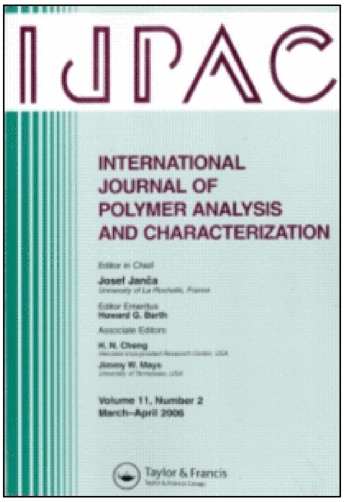

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Conductive Carbon Loaded Polymer Film Electrodes for Pulsed-Power Applications. Part I: Determination of the Film Properties

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Abstract: Electrically conductive polymer composites consisting of a nonconductive polymer matrix and conductive fillers, such as carbon black, are widely used. This contribution describes the specific electrical properties of polymer composite films for pulsed conditions in the microsecond (10^{-6} s) range. Investigation of an industrially available volume conductive polymer film (Carbostat) showed that the electrical properties of this material, which can be considered for electrodes in pulsed power applications, are quite different from the properties for DC conditions.

Keywords: Plastic electrodes; Polymer characterization; Polymer composite; Pulsed power

INTRODUCTION

Nonconductive thermoplastic polymer films are traditionally used as a packaging material for food as well as nonfood products. For some

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packaging applications a specific degree of electrical conductivity is required, e.g., for electrostatic discharge (ESD) protection. Since polymers are usually electrical insulators with a typical conductivity of $\sim 10^{-12}$ S/m,[1] one method to increase the electrical conductivity of a polymer is by the addition of different conductive fillers, thus creating a conductive polymer composite. Extensive research has been done on different polymer matrices using metal particles,[2] graphite and expanded graphite,[3] carbon black,[4,5] carbon nanotubes,[6] carbon fibers,[7] and metal-coated carbon fibres.[8] For most of the packaging applications, however, the preferred filler is carbon black (CB).

Due to the unique mechanical properties (i.e., easy to shape, elastic, and adaptable) and the electrical properties (i.e., the current limiting effect at temperature increase) of these conductive polymers, it is possible to explore their use as electrodes in newly developed pulsed power applications. These applications require conductivity throughout the volume, where the conductivity should be high to avoid excessive loss during conduction. By applying short pulses, the current density can be increased, while the temperature rise is equal to or lower than that for operation at DC conditions. The behavior of these polymers, especially in pulsed situations, is poorly described in the literature. Also, the specifications, which are mainly based on one certain application area (e.g., ESD protection, electromagnetic interference (EMI) shielding, or capacitor foils[9–12]), do not describe the properties for pulsed applications. In the international standard for resistivity measurements on composite conductive plastics, only a method for DC has been specified,[13] where it is stated that the contact resistance can be 1000 times larger than the sample resistance. So resistivity parameters specified by the manufacturers can be largely influenced by the contact resistance. To greatly reduce the effect of the contact resistance on the measurements, conductive glue has been used. A special measurement setup has been developed to perform the pulsed experiments.

The results on film properties and the development of the measuring setup are described in two separate articles, Part I and Part II respectively. Part I (this article) gives a detailed characterization of the electrical and mechanical properties of an industrially available conductive film (Carbostat), which have been determined with the newly developed measuring setup. In Part II the developed measuring setup is described, and a systematic analysis of contact resistance and three contact resistance minimization methods, namely pressing, wetting, and gluing, is given. The experiments shown have been carried out in the microsecond (10^{-6} s) pulse range and are compared with DC measurements.

Conductive Polymers

Figure 1 shows the typical form of the conductivity dependence of a conductive composite material on the filler content. Upon an increase of the filler concentration, Φ, a critical value, Φ_c, is reached above which the conductivity of the composite, σ_c, rapidly increases. Within the percolation theory framework this critical concentration is referred to as the percolation threshold.[14] Below this concentration the filler particles are separately dispersed or form isolated clusters of finite size distributed within the polymer matrix. At Φ_c, however, the clusters become interconnected and produce an infinite continuous path of conductive particles within the insulating matrix, which allows a current to flow through the composite material. Adding more filler results in an increasing number of conductive paths, and a fully developed conductive network is formed. Figure 1(b) shows a schematic representation of such a conductive network. According to the percolation theory the conductivity of the composite, σ_c, above the percolation threshold can be well described by power law dependence[14]:

$$\sigma_c = \sigma_f(\Phi - \Phi_c)^{\upsilon} \tag{1}$$

where σ_f is the conductivity of the filler and υ is a critical exponent.

In Equation (1) the concentration of the filler, Φ, and the critical concentration, Φ_c, are expressed as volume ratios. The universal value of υ for a three-dimensional lattice is between 1.65 and 2.00.[6]

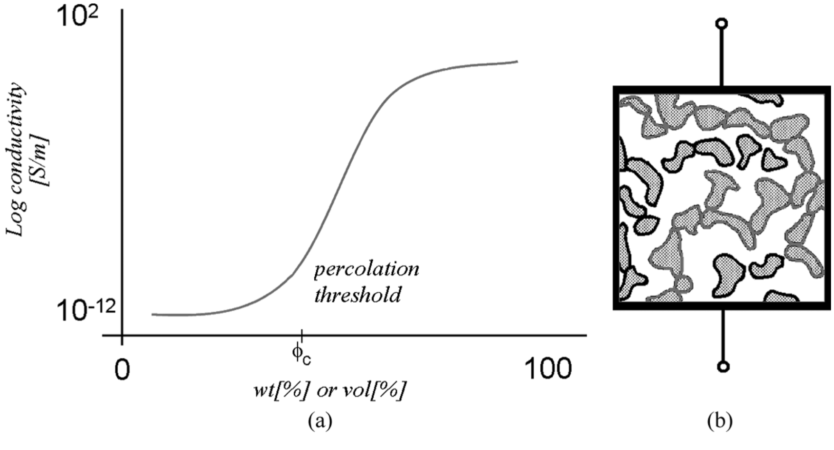

Figure 1. (a) Typical conductivity dependence of a conductive composite material on filler content, (b) schematic representation of the composite after percolation.[14]

Experimentally, however, higher values of υ are reported,[15] and it seems to be influenced by the nature of the filler and the matrix, as well as by the preparation conditions.[16] For instance, by the production of films, during the manufacturing process some orientation of the filler particles can take place because of specific flow conditions. This can give rise to a higher conductivity in the machine direction than in the perpendicular one and, correspondingly, leads to different critical concentrations and exponents in these two directions. Equation (1) suggests that increasing the filler content increases the conductivity of the material; however, some of the other properties (e.g., ductility, scratch resistance, process ability) deteriorate. For applications that require a high conductivity, the concentration of the conductive filler is often chosen slightly above the percolation threshold as a compromise. In that way, continuous pathways are always present within the composite, but a large number of closely spaced conductive particles are also present that are isolated only by thin layers of the matrix material. For the chains that form the pathway, a standard ohmic behavior is typically observed, whereas for the closely spaced conductive particles a second conduction mechanism is possible. At the sharp particle protrusions, where high local electric fields are obtained, induced quantum-mechanical tunneling of electrons can occur. This phenomenon can lead to a nonlinear volt-ampere characteristic of the composites.[2]

Considered Material

The Carbostat film used in this study is based on ethylene vinyl acetate copolymer (EVA). Using dynamic mechanical analysis (DMA) the values of the storage and loss moduli of the composite film at room temperature are found to be $E' = 870\,\text{MPa}$ and $E'' = 98\,\text{MPa}$, respectively.[17] The storage modulus is a measure of the ability to store elastic energy and the loss modulus is a measure of the energy lost in the polymer film. DMA gives also the value of $\tan(\delta)$, where δ is the phase difference between the deformation imposed on the sample and the resulting force. The peaks in $\tan(\delta)$, shown in Figure 2, specify the transitions taking place in the polymeric matrix. Based on the transition temperatures, the relative weight of the vinyl acetate can be estimated at about 10 wt%.[18] The weight content of the conductive filler, carbon black (CB), was estimated at approximately 30 wt% by thermogravimetric analysis (TGA). Figure 3, which is a scanning electron microscope image, shows that the conductive filler consists of aggregated molecules ranging in size from 50 to 200 nm.

Thermoplastic molecules gain some orientation during processing, which makes the macroscopic properties of stretched polymers such as

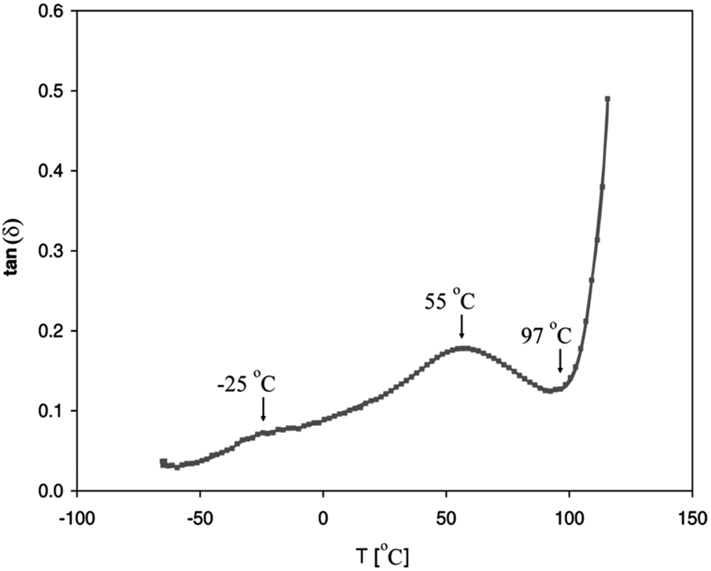

Figure 2. Dynamic mechanical analysis (DMA) data of Carbostat. Tan(δ) = f(T) at 1 Hz. Visible transitions: glass transition at (–25°C; first melting process around 55°C and a second melting process with an onset at 97°C.

strength, optical, and electrical properties, depend on direction. Generally, the conductivity along the chains (parallel to the film) is higher than the conductivity in normal (perpendicular) direction. So the

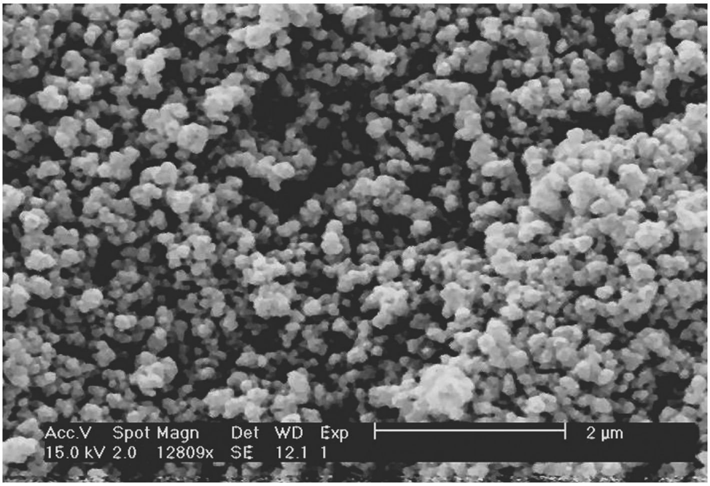

Figure 3. Scanning electron microscope (SEM) image of the conductive filler.

conductivity for films should be defined in two and sometimes three directions, which are mainly called:

- Machine direction (σ_{MD}), parallel to the film surface in the machined direction
- Transversal direction (σ_{TD}), parallel to the film surface
- Perpendicular direction (σ_{PD}), normal to the film surface

Carbostat technical information[19] shows only the typical (parallel) surface resistance, R_S, 10^4–$10^5\,\Omega$, while the material is volume conductive. This surface resistance is determined by the manufacturer with the well-known concentric ring electrode assembly conforming with the European EN 61340-2-3 standard.[20] The inner ring electrode has a diameter of 30.5 mm, and the gap distance between the inner and outer ring electrode equals 13.3 mm. It is assumed that the current penetrates the film thickness completely (i.e., homogeneous distribution) and there is no contact resistance between film and probe; the volume conductivity of the film in parallel direction should be between 0.012 and 0.12 S/m. This is valid only when the film thickness is much smaller than the gap distance of the probe, here 80 μm and 13.3 mm respectively. The relationship between the specified surface resistance R_S and the conductivity in [S/m] in parallel direction approximates, in that case:

$$\sigma_{MD,TD} = (dR_s k)^{-1} \rightarrow k = \frac{\pi(d_e + g)}{g} \tag{2}$$

where d is the film thickness, g is the gap distance of the probe and d_e is the diameter of the inner electrode, all in [m]. The factor k is often called the geometry factor of the probe and equals ~10. For the electrical characterization described in this contribution two production batches of Carbostat have been used, no. 08/2005 and no. 06/2006. For the new proposed applications the parallel and perpendicular volume conductivity, which is not specified by the manufacturer, needs to be determined. The parallel conductivity determined in this contribution will be compared with the estimations from Equation (2).

CONTRIBUTORS TO RESISTANCE

A film sample, which is connected to an electrode, is a series connection of two resistances, namely the contact resistance and film resistance. These resistances can be strongly influenced by temperature, pressure, and applied electric field. The following contributors that will influence the measured resistance have been investigated:

- Time- and pressure-dependent film conductivity, Part I
- Electric field–dependent film conductivity, Part I
- Contact resistance, Part II

The measurements, which are described in Part I, were carried out to determine the film characteristics. During these measurements the contact resistance needs to be eliminated, which is done by using conductive glue. The followed procedure for the determination of the film characteristics is depicted in Figure 4.

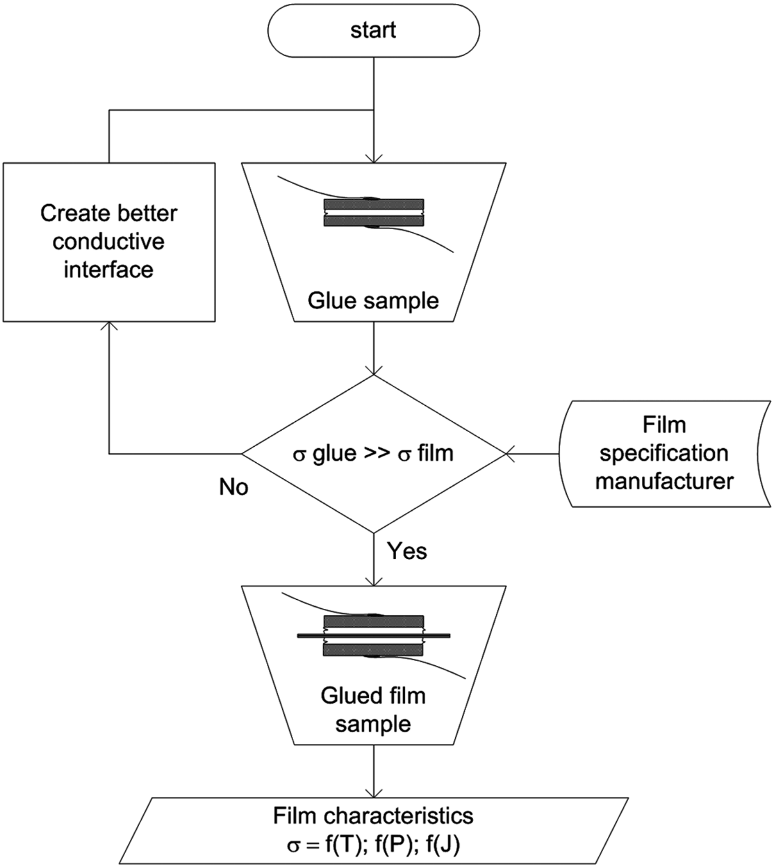

Figure 4. Process flowchart for the determination of the film characteristics.

In general the conductivity in [S/m] of the glue or the film sample can be calculated with the measured resistance in [Ω] from

$$\sigma = \frac{l}{AR_m} \tag{3}$$

where l is the length of the sample in [m], and A is the conductive cross section in [m^2].

Applications that have to deal with interface problems between different materials (contact resistance) are widespread, such as in circuit breaker contacts or on potentiometer tracks.[21,22] There can be several reasons to reduce the contact resistance: a good thermal conductivity[23] or high electrical conductivity. Part II of this contribution focuses on the latter, with the following main goal: a homogeneous current distribution across the polymer-metal interface to avoid local i^2R losses and thereby early breakdown of the polymer electrode.

Time- and Pressure-Dependent Film Conductivity

Applying an increasing compressive force on the film decreases the measured resistance, which can have two causes. First, an increased force on surfaces with a certain roughness creates a larger contact area and results in lower contact resistance. This is described in Part II. A second possible explanation is that with increasing force a higher compression of the film is achieved, and that brings the dispersed conductive particles closer together. The number of the available conductive pathways increases and thereby the resistance decreases. For both cases the deformation does not occur instantaneously; the decrease of thickness, and thereby the film resistance, is also time dependent. One can apply for this behavior the linear viscoelastic Maxwell model with one relaxation mode:

$$P = \eta \frac{d\varepsilon}{dt} + E'\varepsilon \tag{4}$$

where $P = F/A$ is the stress applied on the film in [N/m^2], A is the film surface area [m^2], $\eta = E'/\omega_m$ is the viscosity of the film (ω_m scales the rate at which the process happens), and ε is the compressive strain. Solving Equation (4) and taking into account that $\varepsilon(t) = \Delta d(t)/d_0$ ($\Delta d(t)$ is the variation of the initial thickness d_0 due to the compression), the following expression can be written:

$$d(t) = d_0\left[1 - \frac{P}{E'}\left(1 - e^{-t/\tau}\right)\right] \tag{5}$$

where $\tau = \eta/E'$ is the time constant of the compressive deformation process. With this time-dependent thickness, the time-dependent film resistance for compression can be determined, but due to its complexity it falls beyond the scope of this article. Modeling and experimental results can be found in various publications.[24,25] The variations in the contact area due to the force applied seems to be the more important cause for the time-dependent behavior of the resistance, however up to now the two mechanisms were not quantified independently. In Part II of this contribution, the time constant of the deformation process is determined experimentally from the resistance measurements ($\tau \approx 20\,\mathrm{s.}$), and together with two contact resistance elimination methods a split up has been made.

Electric Field–Dependent Film Conductivity

Especially for applications in power equipment, the current density and the achieved field strength in the material will be high. The composite polymer film, filled with small CB grains, is loaded locally with even higher electric fields at sharp particle protrusions of incomplete pathways. Due to local high electric fields, the electrons can jump from one particle to another, which is known as tunneling or hopping conduction.[2] At low electric field strength, only the full conductive pathways contribute to the total resistance, and at higher field strengths extra parallel pathways are formed that reduce the resistivity. So the materials resistivity (and hence the conductivity) depends on the electric field, which can lead to nonlinear volt-ampere characteristics and finally to breakdown.[25] Breakdown due to local high electric fields caused by sharp inclusions is generally known as treeing.[27] A so-called thermal breakdown happens when local losses (e.g., caused by nonhomogeneous current distribution) heat up the material above the melting temperature. Determination of the types of breakdown for the described experiments falls beyond the scope of this contribution.

MEASUREMENT SETUP

Characterization of the polymer film is possible only when the copper electrodes, which are used for the measurements, can be connected to the sample with great reproducibility and with a contact resistance that is as low as possible. For this reason all samples were connected to the electrodes with conductive glue. The electrical properties of this glue have been determined first and have been compared with the properties of the film; see Figure 4. After elimination of the contact resistance, the film has been glued to the electrodes and the influence of the film conductivity on the pressure, temperature, and electric field have been investigated.

Glue Samples

Two types of glue have been investigated by preparation of the glue samples, Bison Electro[28] and Circuit Works 2400 from Chemtronics,[29] which are a single-component and two-component product respectively. In all samples, the glue has a small area in direct contact with the surrounding air. This results for the single-component product, where hardening takes place by evaporation of the solvent in long and undefined curing times (i.e., >48 h). The best results were obtained with the two-component epoxy product, and that one was used to prepare all the samples described in this contribution. This glue has a specified volume conductivity of at least 10 S/m. This is larger than the volume conductivity specified by the film manufacturer, which is between 0.012 and 0.12 S/m. Before gluing a sample, both electrical connections were soldered to the electrodes. The electrodes were cleaned with Scotch-Brite Grade 400 and degreased with alcohol of 70% in advance. All samples used for the determination of the conductivity of the glue are typically 20 × 20 mm.

Glued Film Samples

Figure 5 shows a typical 20 × 20 mm glued film sample for the determination of the conductivity in normal direction. These samples are

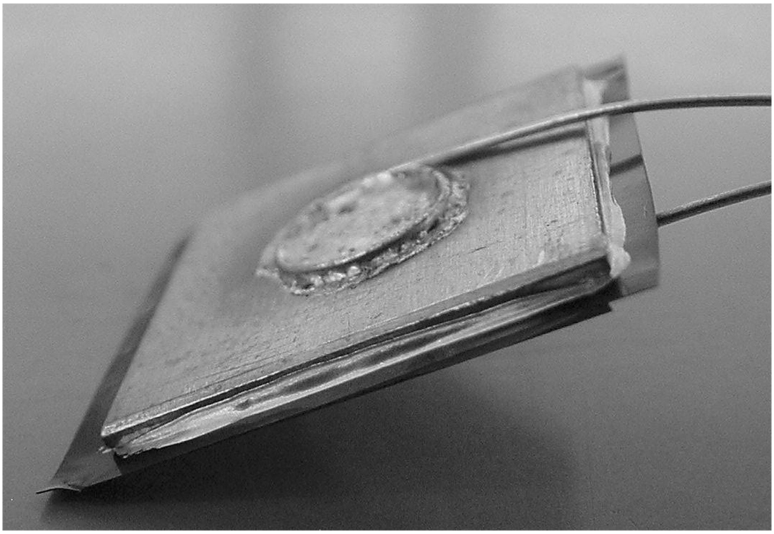

Figure 5. Glue sample used for the determination of the normal conductivity (20 × 20 mm). Material buildup: copper electrode, glue, film, glue, and copper electrode.

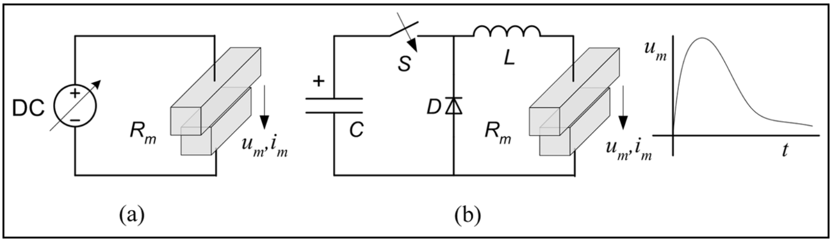

Figure 6. (a) DC measurement setup, (b) RLC circuit used for pulsed measurements with typical waveform.

prepared in the same manner as the glue samples. For the conductivity measurement in parallel direction, thin copper wires were directly glued to the film surface.

Electronic Setup

Figure 6 shows the DC and pulsed measurement circuits. Both samples have been first fed with a low DC voltage to determine the conductivity and their temperature dependence. The separate voltage and current readings (u_m and i_m) are used to determine the measured resistance, R_m, and by using relationship (3) the specific conductivity of the sample was calculated.

For the pulsed measurements, which are needed to increase the current density (and thereby the electric field), an electronic circuit has been developed. This damped oscillatory *RLC* circuit generates pulses of ~3 μs with a limited amount of energy, which is initially stored in the capacitor, *C*. Via the switch *S* the energy is transferred to the resistive load, *R* (i.e., the film). The discharge becomes oscillatory due to the stray inductance (*L*) of the attached cabling. A detailed description of the electronic setup is given in Part II of this article.

EXPERIMENTAL RESULTS

To define the glue conductivity more accurately, three samples were made where two copper electrodes were directly glued together (*glue samples*). After 24 h of curing at a temperature of ~25°C, the maximum conductivity was reached and was calculated by using Equation (3). The calculated conductivities are between 57 and 220 S/m, which is much higher than the minimum specified value given by the manufacturer, >10 S/m.[29] Also, the thickness of the glue layer differs from that specified; it is typically one to three times less than the film thickness, which is 80 μm. So the measured resistances in case of the glued film samples approximate the film resistance and can be used together with the geometry to determine the film conductivity with an acceptable error.

Influence of Temperature

Because the first melting process, depicted in Figure 2, starts at 55°C, the maximum safe operation temperature for this conductive EVA polymer is 50°C. From room temperature, approximately 20°C, up to 50°C the parallel conductivity, the normal conductivity, and the ratio between both have been determined and are depicted in Figure 7. To investigate the dispersion of the data the experiments were done on different samples and were repeated at least three times. Each dot represents a single measurement. All data shown were measured with the DC measurement setup and the current for all measurements was 1 mA, which equals a current density of approximately $2.5 \times 10^{-6}\,\mathrm{A/mm^2}$.

The film conductivity is assumed to be linear function with respect to temperature within the 20°–50°C range, and all data points are averaged and fitted in the following general equation:

$$\sigma_c(T) = \sigma_{T_0}(1 + \alpha_T \Delta T) \tag{6}$$

where σ_{T_0} is the conductivity at zero Celsius, α_T is temperature coefficient in [°C^{-1}], and ΔT is temperature difference with regard to T_0. For higher temperatures, the polymer matrix expands and thereby the filler concentration decreases. If two expansion coefficients have been considered (i.e., α for parallel film direction and β for normal film direction in [°C^{-1}]) and Equation (1) is substituted into Equation (6), the temperature-dependent composite conductivity can be approximated by

$$\sigma_c(T) \approx \sigma_f(\Phi_{T_0} - \Phi_c)^{\upsilon}(1 + \alpha_T(T - T_0)) \quad \text{with } \alpha_T \approx \frac{-\Phi_{T_0}}{\Phi_{T_0} - \Phi_c}\upsilon(2\alpha + \beta) \tag{7}$$

This approximation has been obtained by using Taylor series expansion. The conductivity, σ_{T_0}, and the temperature coefficient, α_T, for the parallel and normal measurements are 9.05 S/m, 0.83 S/m, −0.0071°C^{-1}, and −0.0155°C^{-1} respectively. Both fits have a so-called *R-square* value of 0.996 and 0.970 respectively. The typical measured temperature-dependent conductivities in normal (i.e., perpendicular) direction and parallel direction are in the range of 0.1–0.9 S/m and 5.0–7.9 S/m respectively. Variation has been caused by temperature and hysteresis between cooling and heating as well as by the use of different production batches of the film, namely 08/2005 and 06/2006.

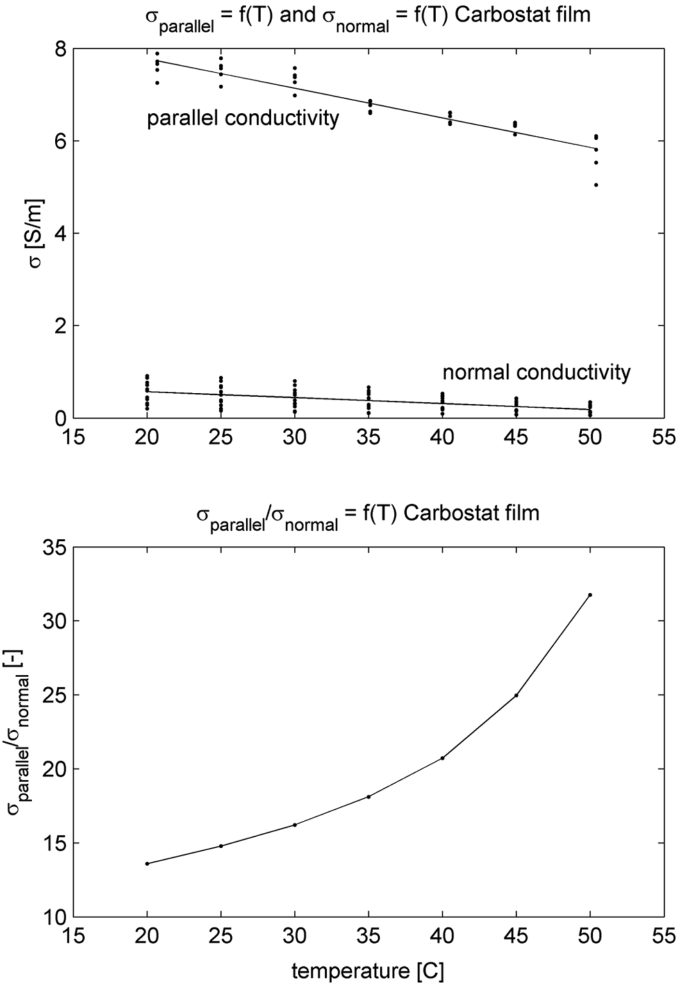

Figure 7. (a) Fitted parallel and normal conductivity as function of temperature. The dots represent the individual measurements. (b) Ratio between the fitted parallel and normal conductivity.

Influence of the Pressure in Normal Direction

For pulsed power electrode purposes the film will be generally used as electrode covering. The current will flow in normal (i.e., perpendicular) direction, and therefore the conductivity dependence on the pressure in normal direction has been examined. The glued film samples were clamped from 3 to 22 kPa and the normal conductivity after the relaxation time (i.e., $5\tau \approx 100\,\mathrm{s}$.) was examined. The average value of the measured minimum values and maximum values for 3 and 22 kPa are 0.45 and 0.47 S/m respectively. One can conclude that the film conductivity itself in this pressure range is constant. This also matches with Equation (5); a decrease in thickness directly relates to an increase in film conductivity, and there is no reduction in film thickness due to the applied pressure of 22 kPa.

Influence of the Electric Field

As described above, a composite polymer film loaded with small grains has a conductivity that depends on the applied electric field. The electric field and therefore the current density are increased by applying more voltage across the sample. To avoid too much heat generation and thereby early breakdown of the film sample, the voltage has been applied with the pulsed measurement setup of Figure 6(b). Figure 8 shows the film conductivity and electric field (at room temperature) as a function of the current density for a glued film sample. Calculation of R_m is done at the maximum achieved voltage and current. For low current densities (i.e., $J < 10^{-2}\,\mathrm{A/mm^2}$), the average film conductivity is constant, approximately 0.45 S/m, as described above. At higher current densities, the film conductivity and electric field strength increase.

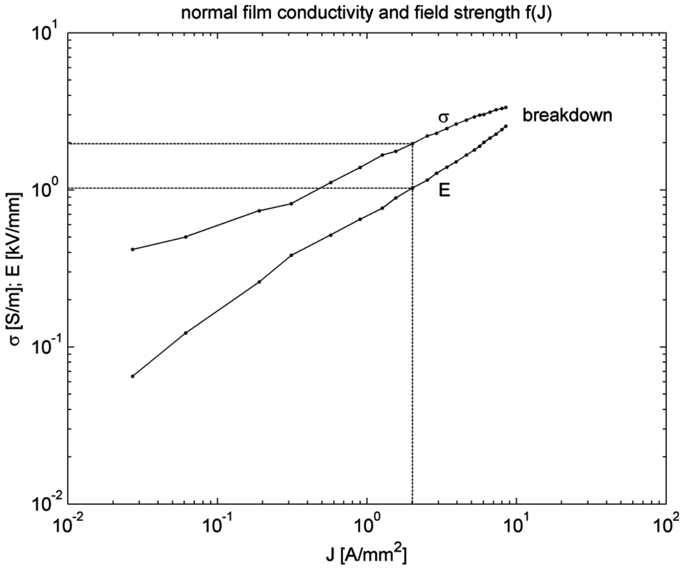

Figure 8. Film conductivity and electric field strength as function of the current density.

At 2 A/mm^2 the normal film conductivity is already four times larger. During these pulsed experiments it was possible to increase the current density up to 8.5 A/mm^2 before breakdown occurred. The corresponding achieved conductivity and field strength in normal direction is 3.3 S/m and 2.5 kV/mm respectively.

To avoid misinterpretation of these data, the glue needs to be examined too. Thus, for a glue sample, the sample without polymer film, the glue conductivity as a function of the current density was determined. It is imaginable that the glue exhibits the same behavior as the film, namely, it is also a nonconductive material filled with granular silver particles. The conductivity of the glue is determined with the DC and pulsed setup and is more or less constant in the range from 10^{-4} to 10^{0} A/mm^2 and equals at least 57 S/m.

DISCUSSION

The investigated film, Carbostat, which is anisotropic and conductive throughout its volume, is specified only by its surface resistance, conforming to European standard EN61340-2-3. The field of use, ESD safe packaging of electronic components, is the main reason for this. Initially, by using relationship (2) the parallel volume conductivity of the film was estimated, but these values are much lower than the values depicted in Figure 7, 0.012–0.12 S/m and 5.0–7.9 S/m respectively. Several reasons can cause this difference, like a nonuniform current distribution or a high contact resistance between film and the standard concentric ring probe assembly. In addition, the manufacturers of volume conductive materials need to expand the specification with volume conductive measurement data, which is also described in the European standard EN 61340-2-3.

Does the determined breakdown field strength of film match with the breakdown field strength of the polymer matrix material (EVA) and the so-called hopping distance? Due to local high electric fields at sharp particle protrusions in the incomplete pathways of the film, the electrons can jump from one particle to one other, which can result in resistance decrease and finally to a breakdown. The voltage drop in these incomplete pathways occurs between the separated conductive particles. By assuming that the shortest conductive path is a one-dimensional string of conductive particles (in perpendicular film direction) and knowing that the average particle size is approximately 125 nm (i.e., 50 to 200 nm), the average number of the particles within the path (neglecting the distance between them) is 640 (i.e., 80 μm ÷ 125 nm). Since the breakdown voltage of EVA reported in Murakami et al.[26] is 200 kV/mm, one can estimate the total thickness of the insulating matrix regions (between the particles) over which the breakdown voltage is measured. In this situation, it will be realized as 1.0 μm (i.e., 2.5 kV/mm × 80 μm÷200 kV/mm). Assuming that the total thickness of the polymer matrix is shared equally among all filler particles, an average hopping distance of 1.6 nm can be estimated (i.e., 1.0 μm ÷ 640). This value is close to the value determined in Murakami et al.,[26] which is 2.1 nm.

In pulsed operation, the moment of breakdown (shown in Figure 8) was reached quickly, within a few μs, so the heating of the sample can be assumed as adiabatical. The dissipated energy in the film per unit volume in the case of a homogeneous current distribution just before breakdown equals $\sigma_f \cdot E^2 \cdot t_p = 3.3\,\mathrm{S/m} \times 2.5^2\,\mathrm{kV/mm} \times 3\,\mu\mathrm{s} = 62\,\mathrm{mJ/mm^3}$, where σ_f, E, and t_p are film conductivity, electric field, and pulse duration, respectively. The maximum operation temperature of the film equals the ambient temperature, T_a, added to $((\sigma_f \cdot E^2 \cdot t_p)/(\rho_f \cdot c_f))$, which equals 51°C, where ρ_f is the specific mass, $1000\,\mathrm{kg/m^3}$,[19] and c_f the average specific heat capacity, ~2045 J/kg·K,[30] of the film. This value is close to the first melting temperature described in Arsac et al.[18] and determined in this contribution (Figure 2), which is 53°C and 55°C, respectively. So, with the determined dissipated energy per unit volume and the melting temperature from the DMA analysis, one is able to estimate the maximum pulse duration, t_p, for different load conditions. This can be obtained as the worst case maximum material loading, when no heat can be removed.

CONCLUSIONS

A new characterization method has been proposed that enables conductive foils to be characterized with respect to pulsed power applications. Carbostat, a carbon-loaded film with an EVA polymer matrix, has been characterized for pulsed operation. To determine the electrical properties of the film, it has been glued to the electrodes to minimize the contact resistance. The typical temperature-dependent DC conductivities (from 0° to 50°C) in normal and longitudinal direction in relation to the film surface, are in the range of 0.1–0.9 S/m and 5.0–7.9 S/m respectively.

For film current densities larger than $10^{-2}\,\mathrm{A/mm^2}$ and thereby higher field strength, which can be achieved with the newly developed pulsed setup, the conductivity of the film in normal direction has been investigated and it has been found that it increases rapidly. At $8.5\,\mathrm{A/mm^2}$ just before breakdown, the conductivity is 3.3 S/m. The corresponding breakdown field strength is ~2.5 kV/mm. With this breakdown information a method to determine the maximum pulse duration at different load conditions has been proposed by using the dissipated energy per unit volume.

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