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B. Roling

Conductivity spectra of disordered ionic conductors: Calculating the timedependent mean square displacement of the mobile ions

introduction

In disordered ionic conductors, such as doped crystals, glasses, and polymers ion transport is considered to arise from thermally activated hopping movements of the mobile ions between different positions in a solid or supercooled liquid matrix.

Conductivity spectroscopy is a well established method for characterising the hopping dynamics of the ions. A variety of different materials have been studied using this technique, and a vast amount of experimental data have been published. For the analysis and interpretation of these data, different methods are being employed. Very often. the frequencyand temperaturedependent spectra are fitted using power Jonscher-type law expressions and the power law exponents obtained thereby are interpreted [1-3]. Other researchers prefer to use the electric modulus formalism and interpret the peak width of the imaginary part of the modulus [4-7]. Again, others describe the electrical properties by means of equivalent circuits and try to understand the physical meaning of different elements in these circuits [8].

In this context, it is surprising that very seldom is the *most direct* information obtainable from frequency-dependent conductivity spectra, that is the time-dependent mean square displacement of the mobile ions, used. *Absolute values* for this quantity can be calculated

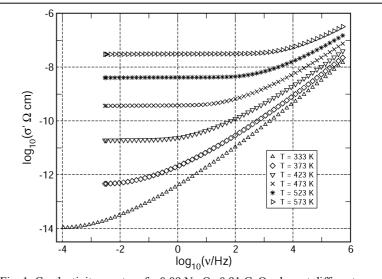


Fig. 1. Conductivity spectra of a 0.09 $Na_2O^+0.91~GeO_2$ glass at different temperatures.

if two conditions are fulfilled. (i) The applied electric field strengths are low, so that linear response theory [9-11] is applicable. Linear response theory relates the conductivity spectra to the equilibrium dynamics of the ions, i.e. the dynamics in the absence of an electric field.

(ii) The Haven ratio is of the order of unity. That means correlations between the movements of *different* ions are weak and do not influence strongly the frequency dependence of the conductivity.

In the case of disordered ionic conductors, both conditions are generally fulfilled. When one compares the frequency-dependent conductivity of different materials, it is evident that the spectra usually display a quasi-universal shape, i.e. the spectral shape is very similar with different for materials structures and different number densities of mobile ions [1, 2, 12, 13]. Below a characteristic crossover frequency v^* , the real part of the conductivity, $\sigma'(v)$, is independent of frequency and identical to the dc conductivity. According to linear response theory this implies that on time scales > $1/(2\pi v^*)$, the movements of ions in

thermal equilibrium are diffusive, i.e., the mean square displacement $\langle r^2(t) \rangle$ increases linearly with time.

At frequencies $v > v^*$, the real part of the conductivity, $\sigma'(v)$, increases with frequency. This implies that on time scales $< 1/(2\pi v^*)$, the mean square displacement $<r^2(t)>$ increases *sublinearly* with time. Therefore, the short time dynamics is often called 'subdiffusive'.

Subdiffusive movements of the ions on short time scales seem to be a characteristic of almost all disordered ionic conductors. The reason for the quasi-universal spectral shape is still unclear at present. One common viewpoint is the presence of a quasi-universal microscopic mechanism of the ion transport in different disordered materials.

An alternative assumption is that the macroscopic conductivity displays a much more universal behaviour than the microscopic ion dynamics, implying that a lot of information about differences in the microscopic ion dynamics is lost due to the macroscopic averaging over all ions in the sample.

In this paper, I present experimental evidence in favor of

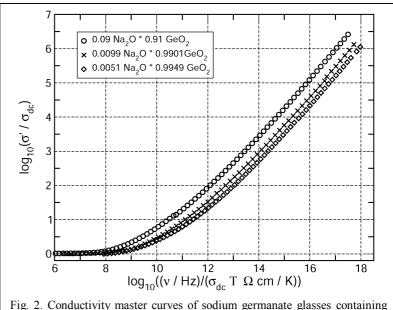
second viewpoint. the This evidence is based on an analysis of the mean square displacement where the subdiffusive ion dynamics passes over into the diffusive dynamics. In the following, this quantity will be denoted by $\langle r^2_{cr} \rangle$, and its square root $\sqrt{\langle r_{cr}^2 \rangle}$ will be called the 'length scale of the subdiffusive ion dynamics'. It will be shown that for ion conducting glasses with different alkali oxide contents and different types of network forming oxides, $\langle r^2_{cr} \rangle$ depends strongly on composition.

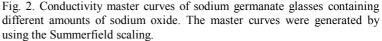
In other words, the length scales of the subdiffusive ion dynamics are non-universal, even though the spectral shapes are quasi-universal. These results suggest that $\langle r^2_{cr} \rangle$ contains valuable information about the microscopic ion dynamics and is, therefore, an important quantity to consider when ion transport processes in disordered materials are modeled.

Ion conducting glasses containing a network forming oxide, and an alkali oxide as a network modifier, are well suited as model systems influence since the of compositional changes on the conductivity spectra can be easily studied. When the alkali oxide content is varied, the number density of the mobile ions changes as well as the structure of the glassy network. On the other hand, when the type of the network forming oxide is varied for a given alkali oxide content, one changes only the network structure, but not the number density of the mobile ions. Thus, by analysing both the influence of the alkali oxide content and the influence of the network forming oxide on the conductivity spectra, it should be possible to distinguish between the respective influences on the microscopic ion dynamics exerted by (i) the number density of the mobile ions and, thus, by the interionic Coulomb interactions and (ii) by the structure of the glassy network.

experiment

Glasses of compositions $x \text{ Na}_2\text{O}$ (1 - x) GeO₂ with x = 0.0051, 0.0099, 0.0201, 0.051, 0.09, 0.213, and 0.40; $x \text{ Na}_2\text{O}$ (1 - x) B₂O₃ with x = 0.10, 0.15, 0.20, 0.25, and 0.30; and $x \text{ Li}_2\text{O}$ (1 - x) B₂O₃ with





x = 0.128, 0.226, and 0.333 were prepared by using the melt quenching technique.

For the preparation of the germanate glasses, Na_2CO_3 and GeO_2 were dried in a platinum crucible at 500 °C for 30 min. Then the raw materials were held at 1000 °C for one hour in order to ensure a complete evaporation of carbon dioxide. After melting the raw materials at 1300 °C for 90 minutes, the melts were cast onto a platinum plate.

For the preparation of the borate glasses, Li_2CO_3 , Na_2CO_3 and B_2O_3 were dried at 170 °C for 1 day and then ground to fine powder using a mortar. The raw materials were melted in a platinum crucible at 1000 °C for 40 min and then cast onto a platinum plate.

All glasses were annealed 30 K below their respective glass transition temperatures for 2 h and then cooled down to room temperature with a rate of 1 K/min. The surfaces of all samples were ground and polished, and metal electrodes were sputtered onto the sample surfaces. Frequency- and temperature-dependent

conductivities were measured using an HP 4192A Impedance Analyzer and a Novocontrol Alpha-S High Resolution Dielectric Analyser.

results and discussion

Fig. 1 shows the frequency dependence of the real part of the conductivity, $\sigma'(v)$, for the glass

0.09 Na₂O 0.91 GeO₂ at different temperatures. The spectra display the typical shape found for many disordered ionic conductors. At low frequencies, the conductivity is independent of frequency and identical to the dc conductivity of the glass. The dc conductivity of ion conducting glasses exhibits generally an Arrhenius-type temperature dependence. At higher frequencies, there is a crossover into a dispersive regime with a strong increase of the conductivity with frequency. The onset frequency of the conductivity dispersion is also thermally activated, and it is generally observed that the corresponding activation energy is identical to the activation energy of the dc conductivity [2, 12-14].

The spectral shape of the conductivity isotherms shown in Fig. 1 is independent of temperature, and therefore, these isotherms can be superimposed by using the so called 'Summerfield scaling' [15]. In this scaling approach, the conductivity axis is normalized by the dc conductivity σ_{dc} , and the frequency axis is normalized by the product $\sigma_{dc} - T$, which by the Nernst--Einstein relation

$$\sigma_{dc} \cdot T = \frac{N_V q^2}{k_B} \frac{1}{H_R} D \quad (1)$$

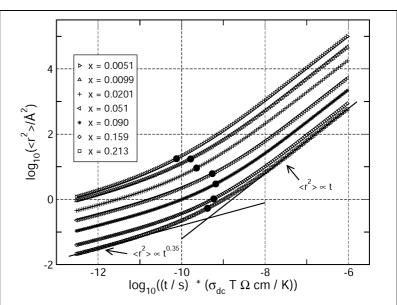
is proportional to the coefficient of self diffusion of the mobile ions, D. Here, N_V and q are the number density and the charge of the mobile ions, respectively, while k_B denotes Boltzmann's constant. H_R is the Haven ratio describing correlations between the hopping movements of *different* mobile ions.

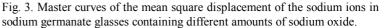
Using the Summerfield scaling procedure, conductivity master curves were generated. In Fig. 2, the master curves of sodium germanate glasses containing different amounts of sodium oxide are shown. Clearly, the shape of these master curves is quasiuniversal, although the sodium oxide content is varied by a factor of almost 18. However, the positions of the master curves on the $\nu/(\sigma_{dc} T)$ axis are different for different glasses.

We will now pass over from frequency-dependent conductivities to time-dependent mean square displacements. In Ref. [16] we have shown that in the framework of linear response theory the following relation between conductivity and mean square displacement can be derived:

$$< r^{2}(t) > = \frac{12k_{B}T}{N_{V}q^{2}\pi} \int_{0}^{t} dt'$$
$$\int_{0}^{\infty} \frac{\operatorname{Re}[\hat{\sigma}(v)\hat{H}_{R}(v)]}{v} \sin(2\pi v t') dv$$
(2)

Here, Re denotes the real part of a complex function. $\hat{H}_{R}(v)$ is a generalized, frequency-dependent complex Haven ratio, taking into account that the strength of many particle correlations may be different on different time scales. The Haven ratio H_R obtained from the Nernst-Einstein equation (1) is the low--frequency limiting value of $\hat{H}_{R}(v)$, i.e. $H_R = \lim_{v \to 0} \hat{H}_R(v).$ For ion conducting glasses with low alkali oxide contents (1 mol% and below), H_R is close to unity [17-19]. In highly modified glasses, H_R is generally around 0.3 [17, 19, 20]. In the limit of high frequencies, $\hat{H}_{R}(v)$ is expected to approach unity, independent of the ionic concentrations. This is because on very short time scales the ionic hops should be independent [12, 21]. Therefore, $\hat{H}_{R}(v)$ should be a weak function of frequency. The real part of the Haven ratio, $H'_{R}(v)$, should increase from H_R at





frequencies below the onset frequency of the conductivity dispersion to unity at high frequencies. Both Maass et al. and the present author have shown by Monte Carlo simulations that this is, indeed, the case for the hopping motions of interacting particles in disordered potential landscapes [21, 22].

In the case of our low frequency conductivity spectra, $\hat{H}_R(v) \approx H_R$ should, therefore, be a good approximation. The mean square displacement of the mobile ions is then given by:

$$< r^{2}(t) > = \frac{12k_{B}TH_{R}}{N_{V}q^{2}\pi} \int_{0}^{t} dt'$$

$$\int_{0}^{\infty} \frac{\sigma'(v)}{v} \sin(2\pi v t') dv \quad (3)$$

This equation was used to calculate $\langle r^2(t) \rangle$ curves for the germanate and borate glasses. Values for H_R were taken from the literature [17, 19, 20].

Fig. 3 shows master curves of the mean square displacement of the sodium ions in $x \text{ Na}_2\text{O}$ (1 - x) GeO₂ glasses with different amounts of sodium oxide, x. The applicability of the Summerfield scaling in combination with Eq. (3) leads to the following scaling law for the mean square displacement:

$$\langle r^2(t) \rangle = f(\sigma_{dc} T t)$$
 (4)

Here, f denotes a temperatureindependent function. This scaling law implies that upon a temperature change, the mean square displacement curves are shifted on the time scale, since the product $\sigma_{dc} \cdot T$ depends strongly on temperature. However, the values of $\langle r^2(t) \rangle$ at characteristic points of the curves are independent of temperature. In the limit of long times, $\langle r^2(t) \rangle$ is proportional to time, while at short times, the mean square displacement depends on time in a sublinear fashion, and the time dependence is, in a good approximation, given by $\langle r^2(t) \rangle \propto t^{0.35}$

Now, we define the time where the two lines with $\langle r^2(t) \rangle \propto t$ and $\langle r^2(t) \rangle \propto t^{0.35}$ intersect as the crossover time from subdiffusive to diffusive movements. In Fig. 3, these crossover times and the associated square mean displacements, $\langle r^2_{cr} \rangle$, are marked by filled circles. Note that for a given glass, $\langle r^2_{cr} \rangle$ is a temperature-independent quantity. The square root of this quantity, $\sqrt{\langle r_{cr}^2 \rangle}$ characterizes the length scale of the subdiffusive ion dynamics.

In Fig. 4, this length scale is plotted versus the alkali oxide content of the germanate and borate glasses, x. As seen from the figure, $\sqrt{\langle r_{cr}^2 \rangle}$ increases with decreasing alkali oxide content of the glasses. While in highly modified glasses,

 $\sqrt{\langle r_{\rm er}^2 \rangle}$ is smaller than 1 Å,

we obtain a value of approximately

9 Å for the sodium germanate glass

with 0.5 mol% sodium oxide.

Let us now consider in more detail the x-dependence of the length scale in the case of the sodium germanate glasses. At low sodium oxide contents, $\sqrt{\langle r_{cr}^2 \rangle}$ is proportional to $x^{-1/3}$, which implies that the length scale of the subdiffusive dynamics is proportional to the average distance between the mobile ions [16, 23].

One possible explanation for this observation is that in this composition range, the Coulomb interactions between the mobile ions play an important role for $\sqrt{\langle r_{cr}^2 \rangle}$. The strength of these interactions also scales with the average distance between the ions. This hypothesis is supported by the results of recent Monte Carlo simulations of the charge carrier dynamics in random barrier potential landscapes [22].

At high sodium contents, the decrease of $\sqrt{\langle r_{cr}^2 \rangle}$ with the sodium oxide content x is much more pronounced than at low sodium oxide contents. In contrast, the lithium and the sodium borate glasses display a $\langle r^2(t) \rangle \propto x^{-1/3}$ relationship at high alkali oxide contents also.

distinctive This dynamic behaviour of the alkali ions in highly modified germanate and borate glasses is most likely caused by differences in the structures of the glassy networks, since the number densities of the mobile ions and thus the strength of the interionic Coulomb interactions in these glasses are almost identical at the same sodium oxide contents.

A detailed discussion of the length scale / alkali oxide relations in germanate and borate glasses based on the structural peculiarities of these glasses can be found in Ref. [16]. The important point I would like to stress here is that the analysis of the length scale yields $\sqrt{\langle r_{cr}^2 \rangle}$ valuable information about the respective influences on the ion dynamics exerted by the interionic Coulomb interactions and by the network structure.

The strong composition dependence of the length scale $\sqrt{\langle r_{cr}^2 \rangle}$ provides strong evidence that the microscopic mechanisms of the ion transport in different

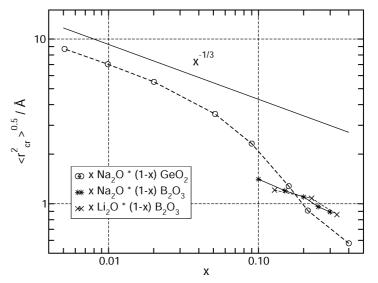


Fig. 4. Plot of the length scale of the subdiffusive alkali ion dynamics, $\sqrt{\langle r_{cr}^2 \rangle}$, versus the alkali content x of sodium germanate, sodium borate and lithium borate glasses.

glasses are non-universal, although the spectral shape of the conductivity is quasi-universal. For instance, the $\sqrt{\langle r_{ex}^2 \rangle}$ values obtained for sodium germanate glasses with 0.5 mol% and 40 mol% sodium oxide differ by a factor of approximately 15. Nevertheless, the spectral shape of the conductivity is virtually identical for these two glasses. This example demonstrates that the length scale of the subdiffusive ion dynamics is an important quantity to consider when microscopic mechanisms of the ion transport in disordered materials are discussed.

conclusions

For alkali germanate and alkali borate glasses, the time-dependent mean square displacement of the mobile alkali ions was calculated from frequency-dependent conductivity spectra, and the length scale of the subdiffusive ion dynamics was derived. It was shown that for a given glass, this length scale does not depend on temperature. However, the length scale depends strongly on glass composition. Generally, $\sqrt{\langle r_{cr}^2 \rangle}$ decreases with increasing alkali oxide content

For alkali germanate glasses with low alkali oxide contents x, $\sqrt{\langle r_{cr}^2 \rangle}$ is proportional to $x^{-1/3}$, implying that the length scale of the subdiffusive dynamics is

proportional to the average distance between the mobile ions. The origin of this distance dependence of the length scale could be from the long range Coulomb interactions between the ions.

At high alkali oxide contents, significant differences between germanate and borate glasses are found regarding the influence of the alkali oxide content x on the length scale $\sqrt{\langle r_{cr}^2 \rangle}$. These differences point to the influence of the network structure on the subdiffusive ion dynamics.

Our results show that conclusions on the universality of the microscopic ion dynamics can hardly be drawn without taking into account the length scale of the subdiffusive ion dynamics. It was demonstrated that for glasses with virtually identical spectral shapes of the conductivity, the length scales $\sqrt{\langle r_{cr}^2 \rangle}$ differ enormously, indicating that despite the similarities in the spectral shapes, the microscopic mechanisms of the ion transport are clearly distinct.

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B. Roling

Institut für Physikalische Chemie and Sonderforschungsbereich 458 (DFG) Westfälische Wilhelms-Universität Münster, Schlossplatz 4/7, 48149 Münster, Germany

G. Williams IDS 2002 international conference

The IDS 2002 Conference, 'Broad band Dielectric Spectroscopy and its Applications', a joint meeting of the $`2^{nd}$ Conference of the International Dielectric Society (IDS)' and the '7th Conference on Dielectric and Related Phenomena (DRP)' took place 2-6 September 2002 in the Physical Institute of the University of Leipzig. Organised by Prof. Friedrich Kremer, it was attended by 140 delegates; 60 formal talks and 81 posters were presented at Modern BDS the Meeting. techniques enable materials to be studied over wide ranges of frequency $(10^{-6} \text{ to } 10^{12} \text{ Hz})$, temperature and applied pressure.

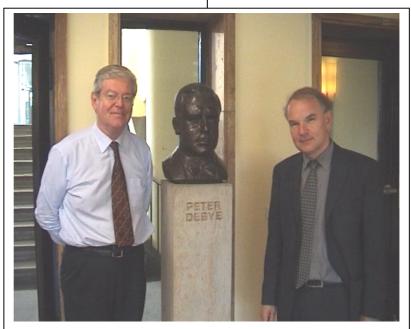
New results at the frontiers of dielectrics research were presented that revealed multiple dielectric relaxations and conduction processes in liquids, solids, liquid crystals and further dynamically disordered systems and were interpreted in terms of particular molecular dynamical processes.

Different themes were developed in the talks at the Meeting as follows.

New studies documented the BDS behaviour of (i) aqueous media (Kumbharkarne, Lyaschenko), (ii) non-associated and (iii) further associated (Hbonded) liquids. The behaviours of (ii) and (iii) were well-illustrated by the studies by Lunkenheimer et al where propylene carbonate and glycerol were each studied over 18 decades of frequency, revealing (a) the α -process (b) its high frequency wing and (c) the boson peak around 10^{12} Hz. In numerous systems a separate secondary (β) loss peak between (a) and (c) was observed.

The origins were discussed of (a) in terms microbrownian motions of whole molecules, and (b) and (c) in terms of cage-effects or local partial relaxations of dipoles in the liquid state (Ngai, Vij, Hinze, Yagihara, Rössler, Ryabov). The large effects of temperature (T) and pressure P (P)('the forgotten variable') on the α process in viscous liquids were revealed (Fontanella, Casalini) and, in combination with PVT data, the inadequacies of conventional freevolume concepts for dielectric relaxation in these materials were demonstrated

Some studies of bulk polymers were described, including those for homopolymers and block copolymers of polyoxybutylene of various molecular weights, architectures, and in different environments, that revealed segmental, normal-mode and secondary relaxations together with interfacial (MW) polarizations



Graham Williams Peter Debye

Friedrich Kremer Photo by M. Wubbenhorst

(Pissis), Poly(lactic acid) gave segmental and normal mode relaxations (Adachi). Segmental and normal mode relaxations for novel linear and star poly propylene oxides with reactive end groups were studied, giving real-time information on the changes in long chain dynamics of chains as a polymer network was formed (Mijovic).

The observation of multiple relaxations in miscible polymer blends (Leroy, Colmonero) and those containing H-bonded structures (Runt) revealed the static/dynamic presence of heterogeneities and concentrationfluctuations in these materials. For hyperbranched polymers the dielectric and dynamic mechanical behaviour indicated the presence of nanoheterogeneities and microphase separation (Boiteux).

Low frequency BDS studies were reported for thermotropic liquid crystalline polymers as a function of T and P (Mierzawa), together with those for a low molar mass LC (8CB) (Zhakarov). New non-linear dielectric studies of surface-aligned ferroelectric and anti-ferroelectric smectic liquid crystals were reported across a wide f-range (Kimura).

The current interest in the dynamics of molecules in spatiallyconfined environments, resulted in many papers/posters concerned with the BDS behaviour of constrained and free-standing ultrathin polymer films and of low molar mass liquids, polymers and liquid crystals contained in nanoand meso-porous materials (Fukao, Wubbenhorst, Kremer, Aliev, Schonhals). 'Hairy-rod' polymers, where the motions of the side groups are constrained within the periodic structure determined by the chain-backbones, were studied by BDS and NMR (Floudas).

The nature of dynamic heterogeneities in liquids, disordered solids and relaxor ferroelectrics was demonstrated using new pulsed DS and dielectric hole-burning techniques (Bohmer, Diezemann).

BDS studies embracing conduction in disordered solids (Malecki, Bohnke, Imre, Badot), Hbonded crystals (Kita), electrolytes (Grosse, Dygas), conductor/ insulator composites (Alig, McLachlan) were reported.

While there exists a large literature for the conduction behaviour of these materials, the new BDS techniques provide accurate data quickly over a wide frequency range which aid the understanding of (i) the common power law behaviour of real conductivity $\sigma'(\omega)$ at high frequencies for all disordered materials and (ii) the different processes that contribute to $\sigma'(\omega)$ heterogeneous for materials/composites such as carbon-loaded elastomers (Alig). Non-linear conductivity measurements of disordered ionconductors were presented for the first time (Roling). A theory of thermally stimulated depolarisation currents (TSDC) was given and was related to BDS (van Turnhout) and the technique was applied to polymers (van Turnhout, Leroy, Dantras).

A further theme was the BDS studies of heterogeneous systems that included bio-related molecules, percolation phenomena, microemulsions, dispersed/suspended systems containing cells, colloidal particles and organelles (Feldman, Gatash, Bruni, Yu, Ermolina, Shilov). Some agricultural applications of BDS were also described (Nelson).

Few papers were concerned with dielectric theory or simulations, but the basis of the Vogel-Fulcher-Tamman equation was investigated (Nigmatullin), the short-time behaviour of relaxation in liquids was described (Coffey) and extensive simulations of the segmental and long-range dynamics of polymer chains of different structures (linear, branched, hairyrod, etc) were described (Pakula). Further papers described relaxation studies that complement BDS (Richert, Kaatze).

A strong Poster Session included topics in the BDS of viscous liquids, polymers, liquid crystals, aqueous systems, confined liquid crystals and polymers, adsorbed species, dispersive charge transport in disordered solids, TSDC, biological and heterogeneous systems, inorganic crystals and newer methodological developments.

A successful Measurement Forum was arranged at which the latest BDS instrumentation was presented by manufacturers. A Panel Discussion of topics in BDS completed the scientific programme.

In addition, on the Wednesday a day-excursion was made to Berlin by over 100 delegates where an excellent lunch was taken on a tour-boat that journeyed leisurely by canal through the heart of the city in glorious sunny weather. This was followed by a visit to one of the main museums where ancient ruins of the Middle East were displayed. The day was completed with the Conference Dinner at a superb restaurant en route back to Leipzig.

The Conference was a great success in that it brought together scientists from many disciplines (physics, chemistry, polymer science, electrochemistry, solid state physics, biology, electrical, chemical and materials engineering) who share a common interest in the use of modern BDS techniques for the study of motional processes in a wide range of materials and chemical systems.

The delegates thank Prof. Kremer and his team for their excellent organisation of this Conference that provided us with a feast of dielectrics research and so many opportunities to discuss new concepts and ideas in the splendid environment of the city of Leipzig.

G. Williams

Department of Chemistry, University of Wales Swansea, Singleton Park, Swansea SA2 8PP, UK.

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OVERVIEW BROADBAND DIELECTRIC SPECTROSCOPY over 16 decades by NOVOCONTROL

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