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The AC and DC Conductivity (Dielectric Constant) of Matrix Dominated Composites

This paper will show how to analyze the AC and DC conductivity, or complex dielectric constant, of matrix dominated binary composites. In these composites, one phase (the matrix) surrounds the granular (particle) phase at all volume fractions (save for $\phi = 0$ or 1). The matrix can be the better or the less conducting phase. We discuss in detail only the case where the matrix is the resistive phase, a microstructure that is the most often encountered in practice. In order for a composite to qualify as matrix dominated, the length of the electrical path between the "grains" must be greater than the tunneling distance for the dominant charge carriers (usually electrons). By this definition, most polymer-nanotube composites, despite their morphology, are not matrix dominated as electrons are usually able to tunnel through the polymer layers between the tubes at their points of closest contact. Such composites are thus percolation systems which are discussed in a previous newsletter [1] and in References [2, 3].

Modeling Composite Data

The AC conductivity of the composites can be modeled using circuits giving a similar response at all frequencies. One of the simplest circuit arrangements is a series connection of two parallel resistor/capacitor elements, Fig. 1. The impedance for ideal resistors and capacitors are $Z^* = R$ and $Z^* = 1/(i\omega C)$, respectively. For suitable values of the resistances and capacitances, this circuit gives rise to semi-spherical arcs in the complex impedance plane which is discussed later. This is the basis of equivalent circuit analysis of impedance spectra, with the circuit elements and their arrangements

usually being more complex. In particular, the measured data is seen to give rise to depressed arcs as in the complex plane, a response not typical of ideal capacitors and resistors.

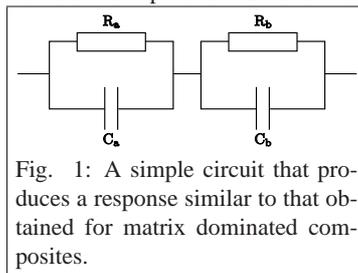


Fig. 1: A simple circuit that produces a response similar to that obtained for matrix dominated composites.

Therefore *constant phase elements* (CPEs) have to be added to the circuit when equivalent circuits are used to fit and model data. The CPEs are usually, but not always, placed parallel with, or in place of, one or more of the capacitors in the circuit. While the individual resistances and capacitances are usually associated with particular constituents of the composite, the constant phase elements are often associated with some non-homogeneity of the system or a distribution (dispersion) of the value of some physical property of the system. Among other things, the CPEs have been associated with electrode roughness, inhomogeneous reaction rates on a surface, varying thickness or composition for coatings and non-uniform current distributions across sample surfaces [4] and the references therein. This paper shows that, in many or most cases, the CPE represents/models the dispersive properties of the conductivity of the components of the composites. The impedance of the CPE is given by the phenomenological equation [4, 5]

$$1/Z = Y = Q_0(i\omega)^n,$$

where Q_0 is the admittance ($1/|Z|$) at $\omega = 1$ rad/s and $0 \leq n \leq 1$. When $n = 1$, this is the same equation as that of the impedance of a dispersionless capacitor. Q_0 and n may both be temperature dependent [5]. The circuit in Fig. 1 is by no means unique, the same response can be obtained using different circuit arrangements and different resistor and capacitor val-

ues. In some cases it becomes unclear what the circuit elements represent and how they relate to the microstructure being studied. Examples of other configurations are the circuits shown in Fig. 2 which, for appropriate values of the resistances and capacitances, give the same response as the circuit in Fig. 1.

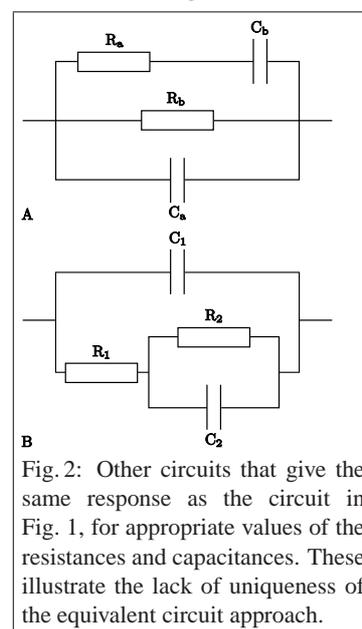


Fig. 2: Other circuits that give the same response as the circuit in Fig. 1, for appropriate values of the resistances and capacitances. These illustrate the lack of uniqueness of the equivalent circuit approach.

The lack of uniqueness of the equivalent circuits and that circuits are obtained which have elements that cannot be readily associated with the composition and microstructure of the composites are major disadvantages of the equivalent circuit approach.

A different approach to the modeling of AC conductivity data is the use of effective media theories and/or the Brick Layer model. Each of these models corresponds to a particular class of microstructure and often requires a single or only a few adjustable parameters, depending on the microstructure used to arrive at a particular model.

The first effective media equations for spherical inclusions were those due to Maxwell for a dilute dispersion of spheres in a host matrix. In the dilute limit, all effective media equations reduce to the Maxwell

equation which is [6]:

$$\Sigma_m = \frac{\Sigma_d + 2 - 2\Phi(1 - \Sigma_d)}{\Sigma_d + 2 + \Phi(1 - \Sigma_d)} \quad (1)$$

where $\Sigma_m = \sigma_m/\sigma_i$, $\Sigma_d = \sigma_c/\sigma_i$ for the insulator host case (dispersed conducting spheres) and $\Sigma_m = \sigma_m/\sigma_c$, $\Sigma_d = \sigma_i/\sigma_c$ for the conductor host case (dispersed insulating spheres). σ_m , σ_c and σ_i are the conductivities of the composite, the more conducting component and the less conducting component, respectively. In either case Φ is the volume fraction of the dispersed phase.

The first two terms in the series expansion of Eq. 1 yield the limits

$$\sigma_m = \sigma_i(1 + 3\phi), \quad \sigma_c \rightarrow \infty, \quad (2)$$

for a dilute suspension of perfectly conducting spheres (insulator host) and

$$\sigma_m = \sigma_c(1 - 3/2f), \quad \sigma_i \rightarrow 0, \quad (3)$$

for a dilute suspension of perfectly insulating spheres (conductor host). Here ϕ is the volume fraction of the more conducting component and $f = 1 - \phi$ is the volume fraction of the less conducting component. These equations are strictly only valid for extremely small ϕ or f , but in practice can often be used for ϕ and $f \leq 0.1$.

The AC conductivity of the media (composite), σ_m , is the sum of the real and imaginary conductivities, which is given by $\sigma_m = \sigma_{mr} + i\sigma_{mi}$. The conductivity of the more conducting component is given by

$\sigma_c = \sigma_{cr} + i\sigma_{ci}$ or simply $\sigma_c = \sigma_{cr}$ if ideal conductivity ($\sigma_{cr} \gg \sigma_{ci}$) is assumed. For the insulating component, the conductivity is $\sigma_i = \sigma_{ir} + i\sigma_{ii}$ where $\sigma_{ii} = \omega\epsilon_0\epsilon_{ir}$. σ_i is often approximated as $\sigma_i = i\omega\epsilon_0\epsilon_{ir}$ (i.e. $\sigma_{ir} \ll i\sigma_{ii}$). In practice, σ_{ir} incorporates both a, usually very small, DC conductivity and the dielectric polarization loss term ($\omega\epsilon_0\epsilon_{ii}$). The expressions for σ_c and σ_i can be frequency and/or temperature dependent. The complex conductivity and dielectric constant are related by $\sigma = i\omega\epsilon_0\epsilon$. In principal, all the effective media equations can be written in terms of the complex permittivities ($\epsilon^* = \epsilon_r + i\epsilon_i$) or, for magnetic materials, the complex permeability ($\mu^* = \mu_r + i\mu_i$). The properties of matrix dominated composites are usually best described using the Maxwell-Wagner effective media equation (also known as the Maxwell-Garnet equation). This model is formally equivalent to the Hashin-Shtrikman lower bound (insulator host) and upper bound (conductor host) equation and will thus be referred to as the MW-HS model [2, 7, 8]. The microstructure upon which the MW-HS model is based is shown in Fig. 3 [9, 10].

This microstructure consists of a large range of conducting spheres coated by a more insulating component with a constant volume ratio of the conducting and more insulating components. The ideal microstructure shown in Fig. 3 is not

found in practice. Figure 4 shows a real matrix dominated microstructure of a ceramic. The high resistivity grain boundaries (matrix) dominate the electrical properties. The insulator host MW-HS microstructure generally leads to two arcs in complex impedance plane plots if the impedances of the individual components are in a suitable range. On the other hand, the conductor host MW-HS microstructure generally leads only to a single arc in the complex impedance plane as there is a continuous conducting path, through the matrix, which dominates the electrical properties of the composite at all but vanishingly small ϕ values.

For the insulator host and conductor host MW-HS microstructures, the complex conductivity of the binary composite σ_m is obtained from: [10]

$$\frac{\sigma_m - \sigma_i}{\sigma_m + 2\sigma_i} = \phi \frac{\sigma_c - \sigma_i}{\sigma_c + 2\sigma_i}, \quad (4)$$

and

$$\frac{\sigma_m - \sigma_c}{\sigma_m + 2\sigma_c} = (1 - \phi) \frac{\sigma_i - \sigma_c}{\sigma_i + 2\sigma_c}, \quad (5)$$

respectively. Equations 4 and 5 are often written in terms of ϵ_m, ϵ_i and ϵ_c and are then called the Clausius-Mosotti relationships. The microstructure upon which the Brick Layer Model (BLM) is based consists of cubic conducting bricks surrounded by insulating mortar, Figure 5 [11, 12].

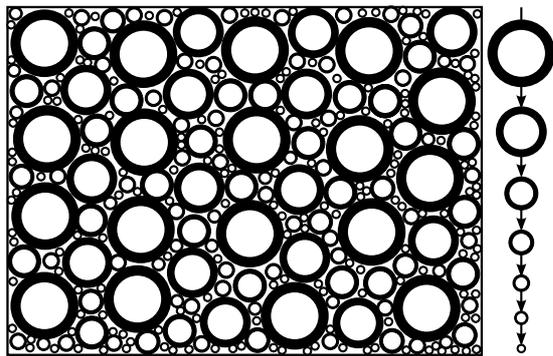


Fig. 3: The ideal insulator host Maxwell-Wagner microstructure consists of a large array of conducting spheres (white) surrounded by a less conducting component (black). In order that the volume is completely filled, there must be a large range of sphere sizes. The volume ratio of the interior to the exterior remains constant for all the coated spheres. In the conductor host microstructure, the white is the insulating spheres surrounded by the more conducting component (black) [13].

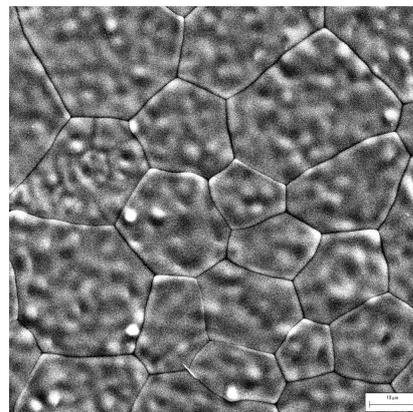


Fig. 4: An SEM micrograph showing the microstructure of a Yttria Stabilized Zirconia ceramic. This microstructure with the conducting grains and insulating grain boundaries approximates that described by the insulator host Maxwell-Wagner model. (Micrograph courtesy of Dr. Tim Armstrong, Oak Ridge National Laboratory, U.S.A.)

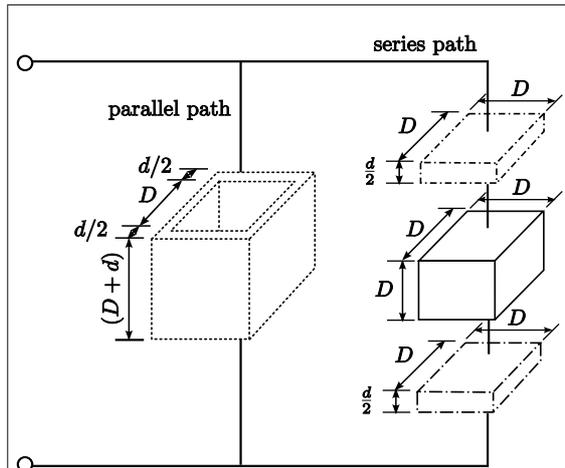


Fig. 5: The microstructure for the Brick Layer Model. This microstructure is composed of conducting bricks (solid) and insulating mortar (dash and dot-dash). Note the end caps (dot-dash).

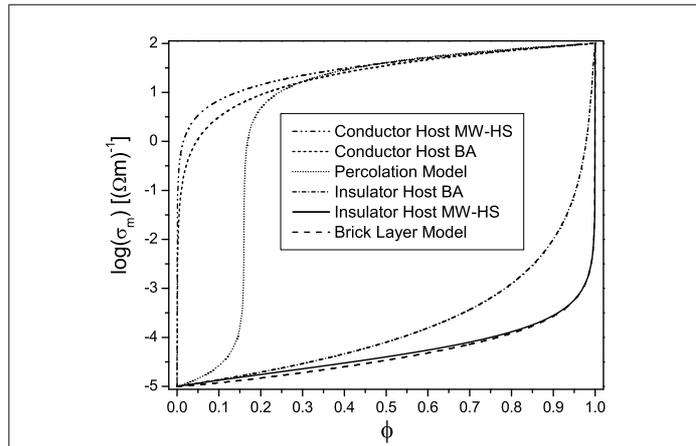


Fig. 6: Simulated dc conductivity curves for insulator host and conductor host matrix dominated composites as well as for a percolation system. For the simulations $\sigma_c = 10^2 (\Omega m)^{-1}$ and $\sigma_i = 10^{-5} (\Omega m)^{-1}$. The percolation system simulation uses Eq. 1 in [1] with a percolation threshold $\phi_c = 0.16$ and critical exponents $s = t = 1$.

The impedance of the composite medium is given by:

$$Z_m = \frac{1}{\frac{1}{Z_i^\parallel} + \frac{1}{Z_i^\perp + Z_c}} \quad (6)$$

where Z_i^\parallel is the complex impedance of the square pipe, Z_c for the cubic brick and Z_i^\perp for the end caps.

It can be shown [13] that Eq. 6 can be expressed in the form

$$\sigma_m = \sigma_i \left[1 + \frac{\sigma_c - \sigma_i \phi}{\sigma_c - (\sigma_c - \sigma_i) \phi^{1/3}} \right] \quad (7)$$

from which the volume fraction of the conducting component (ϕ) can be readily obtained. Another set of effective media equations found in the literature are the Bruggeman asymmetric (BA) media equations for spherical inclusions or grains. The microstructure described by these equations consists of a space filling array of spheres of all sizes as shown in Figure 7. The conductivities of the insulator host and conductor host composites are then given by: [2, 6, 9, 14]

$$\frac{(\sigma_m - \sigma_c)^3}{\sigma_m} = (1 - \phi)^3 \times \frac{(\sigma_i - \sigma_c)^3}{\sigma_i} \quad (8)$$

and

$$\frac{(\sigma_m - \sigma_i)^3}{\sigma_m} = \phi^3 \times \frac{(\sigma_c - \sigma_i)^3}{\sigma_c} \quad (9)$$

respectively.

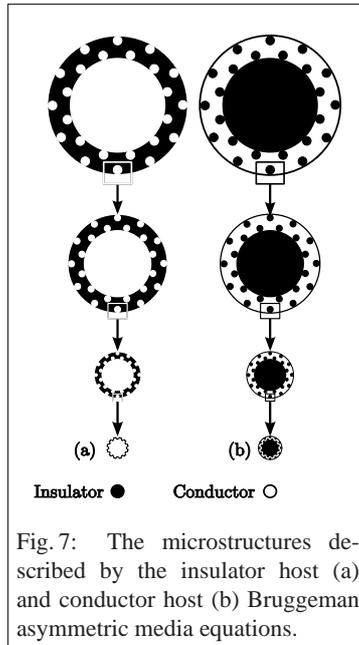


Fig. 7: The microstructures described by the insulator host (a) and conductor host (b) Bruggeman asymmetric media equations.

This equation is not widely used, but some instances of its use in dc conductivity experiments is given in [6].

Figure 6 shows the DC conductivity curves for matrix dominated composites described by Eq. 4 and of a percolation system [1, 2, 9]. For the conductor host systems, the conductivity rises to nearly that of the more conducting components at ϕ s very close to zero while in insulator host systems it remains close to that on the matrix until ϕ is very close to one. Neither the MW-HS nor the Brick Layer model has a percolation threshold, where the electrical conductivity changes extremely rapidly for ϕ not equal to 0 or 1.

Component Properties and the AC Conductivity

When analyzing the properties of practical composites, it is found that the properties of the components of the composite are usually dispersive. In this case, suitable relations to describe these dispersive properties must be used when analyzing the composite data.

Figures 8 and 9 show the ac conductivity data of a polycrystalline Yttria Stabilized Zirconia (YSZ) ceramic with a microstructure such as shown in Fig. 4 at various temperatures. From crystals of Yttria Stabilized Zirconia, it was shown [13] that the properties of the grains are best described using the Universal Dielectric Response (UDR) function although a number of other models exist [13]. The UDR is: [15, 16]

$$\sigma(\omega) = \sigma_{dc} + A\omega^s + i\omega \times (\epsilon_\infty + A\omega^{s-1} \tan(s\pi)/2) \quad (10)$$

where the exponent $0 \leq s \leq 1$ and A is a coefficient. The data is modeled using Eq. 4, with the UDR model used to fit the properties of the grains and non dispersive but temperature dependent properties for the grain boundaries. With the volume fraction of the grains ϕ taken to be temperature independent, as would be expected, it can be seen that the MW-HS model fits the experimental data well. In this case $\phi = 0.9991 \pm 0.0020$ which corresponds to a grain boundary thickness of 5 nm.

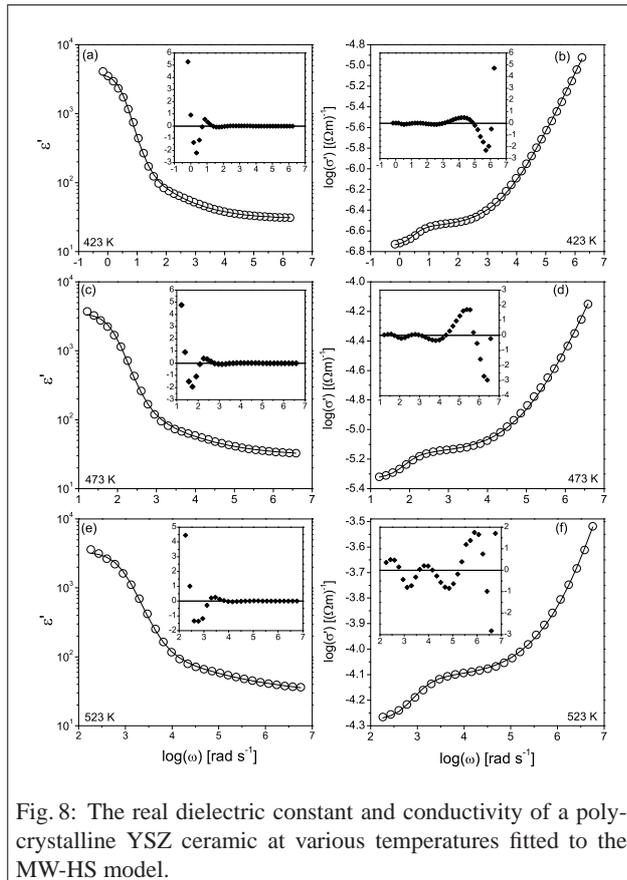


Fig. 8: The real dielectric constant and conductivity of a polycrystalline YSZ ceramic at various temperatures fitted to the MW-HS model.

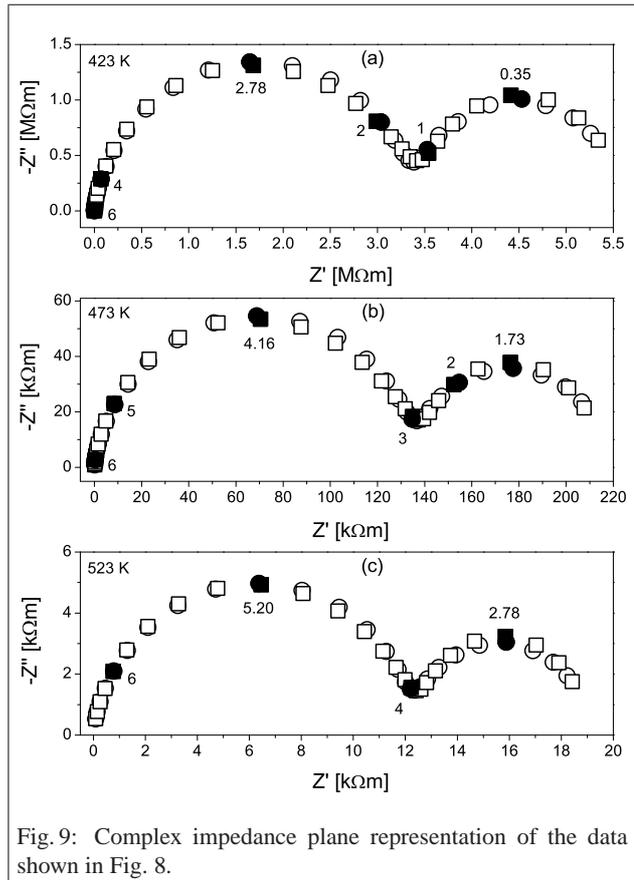


Fig. 9: Complex impedance plane representation of the data shown in Fig. 8.

The ceramic data can also be modeled using the Brick Layer Model leading to a fit which is nearly as good as that obtained with the Maxwell-Wagner model [13]. The reason that these two models which are based on an extremely different or contrasting microstructures is because the samples analyzed are in the extreme dilute limit (see Eqs. 1, 2 and 3). The BLM is widely used in the literature to model the electrical properties of ceramic materials and is explored in detail in [12, 17, 18].

Conclusion

This paper has shown how the AC and DC conductivity (dielectric constant) results of matrix dominated media can and perhaps should be analyzed in terms of the effective media models. The Maxwell-Wagner/Hashin-Shtrikman and Brick Layer models can be successfully used to model real world systems whose microstructure deviates somewhat from the ideal microstructures from which they are derived. In analyzing the data for the composites, it is important to accurately model the dispersive properties of the components (grains or matrix).

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Broadband Dielectric Spectroscopy as a Tool to study Diffusion Coefficients in Conducting Glass-Forming Systems

Due to its unique ability to probe molecular fluctuations and charge transport over a broad frequency and temperature range, broadband dielectric spectroscopy (BDS) has proved indispensable in the quest to understand the underlying mechanisms of charge transport and dynamic glass transition in ion conducting glass-forming systems [1–5]. Since decades, it is well established that these processes are dominated by diffusion [6]. For conducting systems, BDS delivers DC conductivity which is a product of mobility (consequently, the diffusion coefficient) and the number density of charge carriers. This has the limitation that one is unable to unambiguously separate

the effect of each of these two quantities on charge transport. Here we present a novel approach to circumvent this difficulty and provide a direct access to the diffusion coefficient over an unprecedented range. This opens up a new field of possibilities in the study of charge transport and dynamic glass transition.

The dielectric spectra of conducting systems are characterised by a plateau σ_0 (dc conductivity) and a critical frequency ω_c describing the onset of the dispersion of σ' . Empirically, it is known that $\omega_c \cong \omega_M \cong 2\pi/\tau_c$, where ω_M is the radial frequency corresponding to the peak in the imaginary part of the electrical modulus and τ_c is a characteristic time that defines the attempt rate $\omega_e = \tau_e^{-1} = 2\pi f_e$ of the charge carriers to overcome the highest energy barrier (limiting the σ_0), thereby enabling the physical interpretation of σ_c within the random barrier model [1–5,7]. Within the framework of this model, charge carriers hop in a random spatially varying potential landscape. The transport process is governed by the ability of charge carriers to overcome the randomly distributed barriers. The highest barrier that must be overcome to achieve an infinite cluster of hopping sites determines the onset of dc conductivity [8]. Solved within the Continuous-Time-Random Walk approximation, Dyre [7] obtained the following analytical expression for the complex conductivity:

$$\sigma(\omega) = \sigma_0 \frac{i\omega\tau_e}{\ln(1+i\omega\tau_e)}. \quad (1)$$

The dielectric spectra of an ionic liquid 1-butyl-3-methylimidazolium tetrafluoroborate -BMIM BF₄ is shown in Fig. 1, together with fits (eq. 1) at different temperatures. From elementary electrodynamics and using the Einstein and Einstein-Smoluchowski equations, the dc conductivity can be expressed as:

$$\sigma_0 = nq\mu = \frac{nq^2}{kT}D = \frac{nq^2}{kT} \frac{\lambda^2}{2\tau_h}, \quad (2)$$

where D denotes the diffusion coefficient, n is the effective number density of the charge carriers, q the elementary charge, k the Boltzmann

constant, T the temperature, λ the hopping length and τ_h the characteristic hopping time. By setting $\tau_h \cong \tau_e$ and taking, for the hopping length, values comparable to the Pauling diameter [9], i.e., $\lambda = 0.17$ nm (for 1-butyl-3-methylimidazolium tetrafluoroborate -BMIM BF₄) and $\lambda = 0.19$ nm (for MMIM Me–2PO₄), diffusion coefficients are readily determined. Independent measurements performed by PFG NMR (which measures the diffusion coefficient directly), together with the diffusion coefficients obtained from dielectric measurements, are shown in Fig. 2. As evident from the results, this approach yields diffusion coefficients in excellent agreement with those given by PFG NMR. We have recently shown that the approach holds for other conducting glass-forming systems as well [10–13]. This opens up a new field of possibilities for the application of broadband dielectric spectroscopy to study charge transport in conducting glass forming systems.

Two methods, PFG NMR measuring the mass transport cations and anions and BDS probing charge transport, are combined. Based on the Einstein and Einstein-Smoluchowski equations diffusion coefficients are determined under the assumption that the hopping length λ is equal to the Pauling diameter.

This is proven explicitly by considering that the measured diffusion coefficient is a contribution of both the anions and the cations within the linear response regime. Quantitative agreement is found between the diffusion coefficients from PFG NMR and BDS. This enables one to separate $n(T)$ from $\mu(T)$ and to analyse the character of their thermal activation. For $\mu(T)$ a Vogel-Fulcher Tamman type is obtained, whereas $n(T)$ exhibits an Arrhenius-type temperature dependence.

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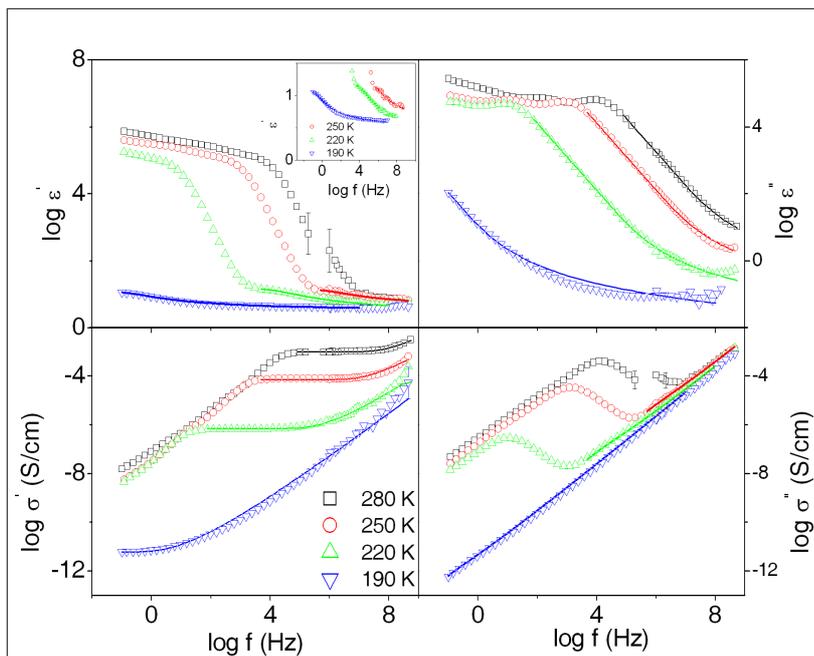


Fig. 1: Complex dielectric function and complex conductivity of [BMIM][BF₄] as a function of frequency at different temperatures, as indicated. The fits are made using eq. (1). Inset: the real part of the complex dielectric function frequency enlarged to show fits by the random barrier model. The fit parameters are: for 190 K: ($\sigma_0 = 4 \cdot 10^{-12} \text{ S/cm}$, $\tau_e = 1.55 \text{ s}$), 220 K: ($\sigma_0 = 1 \cdot 10^{-7} \text{ S/cm}$, $\tau_e = 2 \cdot 10^{-5} \text{ s}$), 250 K: ($\sigma_0 = 5.1 \cdot 10^{-6} \text{ S/cm}$, $\tau_e = 3.5 \cdot 10^{-7} \text{ s}$) and $\epsilon = 3 \pm 1$. The error bars are comparable to the size of the symbols, if not specified otherwise.

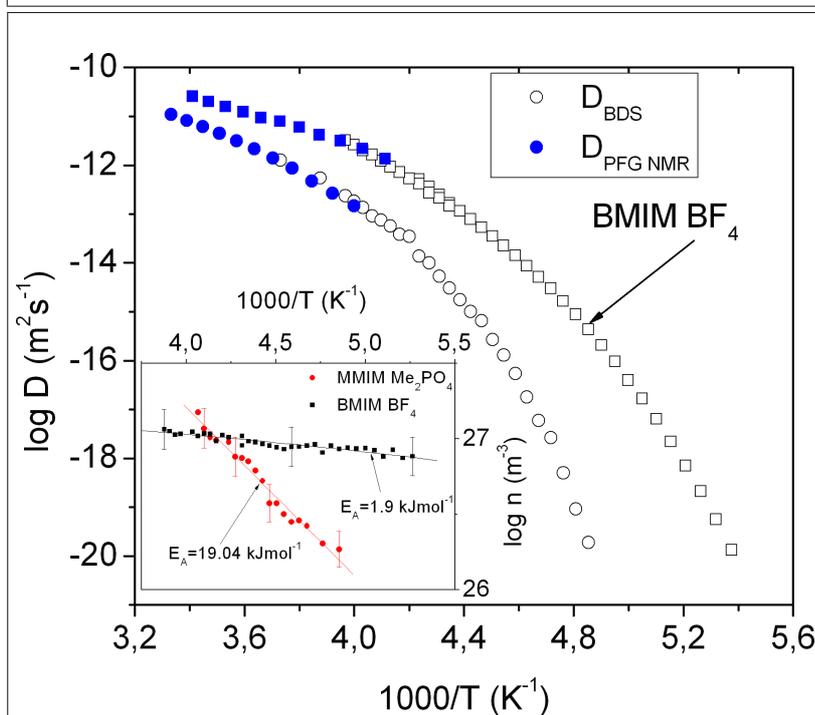


Fig. 2: Diffusion coefficient determined by the novel approach involving application of the Einstein-Smolukowski equation (using ω_c as hopping rate and with λ equal to the Pauling diameter of the ions as hopping length), compared with the diffusion coefficient measured by PFG NMR (in blue colour) for two ionic liquids: BMIM BF₄ and MMIM Me₂PO₄ [6]. Inset: effective number of charge carriers as a function of inverse temperature (the respective activation energies are also indicated). The error bars are comparable to the size of the symbols, if not specified otherwise. Log is used to refer to logarithm to base 10.

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Announcement: Training Course on Broadband Dielectric and Impedance Spectroscopy

Responding to frequent demand from scientists interested in Broadband Dielectric Spectroscopy (BDS), Novocontrol Technologies announces its training course entitled "Broadband Dielectric and Impedance Spectroscopy" to take place **September 28-30, 2009**, at the **University of Marburg**, Germany under the joint guidance of Profs. B. Roling and F. Kremer.

The workshop will not only give an overview on the basics of this experimental technique, but also will elaborate on the following topics:

- fundamentals of Broadband Dielectric Spectroscopy (BDS)
- broadband dielectric spectroscopy on polymers and glass forming liquids
- relaxational dynamics in confined systems, including thin films
- characterisation of electrochemical cells
- electrode polarisation and double-layer formation
- analysis of dielectric and impedance spectra dominated by charge transport

Seminar blocks will be complemented by hands-on experience—under professional guidance, participants perform and evaluate their experiments, if possible on their own samples.

The regular participation fee for this two-day workshop will be € 1500 (€ 700 for academic users). The number of participants is limited. Spaces are allocated on a first come - first serve basis.

Prospective participants are kindly asked to register by email to workshop@novocontrol.de.

D. Wilmer, Novocontrol

Broadband Dielectric Spectroscopy and Its Applications 2008 in Lyon

The 5th International Conference on Dielectric Spectroscopy and Its Applications was held at Lyon 2 University on 26-29 August 2008 in an Amphitheatre named after the famous scientist Jean-Marie Ampère. Organised by Gisèle Boiteux, Gerard Seytre and Isabelle Stevenson helped by the Local Organisation Committee and the PhD students from IMP/LMPB in UMR CNRS 5223, this conference followed the tradition of earlier *International Dielectric Society* (IDS)

and *Dielectric Relaxation Phenomena* (DRP) joined meetings held in Jerusalem 2001 (only IDS), Leipzig 2002, Delft 2004 and Poznan 2006.

BDS 2008 was attended by over 220 scientists presenting 74 talks including 23 invited lectures and 139 posters, describing current research using Broadband Dielectric Spectroscopy (BDS). The large attendance included a high proportion of young scientists.

The variety of topics in the invited talks was exceptional, ranging from the very fundamental (molecular dynamics) to applications of BDS to complex systems (heterogeneous and nanoconfined systems, functional materials, biological systems) with a special session on charge transport. The conference ended by a session on broadband relaxation spectroscopy and its new developments to open the perspective in this research field. As in the previous conferences, many presentations described the molecular dynamics (S. Cappacioli on behalf of K. Ngai, A. Angel, J. Fothergill, Y. Feldman and R. Nigmatullin). A special session dedicated to heterogeneous and nanoconfined systems was well represented by M. Wübbenhorst, K. Fukao, J. Torkelson, J. Runt and A. Serghei. Newer topics included (i) functional materials for which invited lectures were given by R. Gerhard, T. Ezquerra, J. Kenny, G. Floudas and J. Dyre; (ii) biological systems represented by F. Bordi, C. Lacabanne and J. Ulanski. A small session on charge transport was represented by F. Kremer and B. Roling. The session on Broadband Relaxation Spectroscopy and new developments concluded the conference with contributions by A. Schönhals, I. Alig and R. Richert. The large number of posters (most of them on functional materials) complemented the above talks. Poster prizes sponsored by Novocontrol Technologies were awarded

to W. Yamamoto, A. Schoenhals, P. Demont, M. Jasiurkowska, D. Cangialosi, E. Laredo and J. Runt. In the measurement forum, G. Schaumburg gave an overview on NOVOCONTROL equipment in parallel to a presentation of the SETARAM instrumentation.

The *Peter Debye Prize for Young Investigators for Excellence in Dielectric Research*, sponsored by NEXANS was awarded in equal parts to two researchers, i.e., *Periklis Papadopoulos*, now at The University of Leipzig (Germany), and *Daniele Prevosto*, researcher in the Polylab CNR-INFN in Pisa.

On Wednesday evening conferencees enjoyed a boat excursion along the rivers Rhône and Saône followed by dinner in the prestigious restaurant *Abbaye de Collonges*, owned by Paul Bocuse. Another special event was the already traditional concert *Dielectricians play for Dielectricians* on Thursday evening with contributions of F. Kremer, M. Wübbenhorst, R. and C. Gerhard, the Stevenson brothers and M. Wübbenhorst's young daughter.

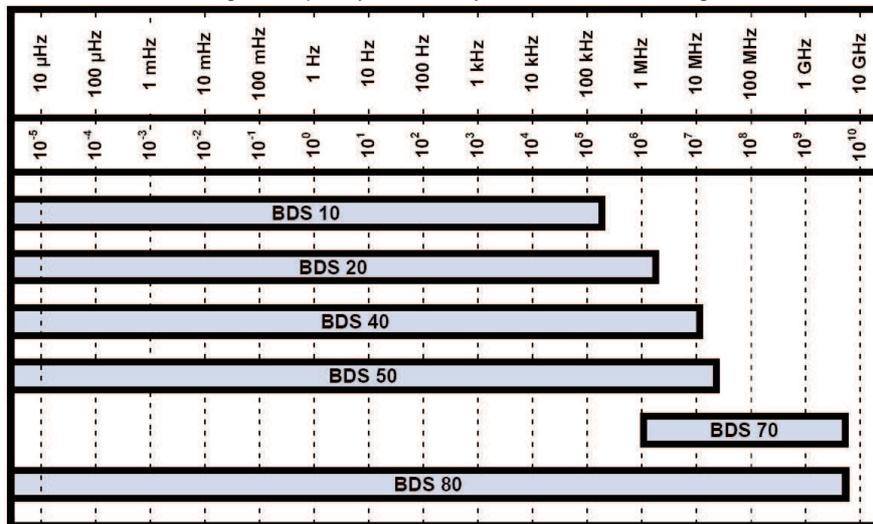
As a conclusion, the organisation of BDS2008 was excellent in every way: nice and warm weather for the whole week and "good food for the brain and the stomach" as John Berberian suggested at the end. We thank Gisele Boiteux and her team for all they did before and during the meeting to make this such a successful and memorable meeting in the beautiful city of Lyon. Further details can be seen at <http://bds2008.univ-lyon1.fr/>.

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Isabelle Stevenson, Lyon



OVERVIEW

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covering 16 frequency decades by Novocontrol Technologies

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