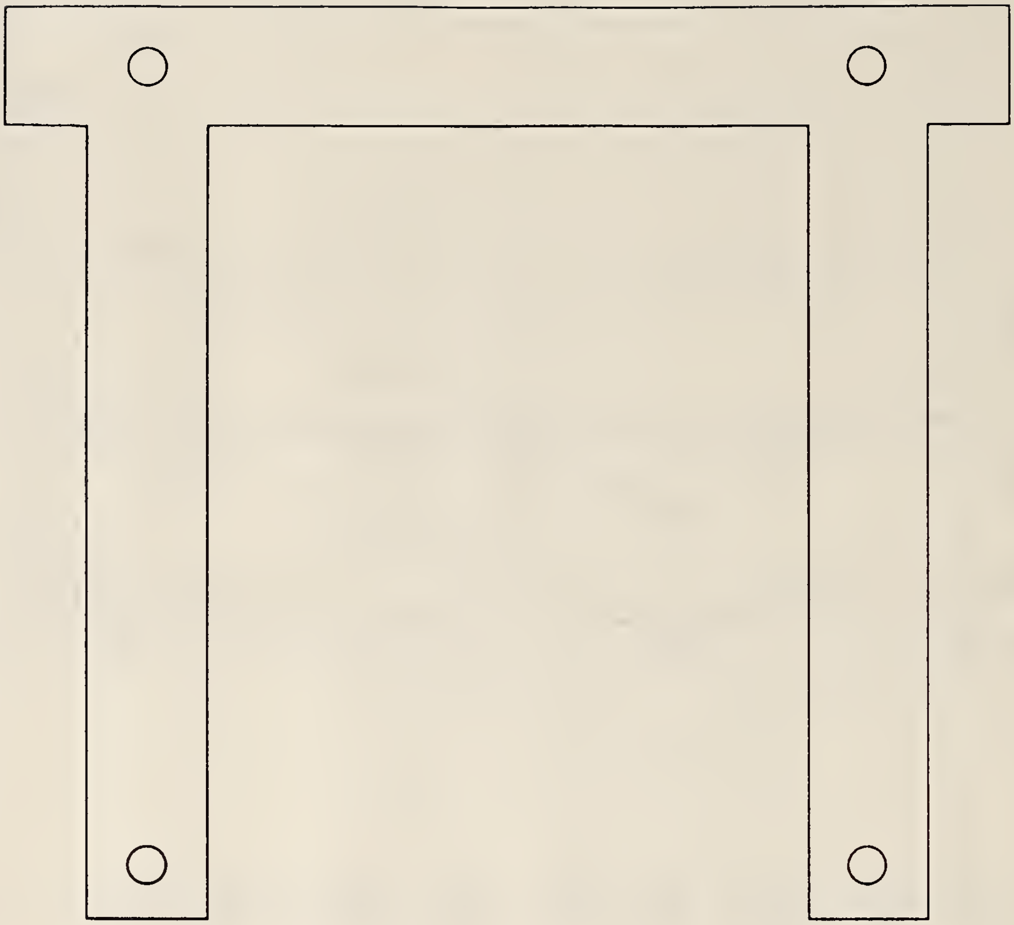

Figure 6. The thin film sample holder. The two plates of aluminum are held together with screws and clamp the film for insertion.

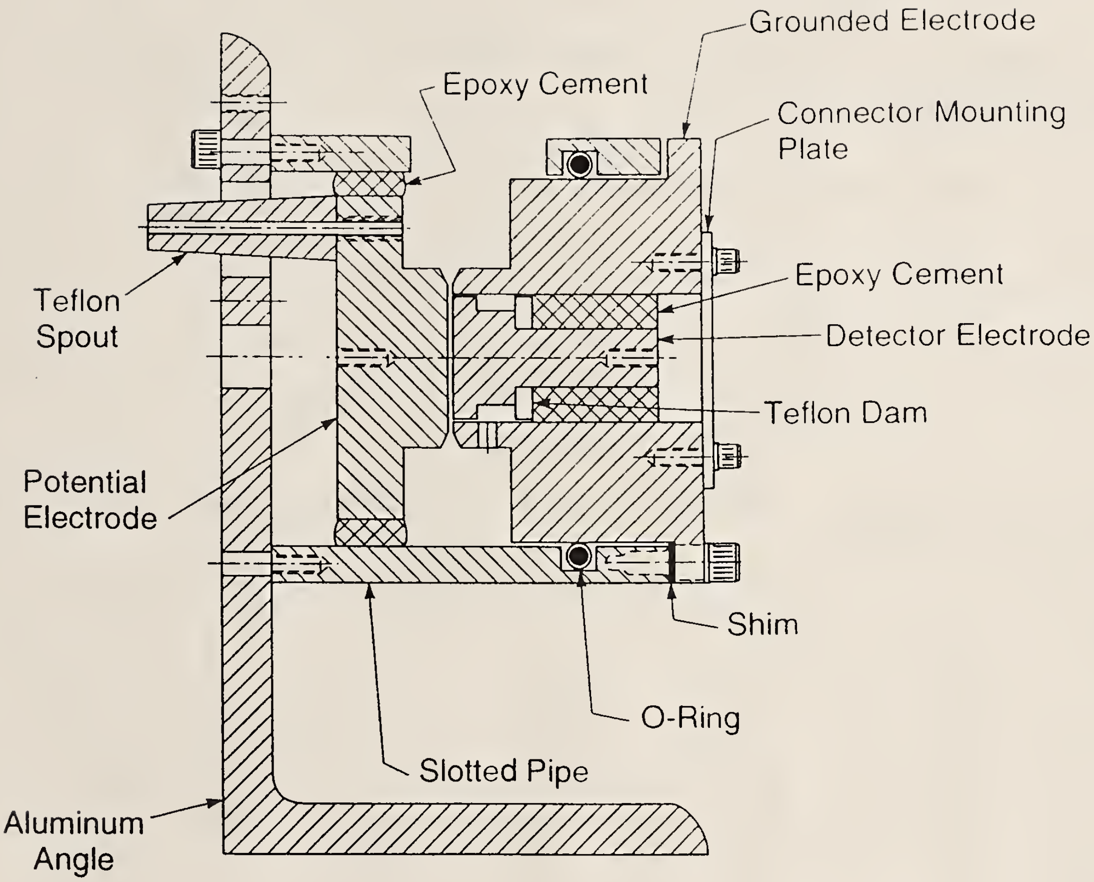

Figure 7. The cross section of the thin film two-fluid dielectric cell. There are three shims (only one is shown) distributed around the slotted pipe. All material is stainless steel unless indicated otherwise. The O-ring is polytetrafluoroethylene covered. The cell was assembled so that the gap between the electrodes was nearly zero without any shim present.

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The two-fluid technique to measure the dielectric constant and thickness of a thin polymeric film is discussed. The advantages include the ability to make a non-contacting measurement both of the effective electrical thickness of the film as well as the dielectric constant. The requirements for an accurate measurement are examined and the error as a function of the cell spacing, sample thickness, and dielectric constant of the second fluid are evaluated. The specifications of both the cell and the second fluid are examined. For the cell, it must be stable to good accuracy with handling, settable to small gaps and have a well-defined electrode area through the use of a guard ring with a narrow guard gap. A design of a holder that is suitable for films from 6 μm to 50 μm is illustrated.

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