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Thermally Stimulated Depolarisation Current and Pyroelectric Measurements Using the NOVOCONTROL Cryosystem QUATRO

The thermally stimulated depolarisation current (TSDC) technique [1] proves to be a powerful tool especially for the investigation of charge carrier relaxations in dielectrics. It is widely used as a complement to dielectric spectroscopy. Usually, the TSDC equipment consists of a cryostat with a sample holder, a dc power supply and a sensitive electrometer. The basic experiment includes two main steps: first the sample material is polarised in a dc field E_P for a time t_P at a high temperature T_P at which particular dipolar units or charge carriers are free to move. In the second step, the sample is shorted at a low temperature where the relaxation times of the interesting species are much longer than the measuring time, and finally it is linearly heated, while the depolarisation current is recorded. Generally, the TSDC consists of relaxation, pyroelectric and conduction contributions which may be separated by special techniques.

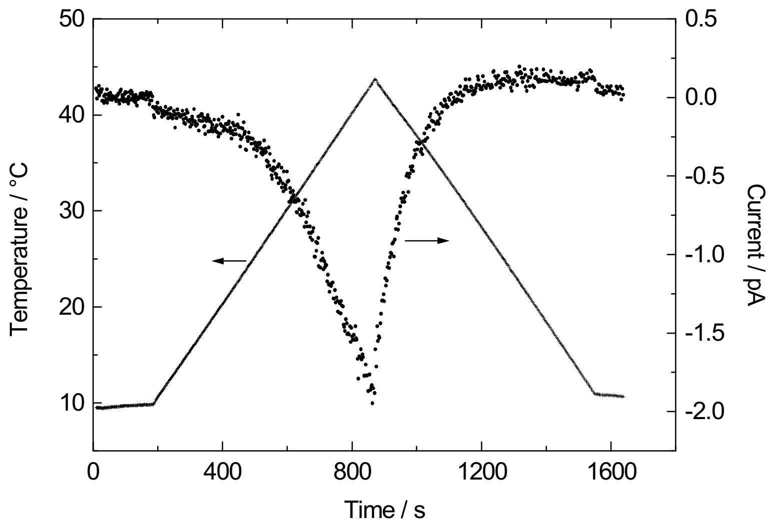

Fig. 3 : Static pyroelectric measurement (heating rate ±3.0 K min^{-1})

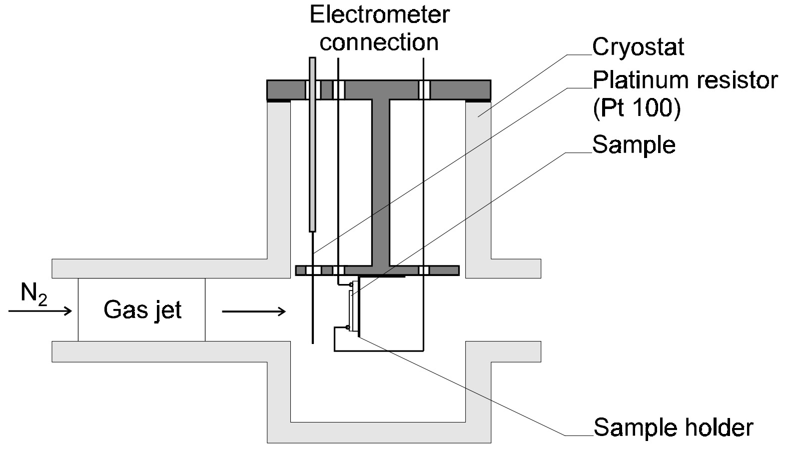

Fig. 1 : Measuring chamber for dc measurements with the cryosystem QUATRO

The TSDC scan corresponds to a temperature dependent loss measurement at an equivalent frequency in the ultra low frequency range (10^{-5} to 10^{-2} Hz). Because TSDC works under non isothermal conditions, the relaxation function is not directly accessible; additional knowledge about the temperature dependence of the relaxation time is necessary. On the other hand, a single TSDC scan yields an overview of all thermally induced processes with high resolution. Moreover, it allows the investigation of a non-linear dielectric response by simply varying the electric field strength over a wide range. This will be demonstrated in the following example.

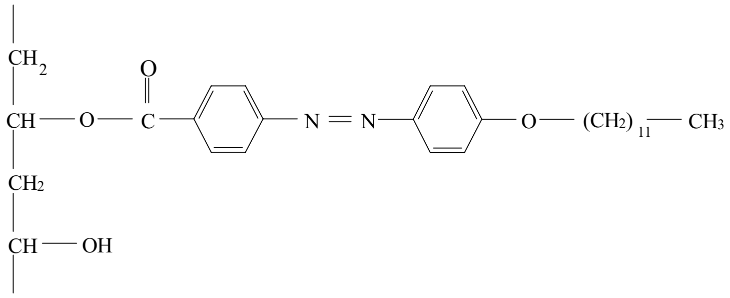

Fig. 2 : Chemical structure of the poly(vinyl alcohol) with azobenzene alkoxy side chains

A NOVOCONTROL Cryosystem QUATRO was used for TSDC measurements on polymer- electret materials. To this end, a special sample holder was built which permits dc measurements down to 10^{-14} A (Fig. 1). The sample is connected to a Keithley 617 electrometer with a build-in dc voltage source. The equipment is fully computer controlled on the basis of Keithley TestPoint software. The sample holder is designed for polymer films or films on a substrate, however, it can easily be modified for samples in any geometry. The gas heating system and the small heat capacity of the sample holder allow relatively fast and well defined temperature variations and reduce temperature gradients in thicker samples, which would otherwise lead to serious errors e.g. in determining relaxation parameters.

Pyroelectricity is the reversible change of the spontaneous or induced polarization P with temperature. If a pyroelectric material, sandwiched between metal electrodes, is shorted through a sensitive electrometer and heated, the pyroelectric current I_p is given by

$$I_p = A\,p(T)\frac{dT}{dt} \quad (1)$$

where A is the electrode area, and $p = dP/dT$ is the pyroelectric coefficient. In the following example, two techniques for determining the pyroelectric coefficient will be presented.

The sample material is a poly(vinyl alcohol) with azobenzene alkoxy side chains (Fig. 2) [2, 3]. The substance contains strong molecular dipoles formed by the donor-acceptor interaction via the delocalised π-electron system of the azobenzene unit. Therefore, it should exhibit pyroelectric activity caused by libration of these dipoles. Polymer films with a thickness of about 2 µm were prepared by casting a 2% chloroform solution on aluminised-glass substrates and evaporating aluminium top electrodes (area 8 mm [2]). A polar order is introduced by thermal poling which is identical to the first step of the TSDC procedure. The poling parameters are $E_p = 25\ \mathrm{MVm^{-1}}$, $T_p = 180\ °C$ and $t_p = 30$ min.

The static pyroelectric measurement [4] resembles the second step of a TSDC experiment. The temperature is increased and decreased in a limited temperature interval at a constant rate as shown in Fig. 3. According to Eq. 1, the pyroelectric current changes its sign when the direction of the temperature scan is changed. In this case, p may be directly determined. Often, however, the current is superimposed by non-pyrolectric currents due to relaxation or conduction, and the various contributions cannot be separated.

In the quasistatic regime [5] (Fig. 4), a slow sinusoidal temperature variation is applied to the sample. The pyroelectric current follows from Eq. 1 as

$$I_p = A\,p(T)\,\omega T_0 \cos(\omega t), \quad (2)$$

where T_0 is the amplitude and ω the angular frequency of the temperature oscillation.

The non-pyroelectric current can be separated using the phase shift between the total current and the temperature, which is determined from sinusoidal fits, as indicated. The pyroelectric coefficient is calculated from the amplitude of the pyroelectric current. In the present example, with $\omega = 0.042\ \mathrm{s^{-1}}$ and $T_0 = 1.0$ K, a pyroelectric coefficient of $p = 0.24\ \mathrm{\mu C\ m^{-2}\ K^{-1}}$ is found at T = 25 °C.

The maximum possible angular frequency and the maximum

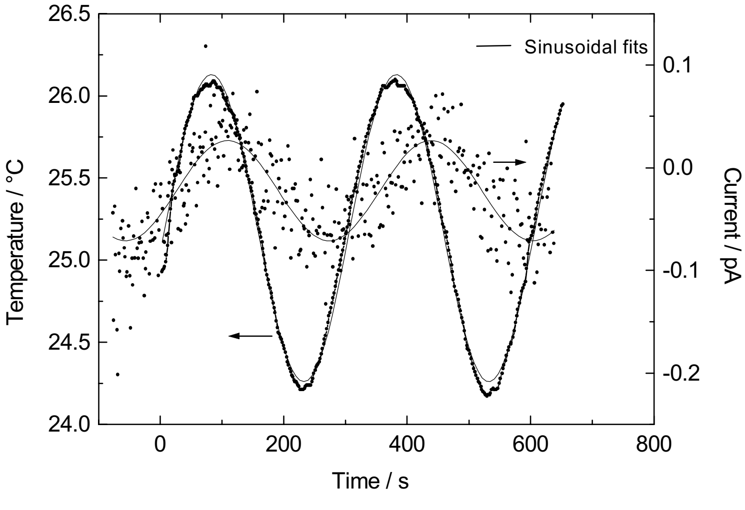

Fig. 4 : Quasistatic pyroelectric measurement (amplitude 1.0 K, angular frequency $0.042\ \mathrm{s^{-1}}$)

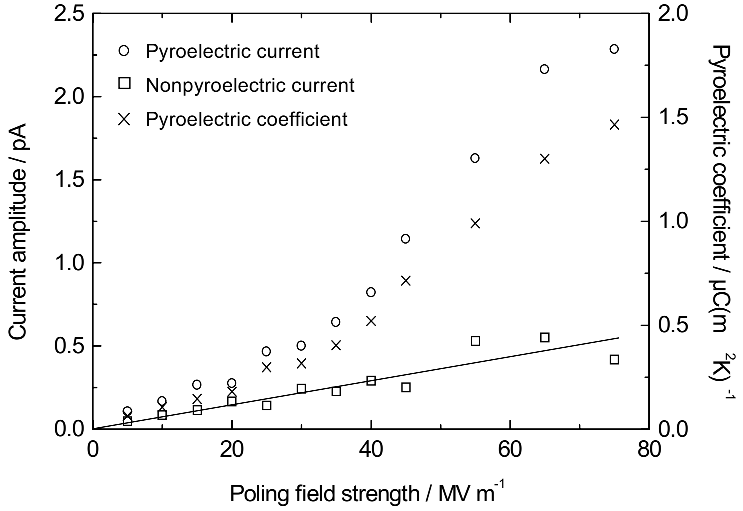

Fig. 5 : Poling field dependence of the pyroelectric and non-pyroelectric current components separated by quasistatic measurements as well as the pyroelectric coefficient of the sample material

possible heating rate are determined by the thermal inertia of the sample holder; here, they are about 0.15 s^{-1} (at T_0 = 1 K) and 8 K min^{-1}, respectively. The noise of the current signal probably arises from microscopic mechanical movements of the sample during the relatively fast temperature variation.

Measurements at various electric field strengths (Fig. 5) show that the small non-pyroelectric current is indeed proportional to the electric field, whereas the pyroelectric current is clearly non-linear. This will be discussed in a separate paper. Nevertheless, the result demonstrates the high current resolution of the dc equipment used together with the Cryosystem QUATRO.

References and notes

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Setup for High-Pressure-Dielectric-Spectroscopy

Introduction

Frequency and temperature are the prime variables for dielectric spectroscopy up to date. Beside these, pressure is a very important parameter for the investigation of molecular properties as conductivity

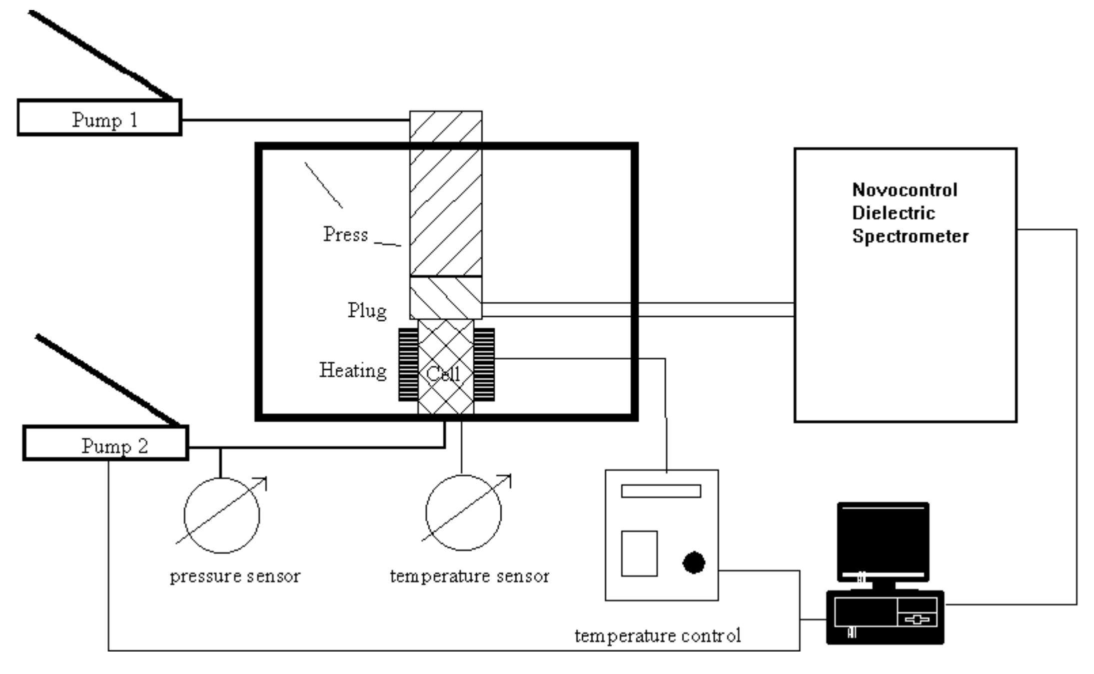

Fig. 1 : Scheme of the setup

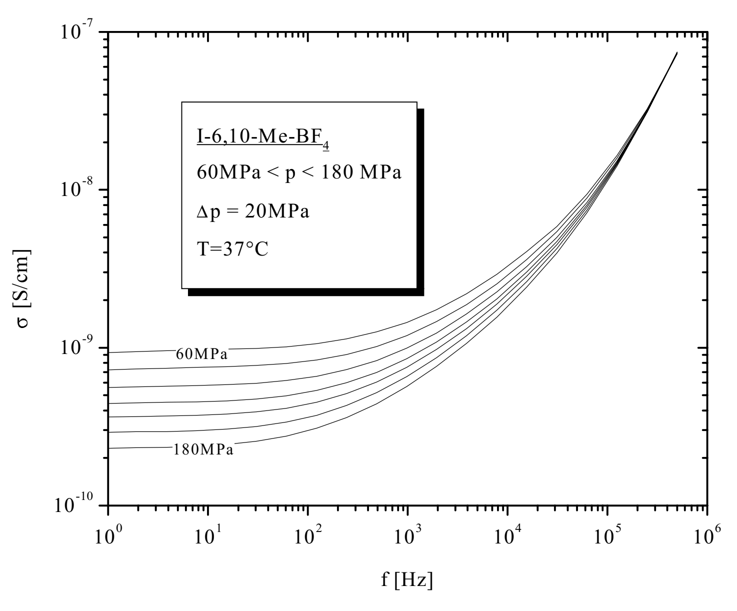

Fig. 2 : Measurement of with I-6,10-Me-BF_4

[1], dielectric properties [2] and phase behaviour [3]. Only a few apparatus to perform dielectric measurements under pressure are already in use [4], but as there is no commercially available system up to date, the existing experimental setups are not optimized for easy handling and sometimes are rather limited with respect to temperature and pressure variations.

A high pressure cell for dielectric investigations should meet some requirements. It should be easy to handle, the pressure range should exceed at least 250MPa (=2,5kbar), the pressure should be homogenous and hydrostatic through the cell, a broad frequency and temperature range should be achievable and the pressure medium should be incompatible with most of the (polymer-) samples. At least all three parameters, frequency, temperature and pressure should be controllable with a automatic computer routine.

The Setup

The setup shown in Figure 1 consists of four parts: 23t hydraulic closing cell press, sample cell, pump for closing pressure and pump for hydrostatic test pressure. Silicone oil is chosen as pressure transducing medium. The sample is placed inside the cylindrical sample cell and the electrodes are directly connected to the electrical feedthrough in the plug, which seals the cell. This plug is kept close with the hydraulic press. The outside pressure to the plug exceeds the maximum inside pressure (300MPa) by 10%. This avoids problems, which would arise if the system is closed with a threaded flange. The cell is made of high-grade stainless-steel, which is stable against pressure and temperature. The inside radius of the cell is 12,5mm and the height is 38mm. The coat is ca. 20mm thick.

Silicone oil is feed into the cell via a capillar tube in the bottom of the pressure cup. One has to make sure, that there is no interaction between the silicone oil and the sample each time a new sample is studied. To separate the sample from the polymer it is possible to cover the condensator with a thin layer of epoxid resin. If the layer is thin enough, there is no influence in the measurement.

The pressure is measured by an electrical pressure sensor at the pump in a distance of 500mm from the cell, but may also be determined directly in the sample cell. The system is heated by an external heating device which surrounds the cell. Temperatures from room temperature up to 280°C are achievable. In principle it is although possible to cool the system down to about - 100°C, but this causes viscosity problems with the pressure transducing medium. The temperature is measured at the sample location with a PT100 device. Each time a new pressure value is adjusted a sufficient time has to be allowed to equilibrate the system with respect to pressure and temperature.

The system is optimized with respect of the ease of handling, because of the special design of the plug. It is easy to replace and adjust and therefore allows for an easy process of sample change.

To change a sample, one has to prepare a capacitor, place it into the cell, connect it with the electrical feedthroughs, fill the cell with silicone oil and close it by pressing the plug with the hydraulic pump into the cell.

With the heating device, the system is heated to the desired temperature, which takes about 45 minutes for a temperature increase of about 100K. Then the desired pressure is applied with a hand operated pump and a frequency sweep is started with a conventional Novocontrol system.

It is planned to make the whole pressure system commercial available.

Results

Figure 3 shows the dispersion of the ionic conductivity of the solid polyelectrolyte I-6,10-Me-BF_4. This is an amorphous polycationic single ion conductor with BF_4^- mobile anions. This polyelectrolyte is described in more detail elsewhere [5]. Preparation of the sample capacitor is described in [6].

A strong effect of pressure is observed, resulting in a decrease of conductivity with increasing pressure. This behaviour is to be correlated to the well known the rise of glass transition temperature with pressure. The general shape of the curves does not change with pressure applied except that a shift of the plateau region to lower frequencies is observed.

These measurements help to further characterize the mechanisms of ion transport mechanisms in solid polyelectrolytes.

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Dielectric Spectroscopy of Polymeric Materials

by James P. Runt, John J. Fitzgerald (Eds.)

American Chemical Society, Washington, DC 1997, ISBN 0-8412-3335-7, 461 pages, hardcover, US$ 124,95

This book provides a comprehensive discussion of the fundamental principles in dielectric spectroscopy. It examines methods used in data modelling and some special techniques such as high-frequency dielectric measurements and thermally stimulated currents. It also discusses applications of dielectric spectroscopy in polymer solutions, polymer blends, polymeric liquid crystals, and other polymeric systems. This book is intended for industrial, academic, and government polymer chemists and chemical engineers, materials chemists and materials scientists or analytical chemists working with polymers.

Contents: G. Williams: *Theory of Dielectric Properties*; F. Kremer, M. Arndt: *Broadband Dielectric Measurement Techniques*; A. Schönhals: *Dielectric Properties of Amorphous Polymers;* R. H. Boyd, F. Liu: *Dielectric Spectroscopy of Semicrystalline Polymers*; R. Diaz-Calleja, E. Riande: *Calculation of Dipole Moments and Correlation Parameters*; S. Havriliak, Jr., S. J. Havriliak: *Unbiased Modelling of Dielectric Dispersions*; S. Mashimo: *Application High-Frequency Dielectric Measurements of Polymers*; G. Teyssedre, S. Mezghani, A. Bernes, C. Lacabanne: *Thermally Stimulated Currents of Polymers*; K. Adachi: *Dielectric Relaxation in Polymer Solutions*; J. P. Runt: *Dielectric Studies of Polymer Blends*; D. E. Kranbuehl: *Dielectric Monitoring of Polymerization and Cure*; G. P. Simon: *Dielectric Properties of Polymeric Liquid Crystals*; P. Avakian, H. W. Starkweather, Jr., J. J. Fontanella, M. C. Wintersgill: *Dielectric Properties of Fluoropolymers*; C. U. S. Fu, M. H. Ostrowski, H. S. Lackritz: *Dielectric Studies of Polymeric Nonlinear Optical Materials*; F. Kremer: *Broadband Dielectric Spectroscopy and Collective and Molecular Dynamics in Ferroelectric Liquid Crystals*.

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On the Accuracy of Dielectric Measurements

In modern dielectric spectrometers, the complete measurement procedure including control of devices and evaluation of the data is automatically performed by computer control. Nevertheless, the quality and the accuracy of the results strongly depend on the proper preparation of the sample. This is especially important if one is interested in absolute values of the dielectric function. This article gives an overview on the most important sources of inaccuracies and some practical recommendations concerning the preparation of samples for accurate dielectric material measurements in combination with Novocontrol dielectric spectrometers.

Introduction

The principle of a dielectric measurement is shown in fig. 1. The sample material is placed between two round metal electrode plates in order to act as a sample capacitor as shown in fig. 2. An generator applies an ac voltage $U(\omega)$ of the frequency $\omega/2\pi$ to one of the capacitor plates. The sample current $I(\omega)$ is determined at the other capacitor electrode by means of a vector current meter that measures in addition to the current amplitude I_0, the phase shift φ of the current with respect to the voltage. For easy calculation and representation of the formulas it is convenient to use complex notations.

$$U(\omega) = U_0 \sin(\omega t) = \mathrm{Re}(U^* \exp(i\omega t))$$

$$I(\omega) = I_0 \sin(\omega t + \varphi) = \mathrm{Re}(I^* \exp(i\omega t))$$

with

$$U^* = U' + iU'' = U_0$$

and

$$I^* = I' + iI''$$

$$I_0 = \sqrt{I'^2 + I''^2}$$

$$\tan(\varphi) = \frac{I''}{I'}$$

From this, the complex measured sample capacity C_m is evaluated by

$$C_m^* = -i\frac{I^*}{\omega U^*} \quad (1).$$

On the other hand, the dielectric function of the sample material is related to the complex capacity of an ideal sample capacitor (see Edge Capacities below for details) by

$$\varepsilon^* = \varepsilon' - i\varepsilon'' = \frac{C_s^*}{C_0} \quad (2)$$

C_0 is the vacuum capacity of the empty sample capacitor. In a first approximation

$$C_s^* \approx C_m^* \quad (3).$$

The accuracy of this method depends on several factors described in the following sections.

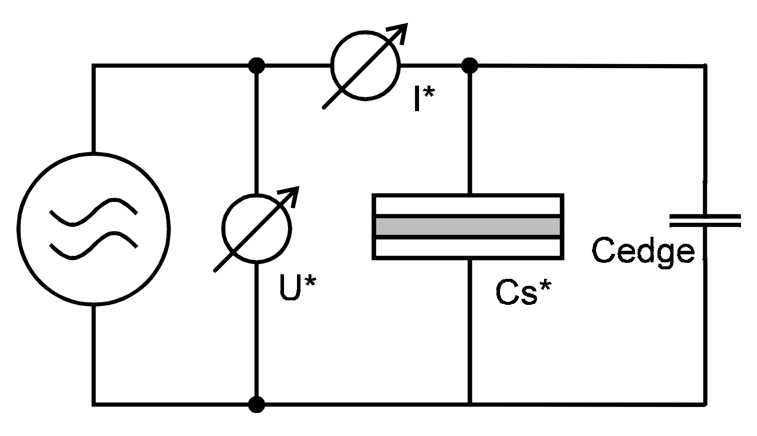


Fig. 1 : Principle arrangement for a dielectric material measurement

I) Inaccuracies resulting from the measurement system

This includes the accuracy of the impedance analyzer and the influence of cables and sample cell. This accuracy strongly depends on the frequency of measurement and the actual sample capacity. It should be specified in detail in the manual of the analyzer used. Unfortunately, most specifications apply only if the measured impedance is directly connected to the analyzer input neglecting the effects of the cables and the sample cell. On the other hand, for high quality impedance analyzers, the cables and sample cells are the dominant sources of errors which limit the overall performance of the system both at the low and high frequency ends of the measurement range.

Better results can be achieved if a system with active sample cell is used, where the impedance converter is incorporated in an optimized sample cell. Those systems can be specified for impedance accuracy at the position of the sample rather than at the analyzer BNC inputs. An example is the Novocontrol Broadband Dielectric Converter BDC which is specified for the frequency range from 10 µHz to 10 MHz and from -160°C to 500°C at the sample position.

II) Inaccuracies resulting from the sample preparation

Another source of error in the dielectric function evaluation arises from the nonideal preparation of the sample capacitor. That means, the system measures accurately the complex capacity Cm* of the sample capacitor, but eqn. (3) is not fulfilled. From our experience, the following four sources of error are the most important ones :

i) Inaccuracies in the sample geometry

These result mostly from uncertainties of the sample thickness. It should be noted, that for a sample with a thickness of 0.1 mm, the error in the thickness has to be less than 1µm for a 1% accurate result. The thickness of most

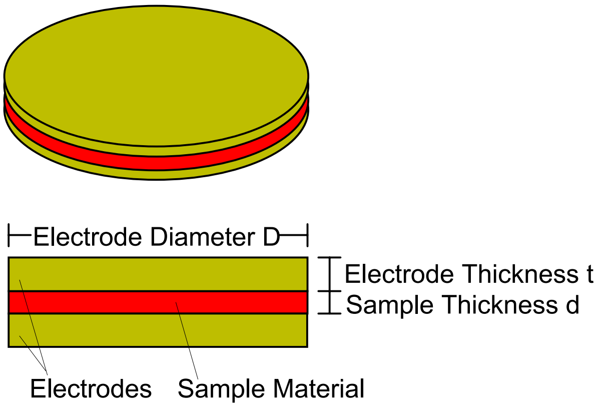


Fig. 2 : External electrodes with the sample material in "sandwich" arrangement

samples will not be homogenous within this range. Moreover, pressure might be applied to the sample which may lower the sample thickness when the sample is mounted between the electrodes of the sample cell.

ii) Bad electrical contacts between sample material and electrodes

These are often caused by inhomogeneous sample surfaces. In this case only a part of the sample material will be in contact with the electrodes. In addition, bad contacts may be caused by microscopic layers covering either the metal electrodes or the sample material (e.g. oxide layers). Bad contacts generally lower the measured ε values and / or introduce additional losses. The contact problem can be solved in most cases if a metal film (e.g. gold) is evaporated on both sides of the sample surface. At least the sample should be covered with a layer of silver paint (solution of colloidal silver). From our experience, for accurate measurements of absolute values, metal deposition on the electrodes is a must. Nevertheless, even in these cases, the measured results may depend on the metal used for sputtering or evaporation, therefore one should use metals of good conductivity (gold, silver, aluminium).

Sample metallization is especially important if materials with large dielectric permeabilities are measured. (e.g. a ceramic material with $\varepsilon' \approx 1500$ can be easily measured as low as 25 without metallized sample surfaces).

iii) Edge capacities

In the preparation technique described above, the sample material is directly prepared between two external electrodes. The sample capacitor forms a kind of sandwich structure as shown in fig. 2. After the preparation, the sandwich is placed between the electrodes of the sample cell. An additional third electrode which acts as a guard ring is not used. This technique has the advantage, that the sample preparation is very easy and straightforward. Disposable electrodes with several diameters can be used for optimal adaptation of the sample material to the impedance measuring system. In this way, the sample cell electrodes are not contaminated by the sample material.

On the other hand, eqn. (3) will be only exact, if the electrical field distribution is homogenous inside the sample capacitor and zero outside. In practice, there will be a non-homogeneous field at the capacitor edges reducing the sample capacity. In addition to this, the field outside the capacitor will not be zero, but there will be a stray field, which contributes to the measured sample capacity as well. Both contributions can be taken into account by an additional capacity C_{edge}. The measured capacity can be written as

$$C_m^* = C_s^* + Cedge \quad (4)$$

where Cm* is the measured capacity and *Cs** the capacity of the ideal capacitor.

The edge capacity could be reduced, if a sample cell with a third guard electrode is used. The disadvantage of this set-up is that the sample preparation becomes more difficult as no disposable electrodes can be used. In addition, at high frequencies technical problems are involved. On the other hand, the errors in a non guarded set-up can be easily kept below other error sources if either a convenient sample cell geometry is chosen and / or the edge capacity is taken into account in the evaluation. Comparing the benefits of a guarded set-up with its disadvantages, Novocontrol favours the second solution without guarding.

C_{edge} can be exactly calculated for two round capacitor plates centred in an evacuated and grounded metallic sphere with infinite radius (capacitor not filled with sample material) /1/.

$$\frac{Cedge}{C_0} = \frac{2d}{\pi D}\left[\ln\frac{8\pi D}{d} - 3 + z(x)\right];$$
$$z(x) = (1+x)\ln(1+x) - x\ln(x);$$
$$x = t/d \qquad (5)$$

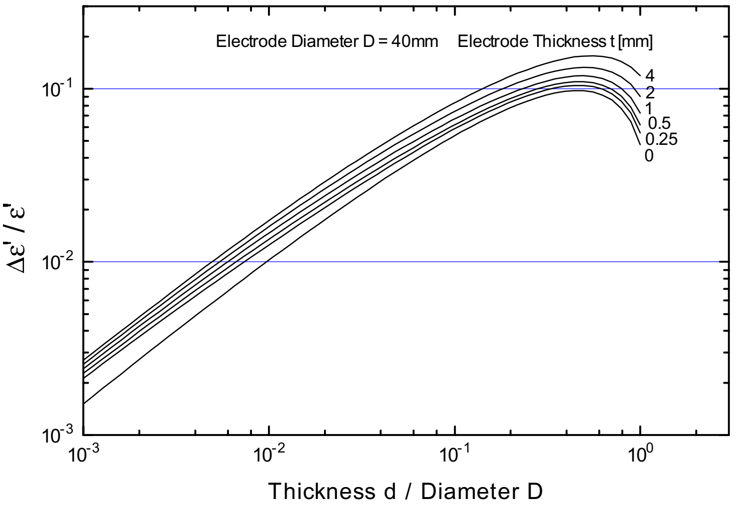


Fig. 3 : Error due to edge effects (without edge compensation) for an electrode with diameter D = 40 mm for several electrode thicknesses t and a dielectric constant ε' = 3

$$C_0 = \varepsilon_0 \frac{\pi\left(\frac{D}{2}\right)^2}{d}$$

D : electrode diameter, d : spacing between electrodes, t : electrode thickness, $\varepsilon_0 = 8.85 \cdot 10^{-12} As/Vm$

If the volume between the capacitor plates is completely filled with a dielectric material, which does not overlap the capacitor plates as shown in fig. 2, the edge capacity will approximately not be changed by the material. In this case the ideal sample capacity *Cs** can be calculated from eqn. (4) and (5). This was verified for several geometries and dielectric materials by /2/ with the result that the residual errors of this method are < 1.1 % of the measured values for all tested samples.

In addition the ratio $Cedge/C_0$ can be used to estimate the maximum error without edge compensation. The relative error of a measured dielectric function ε' due to edge effects (without compensation) is

$$\frac{\Delta\varepsilon'}{\varepsilon'} = \frac{Cedge}{C_s'} = \frac{Cedge}{C_0\,\varepsilon'}$$

Δε' / ε' is shown in fig. 3 as a function of electrode thickness d / diameter D for a material with ε' = 3. The error increases with the electrode thickness and decreases with the ratio d/D. Nevertheless, even for d/D = 1, the errors remain always below 20%. For d/D > 0.3, the error decreases with increasing d/D as the additional edge capacity outside the capacitor is over compensated by the inhomogenous field distribution inside the sample material which has a negative capacity effect on *Cedge*.

iv) Electrode polarisation

For samples having ionic conductivity, mobile ions can migrate within the sample material, hence giving rise to potential differences at the electrode surfaces. This leads to a large increase in both the real and imaginary part of the dielectric function at low frequencies /3, 4/.

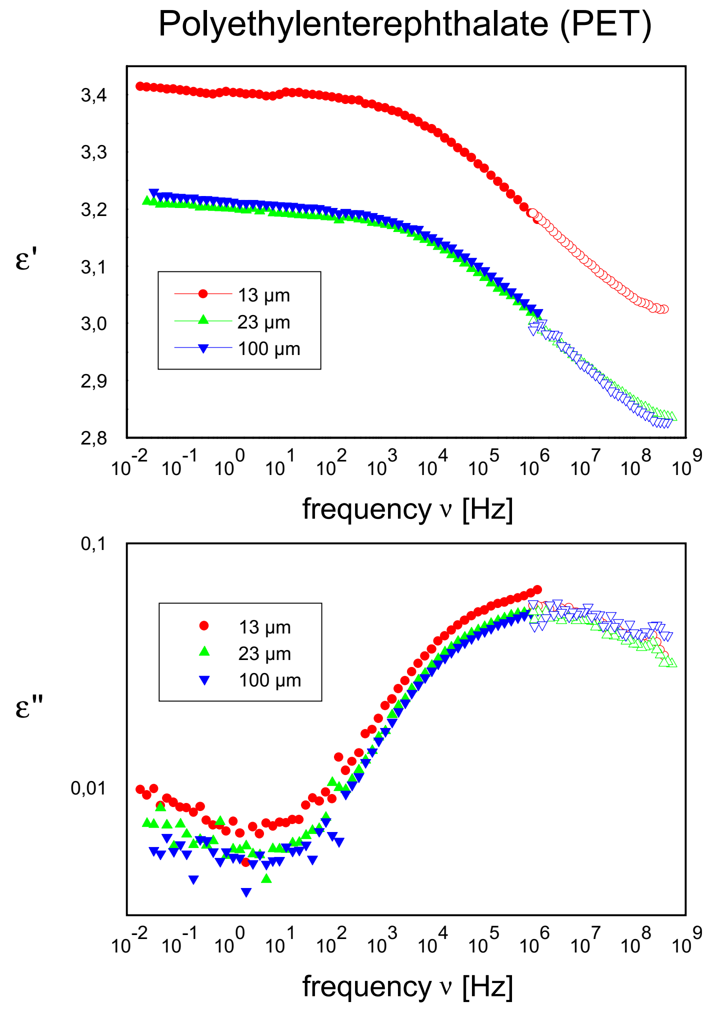


Fig. 4 : Determination of the absolute value of both real and imaginary part of the dielectric function. In the frequency range from 10^{-2} Hz to $2 \cdot 10^{6}$ Hz frequency response analysis was employed, between 10^{6} Hz to 10^{9} Hz coaxial reflectometry was used. It is demonstrated how well both measurement system coincide.

Measurement example

The above techniques were applied to three Polyethylenterephthalate (PET) films with different thickness of 13 µm, 23 µm and 100 µm. The samples were prepared and measured by /5/ using Novocontrol equipment described in /6/. In order to cover the broad frequency range from 10^{-2} to 10^{9} Hz, different measurement systems have to be combined, being based on different measurement principles. At frequencies below 1 MHz, frequency response analysis was applied. At the higher frequencies, coaxial line reflectometry was used. For each method two different samples were prepared. Fig. 4 shows the measured complex dielectric function of the three films. The results show the absolute measured values with edge correction applied. The 23 µm and 100 µm films show a nearly perfect match for all four samples over the whole frequency range with a maximum deviation of less than 1%. For the thinnest (13 µm) film, the values of ε' and ε'' were measured about 6% higher compared to the other two films. Nevertheless, this is well inside the limits of expectation, considering that 6% accuracy corresponds to a uncertainty of the film thickness of 800 nm. The results impressively demonstrate the validity of the measurement technology and systems even for thin films with thickness down to 10 µm.

Recommendations for sample preparation in combination with the Novocontrol systems

Both for the Novocontrol low frequency dielectric sample cell BDS 1200 and for the high frequency sample cell BDS 2100, the sample material should not be prepared directly between the contacts of the sample cell. Instead, the sample material should be prepared in sandwich structure between additional disposable electrodes as shown in fig. 2. Gold plated external electrodes are available from Novocontrol with diameters from 40 mm .. 10 mm for the low frequency systems (below 10 MHz) and 6 mm - 3 mm for the RF system (above 1 MHz).

For solid samples, the distance between the electrodes is determined by the sample thickness. For optimal measurement results, the sample geometry should be chosen according the following rules :

- For the low frequency systems (below 10 MHz), good performance will be reached with sample capacities from 50 pF .. 200 pF (optimum around 100 pF) in the frequency range from 100 kHz .. 10 MHz. At lower frequencies, also higher capacities up to 2 nF will yield good results. Note : The system will be able to measure also capacities in a wide range from 0.1 pF up to 1F depending on frequency, but the accuracy decreases outside the ranges specified above.
- For the high frequency systems (1 MHz - 1.8 GHz) a good sample capacity range is 2 pF .. 10 pF (optimum around 4 pF). Smaller capacities yield better results at high frequencies and vice versa.

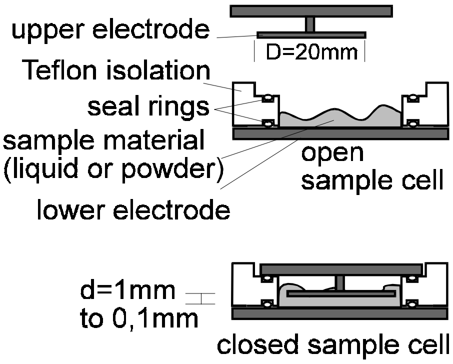


Fig. 5 : Priciple drawing of the Novocontrol liquid sample cell

If the dielectric constant ε' of the sample is approximately known, the sample capacity can be estimated from

$$C = \varepsilon' \varepsilon_0 \frac{\pi D^2}{4d}$$

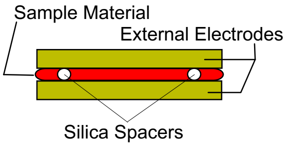
or

$$\frac{C}{\text{pF}} \approx 6.95 \cdot \varepsilon' \frac{(D/\text{mm})^2}{(d/\mu\text{m})} \quad (5)$$

where D is the diameter of the capacitor electrodes and d the distance between them.

- In order to minimize the errors due to edge effects, the ratio d/D should be kept as small as possible. If edge compensation is used, the diameters of both disposable electrodes and the sample material should be equal. Edge compensation is supported by the latest version of the Novocontrol instrument control and evaluation software WinDETA 3.5 /7/.
- The surface of solid samples should be as flat as possible in order to get good contact with the external electrodes. In addition it is highly recommended to metallize the sample surface in the contact regions. This can be done by evaporation or sputtering a metal film on the sample. Another easier and cheaper way is to cover the sample surface with a solution of colloidal silver.

If liquid samples have to be measured, additional spacers can be used in order to adjust the electrode spacing as shown in the figure below.



The spacer material should have a low and frequency-independent dielectric constant and low losses. Convenient spacer materials are e.g. thin silica needles (available from Novocontrol) or small stripes of Teflon or Polyethylene. The effect of the spacers on the measurement can be compensated by the settings of the WinDETA software.

For some low viscosity liquids with high partial pressure, sample material may evaporate out of the standard sandwich capacitor especially at higher temperatures. In order to prevent evaporation, the sealed liquid sample cell BDS 1309 can be used which is shown in fig. 5.

In the open state, the upper electrode is removed from the cell, and the sample material poured out on the bottom of the cell acting simultaneously as the lower electrode. In the closed state, liquid sample material that does not fit between the electrodes can flow around the upper electrode. The two seal rings attached to the teflon isolation prevent evaporation of sample material out of the cell.

The BDS 1309 is mounted in the same way as the standard sandwich capacitor between the electrodes of the BDS 1200 or BDS-S active sample cell.

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Novocontrol News

Novocontrol extends the program for material characterization methods by two new systems.

New TSDC system based on QUATRO available

This system was developed in co-operation with the University of Potsdam. It is based on the Novocontrol QUATRO Cryosystem with a new sample cell for TSDC (Thermal Stimulated Depolarization Current) measurements for temperatures from -160°C .. 500°C. A Keithley 6517A electrometer is used for current measurement from 10^{-15}A .. $2 \cdot 10^{-2}$A and polarization voltage up to 1000V. Measurements are fully automated controlled, evaluated and graphically displayed by the new software package WinTSDC under MS-Windows. The software supports time, temperature and DC Voltage dependent measurements of the depolarization current. In addition a new method with sinusoidal temperature profiles for pyroelectric measurements is included. The system and application examples are described in the first article of this newsletter. The TSDC is especially favourable as an extension to a Novocontrol Dielectric system, as the QUATRO temperature control system and the computer can be used for both systems in this case. Therefore, Novocontrol can offer the TSDC extension at a very competitive price.

New System for Pressure Dependent Dielectric Measurements up to 300MPa (3kBar)

This system was developed in co-operation with the Max Planck Institute for Polymer Research in Mainz. It adds pressure dependency as an additional independent variable to the existing ones (frequency, temperature, dc-bias and time). The system uses a commercially available 23t pressuring system in combination with a new sample cell for dielectric measurements. The dielectric function is measured between 10 µHz and 1 MHz by a Novocontrol dielectric spectrometer based on a Broadband Dielectric Converter BDC. The Temperature range is currently from ambient to 280°C. It may be extended to -100°C if another pressure transducing medium is used. The system is controlled by the Novocontrol software WinDETA. This set-up is the first commercially available system for dielectric or impedance measurements at high pressure. As a commercial system, it is designed for easy operation, wide pressure, frequency and temperature range. The system and application examples are described in the second article of this newsletter.

Novocontrol WWW Homepage

On the international front, the demand for information on the features of NOVOCONTROL products has considerally increased in the past several years.
To fulfill this demand consistently and continuously, NOVOCONTROL decided to install its own homepage in the World Wide Web.
For more information on our scientific and industrial products and expert application support, please visit us on the Internet at http:\\www.novocontrol.com

Conference Announcements

The Dielectrics Society Annual Conference
Rutherford College
University of Kent at Canterbury
6 - 9th April 1998
Topic: Anisotropic Dielectrics and Ferroelectrics
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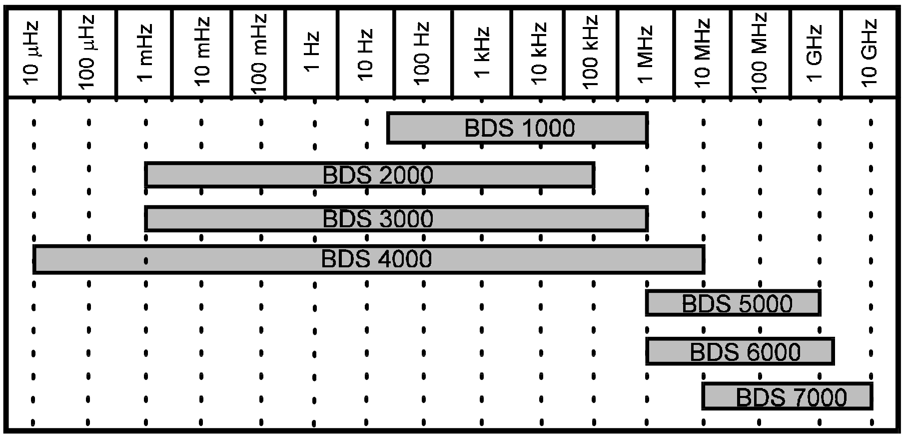
OVERVIEW
BROADBAND DIELECTRIC SPECTROSCOPY
over 15 decades by NOVOCONTROL



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Abstracts and papers are always welcome. We can publish max. 2 pages A4 on each subject. Please send your script to the editor.

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Division ....

Name ....

Title ....

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City ....

Postal Code ....

Country ....

Phone ....

Fax ....

Dielectric Spectrometers and Modules

☐ Economical dielectric spectrometers
(1 mHz - 1 MHz or 100 Hz - 1 MHz)

☐ Broadband dielectric spectrometer
(10 µHz - 10 MHz)

☐ RF dielectric spectrometer
(1 MHz - 1.8 GHz)

☐ Temperature control systems
(+20°C to +400°C or -160°C to +500°C)

☐ Broadband Dielectric Converter

☐ MS-Windows software for automatic measurement of dielectric relaxations, impedance, electrical and magnetic material properties

☐ MS-Windows software for automatic curve fitting data analysis (WLF, VFL) and equivalent circuit transformation

New Instruments

☐ TSDC Instruments

☐ Pressure dependent dielectric spectrometers

Status of my request

☐ Urgent demand. Please send me also your quotation per fax.

☐ Future demand. Please contact me again when I can order your equipment.
This will be: ....
☐ General interest

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