Guarded needle for "charge injection" measurement

Yang Cao[a)] and Grace Ge Jiang[b)]
Electrical Insulation Research Center, University of Connecticut, 97 North Eagleville Road, Storrs, Connecticut 06269-3136

Steven Boggs[c)]
Electrical Insulation Research Center, Electrical Engineering and Physics Departments, University of Connecticut, 97 North Eagleville Road, Storrs, Connecticut 06269-3136 and Department of Electrical and Computer Engineering, University of Toronto, Toronto, Ontario M5S 1A1, Canada

The ability to measure resistive current resulting from high-field carrier mobility in dielectrics provides insight into transport phenomena and high-voltage performance. The threshold electric field for the formation of a space-charge limited field region under highly inhomogeneous field conditions has fundamental significance in that above this field, rapid degradation takes place from hot electrons and UV photons generated by carrier recombination. A guarded needle electrode with a tip radius in the range of 5 μm and an electrical guard which extends to within about 35 μm of the tip provides the basis for measuring high-field mobility as the resistive current can be measured independent of the displacement current. The residual displacement current resulting from the fF capacitance from the needle tip to the opposite plane electrode is compensated out of the measurement. In this article, we provide construction details for such a guarded needle. © *2002 American Institute of Physics.* [DOI: 10.1063/1.1492000]

I. INTRODUCTION

The guarded needle measurement technique was first developed at the Corporate Research Laboratories of Asea Brown Boveri Ltd. (ABB).[1,2] The implementation described in this article was developed with the help of ABB which provided detailed schematics for the measurement apparatus. ABB also provided a general description of needle construction; however, a good 12 months of concentrated effort was required by the authors before "good" needles could be made routinely. Given the great difficulty in fabricating such guarded needles, the authors felt that a detailed description should be available in the literature.

Defect-induced degradation of solid dielectrics often involves a high-field region of localized high carrier mobility during the formation of a space-charge limited field region in the vicinity of impurities or inclusions which serve as local stress enhancements.[3,4] However, the carrier mobility-related prebreakdown phenomena can only be studied within microscopic dimensions, as the power dissipation would cause thermal runaway for a macroscopic geometry. The guarded needle apparatus is designed for such measurement, using a sharp metal needle to create a local high electrical field, which induces carrier mobility. The "resistive" current is measured by guarding the needle to within 30 μm of its tip and compensating electronically for the residual displacement current.[1,2] However, due to the difficult nature of such

a measurement, only limited results have been reported.[2,5,6] The fabrication of the guarded needle electrode is the main obstacle of this experiment. In this article, the details of guarded needle fabrication are provided. The charge injection in commercial polybutadiene with the guarded needle is analyzed as an example of such measurements.

II. BASIS OF "CHARGE INJECTION" MEASUREMENTS

A. What is measured?

In the guarded needle measurement, the needle is inserted into an insulating sample, the other side of which is supported by a plane electrode. A time varying voltage is applied between the needle (at ground potential) and the plane electrode. The variation in the charge induced on the very small unguarded tip of the needle, less the charge caused by displacement current, is measured as a function of time. The residual capacitive (displacement) current between the needle tip and the plane electrode is canceled by a compensation circuit which is driven from a high-voltage divider. The basic measurement circuit is shown in Fig. 1. Thus, this measurement does not determine the charge "injected" from the needle tip into the sample. Indeed, a signal could be generated by carrier mobility within the sample with no charge transfer between the needle tip and the sample. What the measurement determines is the change in the "noncapacitive" image charge on the needle tip as a function of carrier mobility in the sample. Given the complex sample geometry (needle to plane electrode) and the strong field dependence of the sample conductivity at typical measurement fields in the range of 200 kV/mm, the measured signal can only be predicted with reasonable accuracy using software

[a)]Present address: GE, Research Center, Niskayuna, NY 12309; electronic mail: yang.cao@ieee.org
[b)]Present address: 3M, Electrical Products Division, Austin, TX 78726; electronic mail: gjiang@mmm.com
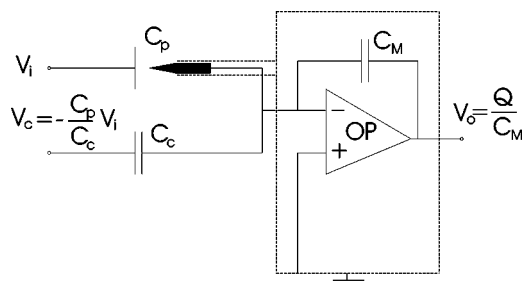[c)]Electronic mail: steven.boggs@ieee.org

FIG. 1. Conceptual schematic of the measurement circuit. V_i is the input high voltage. C_c is the compensation capacitor (10 pF polystyrene capacitor); V_c is the compensation voltage derived from the high-voltage divider (1000:1). After compensation, the remaining resistive current is integrated by the current–voltage integrator (AD 549) into a charge signal.
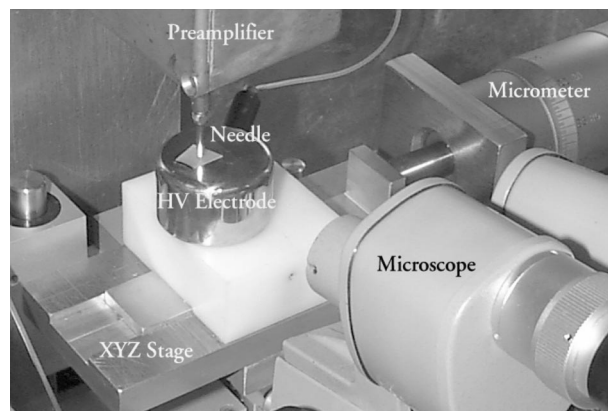

FIG. 2. The principal components for charge injection measurements with guarded needle electrode. Shown in the photo are HV electrode, preamplifier, guarded needle electrode, *XYZ* stage, and microscope.

for transient nonlinear finite element analysis, and the use of such software allows comparison between predicted and measured data as a function of conductivity model.[7]

B. Measurement overview

The guarded needle used to make such measurements has a coaxial structure with the needle tip protruding from one end. The tip of the needle protrudes from the guard structure about 30 μm, so that the capacitance from the needle tip to ground is reduced from about 10 pF to a few fF. The residual capacitive current can be compensated electronically as shown schematically in Fig. 1. This allows resistive current in the fA range to be measured without interference from the displacement current.[1] A function generator is used to provide a unipolar, single shot ramp, sine or triangular signal, or a continuous wave form. The unipolar wave form can be either negative or positive. The output of the function generator is passed through an audio amplifier and polarity reversing switch to a 100:1 stepup transformer, the output of which is connected to the plane electrode opposite the needle. Normally, triangle wave forms are applied with peak voltage typically in the range of 1 to 5 kV. A high voltage probe (Tektronix P6015A, 1000:1) is used to derive the input for the compensation circuit. The voltage signal from the probe is buffered, inverted, and adjusted through a single stage Kelvin–Varley divider to feed into the compensation capacitor as shown in Fig. 1. The remaining resistive current is integrated by an operational amplifier to generate the charge signal which is proportional to the change in induced charge on the needle tip. A digital oscilloscope (or a computer with A/D board) is used for data acquisition.

Figure 2 shows the main components of the measurement, including the high-voltage electrode, preamplifier, guarded needle electrode, and microscope. As shown in Fig. 2, the *XYZ* stage allows the needle to be inserted at various locations to a controlled depth in a dielectric with the aid of the optical microscope. The construction details of the guarded needle will be discussed next.

III. CONSTRUCTION OF GUARDED NEEDLE

The guarded needle has a coaxial structure with the needle as the central conductor and a tubular metal member, typically thin wall stainless-steel (SS) tubing, as the ground shield, which is used as a guard in the experiment. The tip region of the needle is coated with a thin layer of epoxy or a polymer which grows spontaneously on metal from a solution. Mineral-filled epoxy is applied between the SS tube and the polymer coated needle. The filled epoxy and polymer coating on the needle are then metallized to provide a continuous guard from very near the tip of the needle to the SS tube. The means by which a very small region near the needle tip is left exposed (bare metal) is described in detail next.

A. Epoxy insulated needle

The steel needles are supplied by Ogura Jewel Industry Co. Ltd., Tokyo, Japan, with a fixed cone angle of 30° and a spherical tip radius of 5 μm. The objective of guarded needle preparation is to provide a conducting guard over the needle to within about 30 μm of its tip. The upper part of the needle is guarded by a 3 mm diameter thin wall SS tube. The SS tube is connected to the needle using mineral-filled epoxy. The needle surface very near the tip is guarded by a ~2 μm layer of epoxy. Once the needle structure is assembled, it is coated with chromium to provide a continuous conducting layer, which forms a continuous conducting guard from the SS tube down to about 30 μm from the needle tip.

The region within about 30 μm of the tip of the needle is coated with nail polish prior to the application of the epoxy coating. This allows the chromium, epoxy, and nail polish to be removed from the tip region to form the final needle configuration, with about 30 μm of the needle tip exposed and a conducting guard electrode over the remainder of the needle.

Preparation of the guarded needle electrode can be divided into following steps.

(1) The raw metal needle is preselected based on tip quality using a scanning electron microscope (SEM, Philips ESEM 2020). Each tip needs to be examined twice, to see both sides of the needle. Only about 25% of the raw needles have a sufficiently good tip configuration and smooth surface contour for making a guarded needle electrode. Figure 3(a) shows a typical good raw needle with machine scratches.

(2) The needle shank is cut to 38 mm long. Then, the needle is tip polished with a mixture of 1 μm alumina powder and 0.3 μm alumina abrasive in a water solution (1:3 in
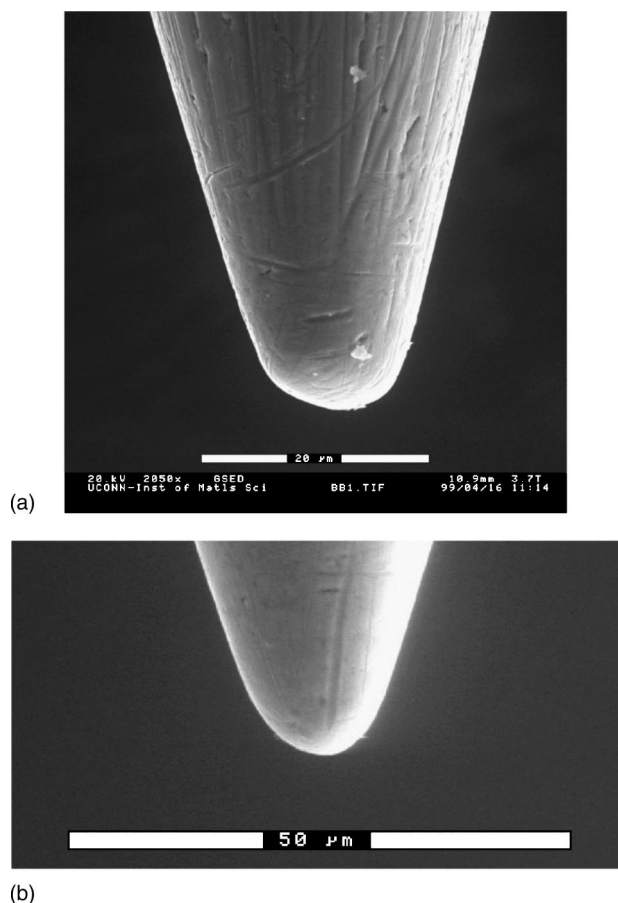
(a)

(b)

FIG. 3. (a) The raw 5 μm metal needle tip; (b) the same tip after polishing, cleaning in an ultrasonic bath with acetone, and coating with chromium.

10 wt % of water) for 20 to 30 min using a Dremel tool to rotate the needle in the polishing solution. The needle is cleaned in an ultrasonic bath with acetone for 15 min. The needle shank needs to be cleaned carefully if the polymer coating is to be used. Care must be taken to prevent needles from bumping into the container wall during the ultrasonic cleaning. The needle is checked again using a SEM, with typical results as shown in Fig. 3(b).

(3) The steel needles rust easily, and the prepared needle is usually coated with evaporated Cr, which provides good adhesion and corrosion resistance.

(4) This procedure needs to be performed under a microscope, such as the previously described *XYZ* stage and stereo microscope. The needle is partially assembled using the needle shank, SS tube, and spacers. The assembly is inserted in the LEMO LUS24337 connector of the preamplifier. The *XYZ* stage is used to dip the needle tip into a droplet of nail polish. The key to achieving no more than 30 μm coverage of nail polish over the tip is to dip the needle into a nail polish droplet with the proper viscosity. When the needle tip is very close to the surface of the nail polish, the center of the droplet will be lifted by the surface tension from the needle tip. If the nail polish is too thin (dilute), nail polish will wet the needle tip, resulting in a coverage much larger than 30 μm. On the other hand, if it is too thick (viscous), the nail polish will not stick to the tip. Nail polish with the right viscosity will be lifted slightly by the needle tip, resulting in
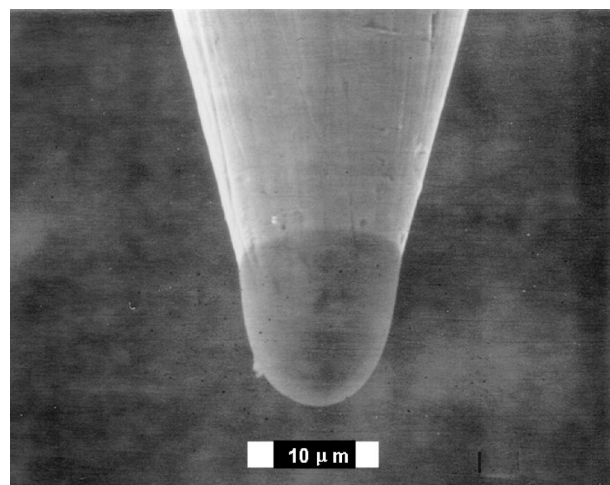

FIG. 4. The needle tip with less than 30 μm tip region covered with nail polish.

a proper coating of less than 30 μm from the tip. According to our experience, the proper time to let a small drop of nail polish thicken is around 1 min at 28 °C. After coating, the tip is checked under a higher-power optical microscope. The SEM photo of a tip covered with nail polish is shown in Fig. 4. If needed, the nail polish can be removed using a cotton pad with the aid of acetone.

(5) The SS tube is 3 cm long with 2 mm inner diameter (ID) and 3 mm outer diameter (OD). The end of the tube close to the needle tip is sharpened to prevent a step along the needle. The needle shank is 38 mm long and 1 mm in diameter. Teflon spacers (1 mm ID, 2 mm OD) are placed over the needle shank and into the SS tube. After assembly, the needle sticks out the SS tube 2 mm at the tip end. Figure 5 shows the photo of Teflon spacers, the punch metal piece for making the Teflon spacers, and a SS tube with one end sharpened. The needle is fixed in the tube using mineral-filled epoxy, which has less thermal expansion than unfilled epoxy. The epoxy used is Bondo 5 min epoxy resin mixed thoroughly with fine alumina power, 1:1 by volume, after which the hardener is added. After degassing under a vacuum of 0.1 Torr for 30 s, the epoxy is applied to fill the space between needle shank and the steel tube, leaving the needle tip sticking out only around 0.5 mm from the filled
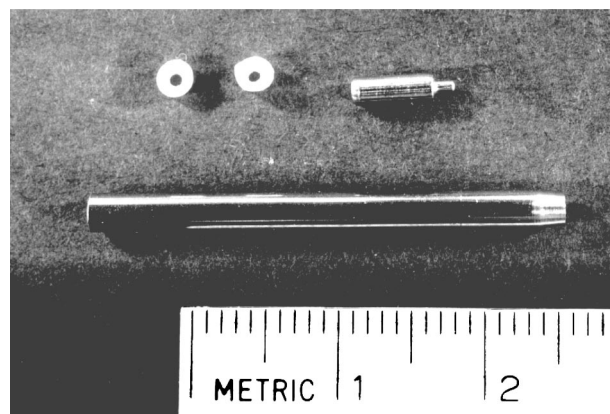

FIG. 5. The SS tube with one end sharpened. Shown also in the photo are the Teflon spacers and the metal punch used for making the spacer.
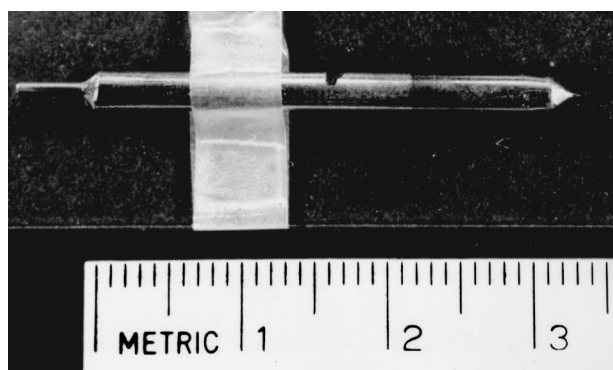
FIG. 6. A fully assembled needle. The sharpened end of the SS tube is fixed using the filled epoxy, forming a smooth cone structure. The needle tip sticks out only around 0.5 mm from the filled epoxy. The space between the other end of the SS tube and the needle shank is also fixed with the filled epoxy. The notch on the body of the SS tube is to prevent the pressure building up during the coating of the Cr shield.

FIG. 7. The needle tip coated with epoxy after being assembled into the tube. The objective is to form an epoxy coating which is ~2 μm thick.

epoxy. The surface tension of the epoxy leads to a smooth conical surface between the SS tube and the needle shank. The epoxy is cured at room temperature (RT) for at least 1 h, with the tip pointing downward. An assembled needle is shown in Fig. 6, with both ends fixed with mineral-filled epoxy. The hole in the body of SS tube is to vent the coaxial cavity during the coating of the Cr shield.

(6) The previous step leaves about 0.5 mm of bare tip to be coated with ~2 μm thick of epoxy insulation. This is done by applying several layers of Decon 2-Ton epoxy. The Decon 2-Ton epoxy cures totally in 8 h at RT, or 2 h at 55 °C. After mixing the resin and the hardener thoroughly, the needle is coated by dipping into the mixture under the microscope. The viscosity of the epoxy is 105 Poise at 30 °C, measured 1 min after mixing. This will form a uniform thin coating on the needle tip. The epoxy needs to be half cured prior to the next coating. Normally, it is cured at RT for 4 h or at 55 °C for 1.5 h with the tip pointing downward. Typically, six to eight layers of epoxy coating are required. The needle must be handled with great care, as the tip can be damaged easily. After the last coating, the epoxy is cured fully at RT for 8 h or at 55 °C in an oven for 2 h. Figure 7 shows a photo of a needle tip coated with epoxy insulation.

(7) After the epoxy is fully cured, chromium is sputtered/evaporated onto the needle. The needle must be rotated during coating or must be coated in several steps to form a continuous coaxial coating of the needle tip region, exposed needle shank, conical filled epoxy region, and adjacent end of the SS tube. The coating is performed with chromium coating rod (R.D. Mathis, CRW-1, 40) every 45° of rotation for 20 s at 1.4 A at a distance of 10 cm from the source. A low resistance uniform conducting coating is crucial for making good needles. Whenever a needle cannot be well compensated, chances are very high that the conducting coating is poor. A chromium coating is more effective than Al, Ag, or Au as it has the best adhesion to the relevant surfaces among conventional evaporating metals, although the high temperature at which it evaporates requires good thermal propert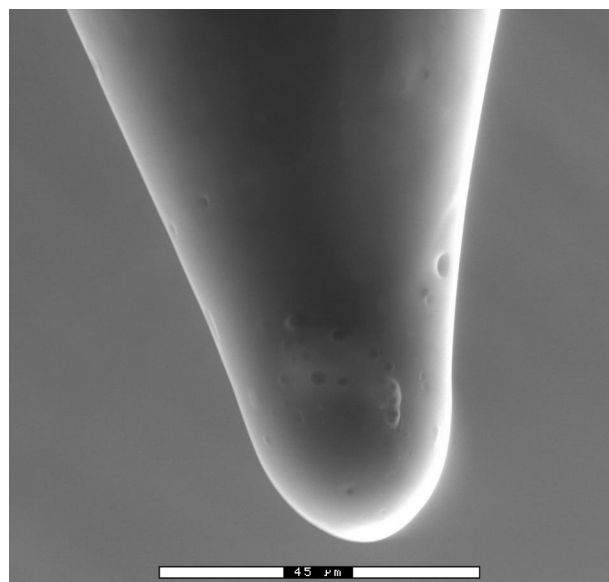ies for the epoxy. This motivates the use of filled epoxy with low thermal expansion and relatively high thermal conductivity.

(8) In order to expose the needle tip, the chromium coating, epoxy coating, and underlying nail polish must be removed from the tip to expose less than 30 μm of metal from the tip. The fibers of a wooden medical examination stick are used to remove the brittle nail polish from the needle tip. This procedure is carried out under a microscope with great care. Figure 8 shows a SEM photo of a completed needle tip.

(9) After a guarded needle is completed, the resistance between the needle shank and the SS shield was measured with a high resistance meter (Hewlett Packard 4329A) with a measuring voltage no greater than 100 V to check the quality of the needle electrode. A very good needle normally has a
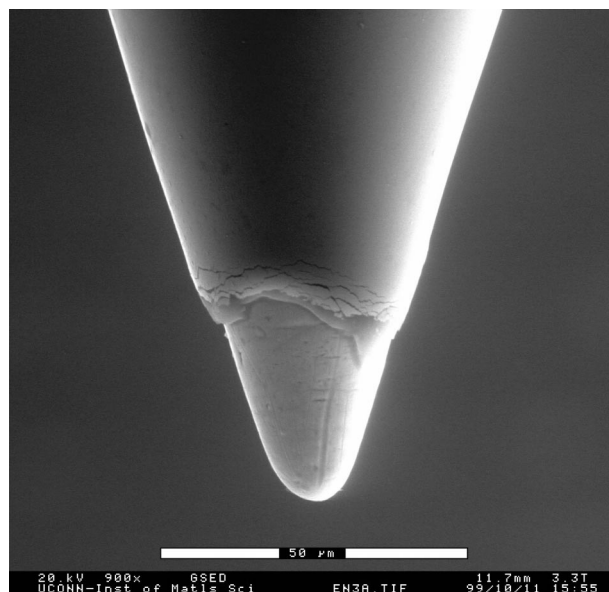

FIG. 8. The exposed needle tip after removing the nail polish. The needle is guarded with epoxy insulated, evaporated chromium shielding with an exposed region from the tip of $<$30 μm.
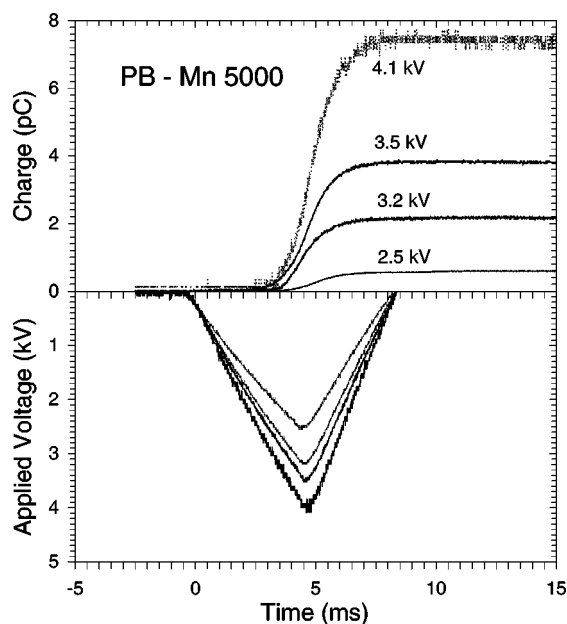
FIG. 9. Guarded needle measurements on polybutadiene (Mn-5000) with a needle-plate separation of 20 μm. Above a threshold voltage an increasing image charge on the needle tip is observed. The frequency of the triangular waveform is 50 Hz with data shown for peak voltages of 2.5 kV, 3.2 kV, 3.5 kV, and 4.1 kV. All four image charge signals start from the same threshold voltage, which is shown more clearly in Fig. 10.
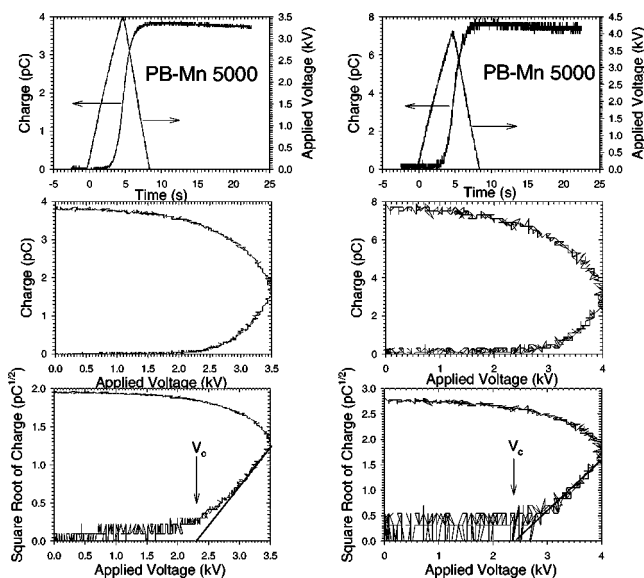

FIG. 10. The same experimental data as shown in Fig. 9. Left-hand side: peak voltage of 3.5 kV; right-hand side: peak voltage of 4.1 kV. Top plots show both the applied waveform and the image charge signal. The middle plots show the image charge vs applied voltage, while the bottom plots show the square root of the image charge vs applied voltage. The linear dependence of the square root of charge vs voltage (bottom plots) is in agreement with the FLSC model (see Refs. 2 and 10). The onset voltage for high carrier mobility, V_c, is determined by extrapolation to zero charge. As shown in bottom plots, the threshold voltage, V_c, is about 2.3 kV.

resistance greater than 10^{12} Ω while a needle with a resistance of less than 10^9 Ω is unlikely to perform satisfactorily.

B. Polymer insulated needle

The procedure for preparation of a polymer insulated needle is very similar to that for an epoxy insulated needle, except that the insulation layer is a special spontaneously electropolymerized polymer.[8,9] The polymer coated on the needle is a copolymer of N-phenyl maleimide (NPMI), bis-maleimide (BMI), styrene, and 2(methacryloyloxy)ethyl acetoacetate (MEA). The copolymer will initiate spontaneously on a steel surface, but unfortunately, not on chromium. Thus, a needle without chromium coating is used. The needle needs to be precleaned carefully to ensure copolymer growth. With the tip covered by nail polish, the steel needle is dipped into the N-methyl pyrrolidinone (57 ml) and water (43 ml) solution, with monomers of 1.73 g NPMI (0.1 M), 0.18 g BMI (0.005 M), 2.3 ml styrene (0.2 M), and 1.9 ml MEA (0.1 M). The reaction starts instantly, and a reaction time of 2 to 2.5 min will give a 1 μm thick polymer layer after densification. The needle with the polymer coating is rinsed for 15 min in a solution of 10% N-methyl pyrrolidinone and 90% ethanol to remove the chemical residues. The nail polish is removed with a wooden stick under a stereo microscope before drying, which is carried out in a vented oven at 100 °C for 30 min to evaporate the most of volatile solvent and then at 180 °C for 1 h with the needle tip pointing upward to densify the coating. This procedure forms a dense, pin-hole free polymer coating about 1 μm thick.

The nail polish needs to be reapplied before the needle is assembled into the SS tube for the subsequent metal shield coating. Compared with an epoxy insulated needle electrode,

the polymer insulated needle is less time consuming, as the thickness of the insulating layer is controlled by the reaction time. Polymer coated needles more often meet the resistance requirement and are generally more robust than epoxy coated needles when micropartial discharge occurs during measurements.

C. Summary

These procedures result in needle electrodes which are guarded to within about 30 μm of the needle tip. The capacitance between the needle tip and the high voltage plane electrode is reduced to the range of 2 fF. The residual capacitive current is canceled using the compensation circuit described herein. As a result, the resistive current can be measured without substantial interference from capacitive current with a background noise level in the 5 fC range. Interestingly, the capacitance of the exposed needle tip to the outside world is so small that the system can be operated without the use of a shielded enclosure. The needle electrodes are always stored in dessicators and handled with clean tools. These procedures have resulted in a high yield of good needle electrodes.

IV. CHARGE INJECTION IN POLYBUTADIENE

Measurements were carried out on commercial polybutadiene (Aldrich) with Mn (number average molecular weight) of 5000 using an epoxy insulated guarded needle electrode. To make a thin polybutadiene film, a 10% (weight/volume) solution is prepared by dissolving polybutadiene in toluene by stirring for half an hour in an ultrasonic bath. Polybutadiene film is obtained by solution casting onto a copper elec-

trode. After casting, the sample is kept in fume hood for 2 h and then in a vacuum oven for more than 12 h in order to evaporate the remaining solvent.

Figures 9 and 10 show the experimental measurements for image charge on the needle tip as a function of applied voltage for polybutadiene with Mn of 5000 with a needle-plane electrode separation of 20 μm. As can be seen in Fig. 9, above a threshold voltage V_c an increasing charge signal is observed. The image charge signals start from the same threshold voltage for all four applied voltages, as seen more clearly in Fig. 10. The middle plot of Fig. 10 shows the image charge versus applied voltage. The charge increases supralinearly above the threshold voltage. The square root of the injected charge increases linearly as a function of applied voltage (bottom plot), which is in agreement with the field limited space-charge (FLSC) model[2,10]

$$Q \approx R^2 \left(\frac{V}{V_c} - 1 \right)^2, \quad |V| \geq V_c,$$
$$Q = 0 \quad |V| < V_c, \tag{1}$$

where R is the tip radius and V_c is the threshold voltage. The approximate threshold field at the needle tip is given by[2]

$$E_c = \frac{2 V_c}{R \ln\left(\frac{4D}{R} \right)} \tag{2}$$

for large D/R, where D is the needle-plane separation. On this basis, the threshold field for image charge formation in polybutadiene with Mn of 5000 is 330 kV/mm. The threshold field for image charge formation in polybutadiene is very close to the same value for the positive and negative polarity, which is in accordance with the experimental results by ABB on polyethylene.[5] This implies that the charge can be withdrawn from the dielectric as easily as it can be injected and also the fraction of the traps involved is small. Even under impulse conditions, only 0.04% of the traps are filled in order to form such a space-charge limited field region with a charge density as high as 3500 C/m³.[4] After the maximum pulse voltage, the charge continues to increase as the voltage decreases, as seen in the middle and bottom plots of Fig. 10. This is caused by spreading of the space charge in the dielectric after the peak of the applied voltage has been reached, as discussed in greater detail in Ref. 7.

We would note that the approach taken to deriving Eq. (1) in Ref. 2 is somewhat convoluted as a result of the simplifying assumptions made by the respective authors.[2] As a result, a reader might be misled into thinking that the charge measured experimentally using the guarded needle apparatus is different from that described by the present authors. We therefore offer the following explanation which, we think, explains the difference in approach.

In Ref. 2, Hibma and Zeller base their analysis on the assumption that the space-charge limited field is fixed and does not increase with applied voltage above the critical voltage. This assumption corresponds to a material in which the conductivity goes from a finite value to essentially infinity at the space-charge limiting field. This assumption means that the field on the needle tip is independent of the applied voltage. Thus, above the threshold voltage, application of Gauss' law to the needle tip surface will result in a surface charge which is independent of applied voltage. In reality, the field at the needle tip does increase as a function of the applied voltage above the critical voltage, and the experimentally measured charge can be determined through application of Gauss' law to the needle tip, as the authors have done in previous publications.[6,7]

While the space-charge limiting field does not increase as a function of the applied voltage in the model of Hibma and Zeller,[2] the radius to which the space-charge limiting field extends from the needle tip does increase with applied voltage. Thus, Hibma and Zeller overcome this above problem by dividing the experimentally measured charge into two terms, (i) the surface charge on the needle tip as predicted by Gauss' law which, for their assumptions, is fixed above the threshold voltage, and (ii) the charge induced on the needle tip by the space charge in the dielectric. Of course, they subtract the charge on the needle tip caused by displacement current. Together, these terms add, approximately, to the experimentally determined charge, which can be predicted directly from Gauss' law applied to the needle tip if the electric field distribution is computed accurately using a technique such as transient nonlinear finite element analysis. The approach of Hibma and Zeller provides an approximate analytical basis for the observation that the measured charge increases as roughly the square of the applied voltage above the threshold voltage.

ACKNOWLEDGMENTS

[1] T. Hibma, P. Pfluger, and H. R. Zeller, 1984 Annual Report of the Conference on Electrical Insulation and Dielectric Phenomena, Claymont, DE, pp. 135–140.
[2] T. Hibma and H. R. Zeller, J. Appl. Phys. **59**, 1614 (1986).
[3] H. R. Zeller, IEEE Trans. EI **22**, 115 (1987).
[4] S. A. Boggs and J. Kuang, IEEE Electric. Insul. Mag. **14**, 5 (1998).
[5] T. Baumann, T. Hibma, J. B. Pethica, P. Pfluger, and H. R. Zeller, 1985 Annual Report of the Conference on Electrical Insulation and Dielectric Phenomena, Amherst, NY, pp. 266–273.
[6] G. G. Jiang, Ph.D. thesis, University of Connecticut, 2000.
[7] G. G. Jiang, J. Kuang, and S. A. Boggs, 2000 Annual Report of the Conference on Electrical Insulation and Dielectric Phenomena, Victoria, Canada, pp. 187–190.
[8] J. P. Bell, X. Zhang, and R. Agarwal, U.S. Patent No. 5,807,612, 15 September 1998.
[9] R. Agarwal, Ph.D. thesis, University of Connecticut, 1997.
[10] H. R. Zeller and W. R. Schneider, J. Appl. Phys. **52**, 455 (1984).