

Dielectrics Newsletter

Scientific newsletter for dielectric spectroscopy

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Simona Bauer-Gogonea

Pyroelectric Investigations: Tool for the Study of Thermal, Elastic, and Electric Properties

The investigation of thermal, elastic and electric properties of thin dielectric films is of practical importance in many high-performance applications, i.e. sensor devices or microelectronics. Fast, reliable, and simple-to-use techniques for the determination of such material characteristics are highly demanded. As discussed below, the pyroelectric effect in dielectric materials provides an elegant means

i) for the measurement of pyroelectric, piezoelectric, elastic, and thermal properties of dielectrics /1-4/,

ii) for the investigation of dipole relaxation processes in amorphous polymers and phase transitions in ferroelectric polymers /1/, as well as

iii) for the determination of non uniform spatial distributions of electric field and dipole polarization within insulating dielectric films /5/.

Pyroelectric measurements can be performed in the time or frequency-domain. Time-domain measurements are not only more intuitive, but also fast and thus allow for in-situ measurements of material characteristics i.e. during thermal processing steps.

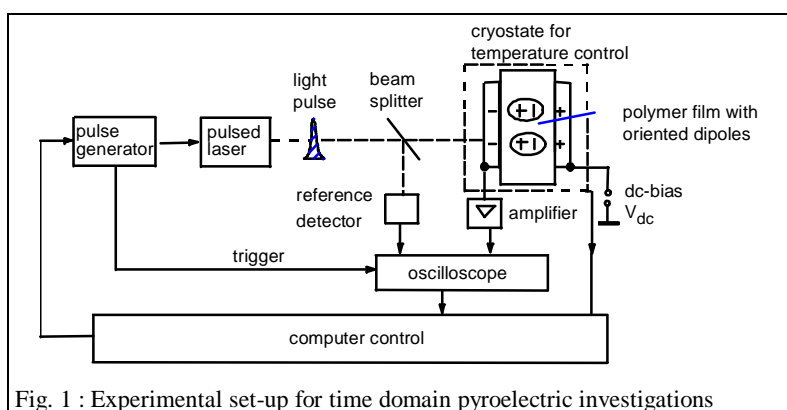


Fig. 1 : Experimental set-up for time domain pyroelectric investigations

Time-Domain Pyroelectric Measurement Technique

Pyroelectricity is the electrical response of a material to a change in temperature. It can be employed in any dielectric containing

- i) spontaneous or
- ii) frozen polarization from oriented dipoles or
- iii) induced polarization in the presence of electric fields /1/.

In the time-domain, a pulsed laser delivers a short light pulse that is absorbed within the opaque metal electrode on top of the polymer film. The pyroelectric signal is amplified and recorded by

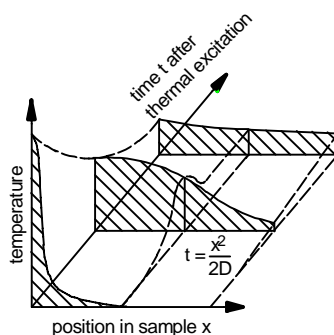


Fig. 2 : Evolution of the temperature distribution in a free-standing polymer film after thermal-pulse excitation

a digital storage oscilloscope. A schematic view of the computer-controllable experimental set-up, including a reference detector for monitoring the pulse energy and a cryostat for temperature control of the sample is shown in Fig. 1 /6/.

Immediately after thermal excitation of the polymer sample, only a layer near to the surface is heated (Fig. 2). In free-standing films, bending piezoelectric signals are observed at short time scales, enabling the determination of pyro- and piezoelectric, as well as elastic film properties /2,3,6/. In films rigidly mounted on substrates, piezoelectric contributions are suppressed. In this case, the polarization is changed only in the heated layer, and thus the generated pyroelectric signal solely contains information on the polarization of this layer. With increasing time after the light pulse, heat diffuses through the sample (Fig. 2), so that more and more of the polarization in the film volume contributes to the signal. Without going into details, a reasonable approximation of the polarization distribution $P(x)$ is obtained by scaling the data

according to $x = \sqrt{2Dt}$, where x is the position in the sample, D its thermal diffusivity and t the time after the light pulse /6/.

Within the same experimental set-up, the thermal pulse response can be recorded at times where heat starts to diffuse in the substrate, therefore enabling the determination of the thermal diffusivity of the dielectric, for example as a function of temperature, during chemical reactions etc. /4/.

Polarization Distribution in Ferroelectric Polymers

From a practical point of view (for example in fast radiation sensors), the knowledge of the polarization distribution within polymer films after various poling procedures is required. During the application of a poling field, injected, internally generated or pre-existing space charges may cause spatially and temporally varying electric fields, leading to polarization distributions within the poled polymer samples. Fig. 3 shows the polarization distribution of a 52 μm thick poly(vinylidene fluoride) (PVDF) film (kindly provided by AMP) /6/. Although the polarization is nearly uniform throughout the bulk of the film, depolarized layers are observed in the vicinity of the electrodes.

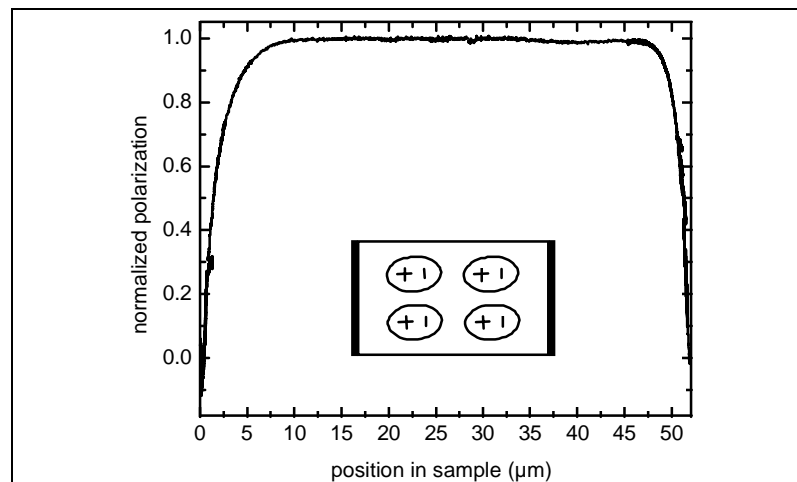


Fig. 3 : Polarization distribution of a well-poled 52 μm thick ferroelectric poly(vinylidene fluoride) (PVDF) film. Depolarized layers near the electrodes are typical after poling, demonstrating the role of injected space charges during poling

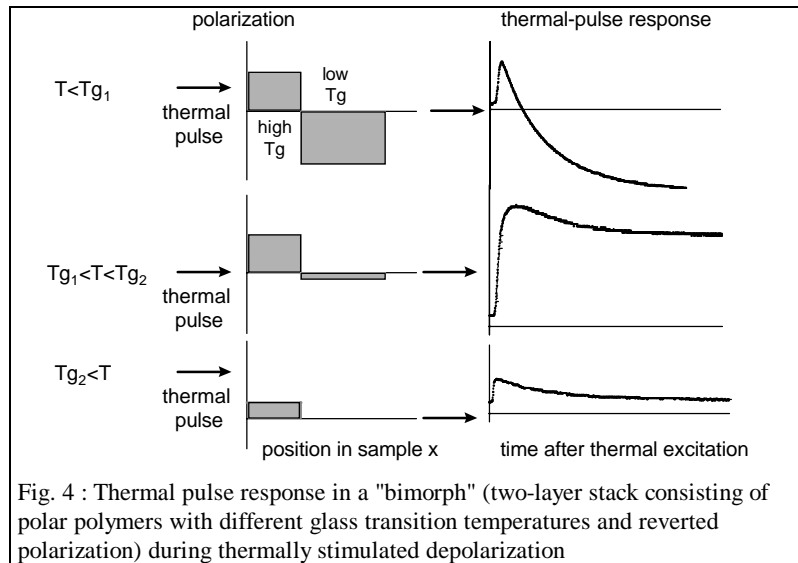


Fig. 4 : Thermal pulse response in a "bimorph" (two-layer stack consisting of polar polymers with different glass transition temperatures and reverted polarization) during thermally stimulated depolarization

Polarization Distribution during Thermally Stimulated Depolarization (TSD)

Current signals obtained by TSD may be difficult to analyze when the poled sample shows non uniform electric field or dipole polarization distributions (as often reported for polymer films). Time-domain pyroelectric measurements allow the determination of the electric field or polarization distribution during the TSD run, and may thus provide for a better interpretation of TSD results.

As an illustration, Fig. 4 shows the relaxation of the dipole polarization in a stack of two polar

polymers with different glass-transition temperatures (T_g 's) and polarization-reverted dipole polarization. Such „bimorph“ structures are required for bending piezoelectric devices, as well as for phase-matched frequency conversion in optical waveguides /7/ and can be prepared by an appropriate two-step poling process /8/.

At temperatures below T_g , the pyroelectric signal changes sign at a thicknesses corresponding approximately to that of the polymer layer near the thermally excited electrode, demonstrating the preparation of a „bimorph“ (Fig. 4a). The dipole polarization in amorphous polymers with frozen-in dipole polarization relaxes in the vicinity of the glass transition temperature. Between the two T_g 's, the polarization is relaxed in the low- T_g polymer (and thus no change of sign can be observed in the response depicted in Fig. 4b). Finally, the dipole orientation vanishes at temperatures above the higher glass transition temperature (Fig. 4c).

As the technique is most suitable on films with thicknesses in the range of one μm or below, a broad new field of research activities can be opened to time domain pyroelectric investigations.

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Friedrich Kremer

The Search for Low-Dielectric Constant Materials

The continuing improvement of microprocessor performance is to a large extent the result of a reduction in the device size. This enables an increase in the device packing density and the processing speed. But – as dimensions shrink to less than 0.25 μm (transistor gate length) – propagation delay, cross talk noise and power dissipation due to resistance-capacitance (RC) coupling become significant. The smaller line dimensions increase the resistivity R of the metal lines, and the narrower interline spacing increases the capacitance C between them. Thus while the speed of the devices increases as its dimensions decrease, the interconnect delay becomes a dominating fraction of the total response hence limiting further improvement in device performance. This development has led to an intensive search for low dielectric constant materials [1-4].

In order to reduce the RC response it is required to implement high conductive metals (such as Cu) and to use low dielectric constant materials. The replacement of Al by Cu will reduce the RC delay by approximately 35%, the substitution of SiO_2 ($k = 4$) with air would result in a reduction of about 75%. This shows how strong the impact of the dielectric materials is for the device performance.

Several developments are in

progress:

- Vapour deposition of parylene and polynaphthalene and polytetrafluoroethylene (PTFE) ($\epsilon' = 1.8 - 3.0$ for fluorinated polymers and $\epsilon' = 2.7 - 3.5$ for non-fluorinated systems) [5]

- Plasma-enhanced chemical vapour deposition and high-density plasma chemical vapour deposition of fluorinated SiO_2 glass ($\epsilon' = 3.5$) [6], amorphous C:F ($\epsilon' > 2.7$) [7] and air-gap formation [8]

- plasma polymerisation of pentafluorostyrene ($\epsilon' = 2.2 - 2.4$) [9] and pulse plasma polymerisation of PTFE ($\epsilon' = 2.0$) [10].

Materials that can be deposited by spin-on casting include:

- organic polymers: $\epsilon' = 1.8 - 3.0$ for fluorinated polymers and $\epsilon' = 2.5 - 3.0$ for non-fluorinated systems [11]

- inorganic polymers: $\epsilon' = 2.7 - 3.5$ for nonporous systems [11]

- inorganic-organic hybrids: $\epsilon' = 2.8 - 3.0$ [12]

- porous materials as xerogels and aerogel with $\epsilon' = 1.8 - 2.8$ [13].

Of course the dielectric constant is not the only feature that has to be optimised. There are other stringent requirements on the chemical, mechanical and the thermal properties as well (s. Tab. I [14]).

Of special importance is the thermal conductivity of low dielectric constant materials. There are three causes of interconnect temperature rise: Joule heating, heat dissipation from the substrate and heat dissipation from nearby interconnect lines. Thus the packing design becomes decisive for an effective heat transfer. On the other hand, the low thermal

Electrical	Chemical	Mechanical	Thermal
k , Anisotropy	Chemical Resistance	Thickness uniformity	High thermal stability
Low dissipation	Etch selectivity	Good adhesion	Low coefficient of thermal expansion
Low leakage current	Low moisture absorption	Low stress	Low thermal shrinkage
Low charge trapping	Low solubility in H_2O	High hardness	Low thermal weight loss
High electric-field strength	Low gas permeability	Low shrinkage	High thermal conductivity
High reliability	High purity	Crack resistance	
	No metal corrosion	High tensile modulus	
	Long storage life		
	Environmentally safe		

Table I: Property Requirements of Low-Dielectric-Constant Interlayer-Dielectric Materials.

conductivity of low dielectric-constant materials does not significantly raise the interconnect temperature if an embedded insertion scheme is used [15].

In summary the integration of low dielectric constant materials requires an optimisation between material properties, device architectures and the manufacturing process. Since the lowest dielectric constant for any dense materials is approximately 2.0 for (PTFE), the evaluation of porous materials will eventually be required to push performance to its fundamental limits [8].

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Gerhard Schaumburg

Novocontrol Provides Application Notes on Dielectric Spectroscopy

From now on, a series of application notes related to the basics of dielectric spectroscopy and its application to several classes of materials will be issued by Novocontrol. The application notes are written by scientists from the point of view of their actual working area. For each special area, a detailed overview on the scientific methods and techniques is given both from the experimental and theoretical point of view.

Application notes maybe requested from Novocontrol by interested readers. In addition, a limited number of application notes will be available on conferences from Novocontrol representatives.

Until now, two application notes are available. The object of this article is to give a short summary of each one. In addition, two further application notes are in preparation, but have not yet been completed. These will be reviewed in the next Dielectrics Newsletter and available from Novocontrol shortly.

Dielectric Spectroscopy on the Dynamics of Amorphous Polymeric Systems

by A. Schönhals, Bundesanstalt für Materialforschung und -prüfung, Fachgruppe VI 3.2, Unter den Eichen 87, 12205 Berlin, Germany

This application note gives an overview on the actual concepts of dielectric spectroscopy used for the characterization of amorphous polymers.

The introduction starts with a general overview on synthetic

polymers as complex systems build up by a large number of atoms. This results in a large number of possible molecular conformations which govern the physical properties of the materials. Since Debye, dielectric spectroscopy has proven very useful for studying the conformation, structure and the dynamics of polymer systems.

It is pointed out, that recently there is an increasing interest for dielectric studies of polymeric materials. This is partly because due to the availability of a broad frequency range instrumentation from mHz to GHz in combination with automated experiment control. In addition, there has been considerable progress both in theory and modelling. Besides from fundamental investigations, dielectric spectroscopy provides information of direct technical impact on

-electric insulation properties of amorphous polymers e.g. used for cable insulation or passive layers in microchips,

-microwave adsorption characteristics for polymers used in telecommunication or radar applications as absorbers, reflectors or transmitters including microwave heating characteristics of polymers in microwave cookers.

The next chapter gives a summary of the theoretical considerations of the dielectric mechanisms in polymers. The complex dielectric function $\epsilon^*(\omega) = \epsilon' - i\epsilon''$ is defined, and a physical interpretation for ϵ' and ϵ'' and its relation to the complex conductivity $\sigma^*(\omega)$ is provided. $\epsilon^*(\omega)$ is derived from the dipole polarization correlation function $\Phi(t)$. The main polarization mechanisms for long chain molecule polymers of type A, B, C and for low molecular weight polymers are discussed. This results in different modes of polymer dynamics which are separated by relaxation times and rates. The polymer dynamics is

closely related to the viscoelastic properties and the glass transition.

As an example, measurements of poly(propylene glycol) are presented covering a frequency range of 14 decades.

As next, the basic principles of dielectric experiments like frequency domain measurement, time domain measurement, their conversion by Fourier Transform and the correlation of $\epsilon'(\omega)$ and $\epsilon''(\omega)$ by the Kramers-Kronig relations are provided. The theoretical chapter closes with the formula of the Onsager, Fröhlich and Kirkwood theory which calculates the dielectric relaxation strength in dependence of the molecular dipole moment.

Chapter three deals with the evaluation of dielectric spectra. The basics of non linear curve fitting and the most often used model functions in the frequency domain like Cole-Cole, Cole Davidson, Fuoss-Kirkwood and Havriliak Negami are introduced. Fitting in the time domain can be done with the Kohlrausch-Williams-Watts-function or by Fourier transformed frequency domain model functions. The chapter closes with the representation of dielectric spectra in terms of the relaxation time distribution describing the spectra as a linear superposition of Debye relaxations.

The last chapter four is specialized on the dielectric relaxations in dense amorphous polymers. The three α , β , and α' (or γ) relaxation types are discussed. The β relaxation is located at higher frequencies than the α and α' . The dependence of the relaxation time follows a simple Arrhenius law. According to most authors, the β relaxation arises from localized rotational fluctuations of side groups or fluctuations of localized parts of the main chain.

The α relaxation is located at frequencies below the β and α' relaxation. The temperature dependence of the relaxation time

follows a Vogel Fulcher (VFT) or Williams Landel Ferry law. The α relaxation is related to the thermal glass transition which is an unsolved problem of condensed matter physics until now. The relaxation is caused by the microbrownian segmental motions of the chain. The relaxation mechanisms are discussed more detailed and model calculations are quoted.

Finally, for type A polymers having a dipole moment parallel to the chain contour the α' or normal mode relaxation at the low frequency end can be observed. It is due to the molecular motion of the of the whole polymeric chain. Theoretical considerations are discussed for non entangled polymers with low molecular weight in terms of the Rouse theory and for high molecular mass entangled polymers in the frame of the reptation or tube model. In both cases, the temperature dependence of the relaxation time follows a VFT law.

Dielectric Spectroscopy of Reactive Polymers

by Jovan Mijovic and Benjamin D. Fitz, Department of Chemical Engineering, Chemistry and Materials Science, Polytechnic University, Six Metrotech Center, Brooklyn, NY 11201, jmijovic@poly.edu, bfitz01@utopia.poly.edu

This application note is reporting about monitoring of materials changing their physical and chemical parameters with time by broadband dielectric spectroscopy (BDS). The focus is on monitoring of cure.

The introduction points out that there is a current surge of interest both in fundamental and applied aspects of BDS. Fundamental investigations of the dielectric response yield a wealth of information about different molecular motions and relaxation processes. As a key feature of BDS, its wide bandwidth relates dielectric response to slow and fast molecular

events from pico seconds to weeks.

The strong industrial interest in dielectric and electric properties of polymers is reflected by their usage in electronic interconnect devices, optoelectronic switches, printed circuit boards, microwave assemblies for radar, batteries, fuel cells and so on.

More recently there are systematic studies by BDS of systems characterized by temporal evolution of structure. Examples are systems that undergo a chemical and/or physical change as a result of chemical reaction, crystallization, vitrification, phase separation, etc. In the following, attention is focused on such studies and especially on BDS for characterization of cure. Compared to other techniques, the dielectric in situ online monitoring has several advantages. Samples must not be measured off line. The continuous measurement is related to the main processing parameters degree of cure and viscosity. The technique is well suited for online process control.

Chapter three describes some aspects of the origin of dielectric response in polymers. General different polarization mechanism are electronic polarization in the UV an visible range, atomic polarization in molecules with inhomogenous electron distribution in the optical infrared range, polarization due to charge migration and dipole orientation in the microwave and low frequency range. Charge migration and dipole orientation are accessible by BDS.

Charge migration is separated and discussed as extrinsic charge migration e.g. due to impurities inversely correlated with viscosity, and intrinsic charge migration e.g. due to proton transfer along hydrogen bonds.

Chapter four is focussing on experimental aspects. The principle of impedance measurement and some equivalent representations in terms of admittance or dielectric function are introduced. The most

commonly used electronic equipment is listed. Novocontrol is quoted as the leading company that offers complete setups for BDS including instruments, high precision heating/cooling control, various accessories, and a wide range of software. The next paragraph describes the sample cells and sensors. As an addition to the widely known parallel plate electrodes, interdigit comb electrodes (both as passive sensors and with active electronics on the sensor) and microdielectrometer sensors are mentioned and quoted.

The experimental chapter closes with some additional aspects of measured results. These are electrode polarization due to accumulation of ions at the polymer-electrode interface, interfacial, space charge or Maxwell-Wagner-Sillars polarization due to charge layers at the interfaces of components in heterogeneous systems.

Chapter five is reporting about polarization due to migrating charges. It starts with equivalent circuits modelling concepts. The model function is the impedance (or dielectric function) of a circuit often build up by a combination of resistors, capacitors and others. The circuit impedance is fitted to the measured data with the values of the circuit components as fitting parameters.

The most important circuits for modelling polarization by charge migration, Debye relaxation, approximations for distributions of relaxation times and electrode blocking layers are discussed.

The next section focusses on the characterization of cure by charge migration. The conductivity is evaluated in a proper frequency window and calculated from maximum in the $Z''(\omega)$ curves. Examples show the conductivity versus cure time for a trifunctional epoxy triglycidylether of paraaminophenol (TGEPA) and a tetrafunctional amine methylenedianiline (MDA) formulation.

The extend of cure (or reaction) is determined by phenomenological equations. From this, in addition the viscosity during the reaction is calculated.

Chapter six is about the effects of cure to the polarization by dipole orientation. The different kinds of relaxation depending on length scale of molecular motion are summarized. At high frequencies, the broad secondary β relaxation appears. It has Arrhenius temperature dependence, low intensity, low activation energy and is also present below the glass cavities.

The primary α relaxation at lower frequencies is associated to the glass transition. It is caused by co-operative segmental motions and shows non Arrhenius temperature dependence. The α process merges with the β process at higher temperatures.

After this more general aspects, the dependence of the dielectric parameters on cure are examined in detail. The dipolar relaxation time as a fundamental material parameter is an effective tool for the study of reactive systems. An example shows a typical epoxy/amine formulation undergoing an isothermal cure. A general concept for characterization of time and temperature dependent curing processes based on reference measurements of the base materials in combination with additional information from e.g. DSC, FTIR, titration, GPC is suggested.

Another important parameter is relaxation strength. The materials dipole composition changes in systematic manner during cure. If reactants and/or products contain some polar portions, the dipole composition will be directly related to static permittivity ϵ_0 . A qualitative relation is given in terms of the Debye theory later modified by Onsager and Fröhlich. Usually, a decrease of permittivity is observed during the cure. This is because mostly the reaction product dipoles are less polar or non polar

entities. Details of frequency dependence and underlying molecular processes are explored in a series of illustrations.

In industrial applications, the relaxation strength is often monitored in the microwave range, where the main polarization effects are due to dipole relaxation. Measurements are mostly done at a single frequency as a function of time. The types of used probes are flat surface strip-lines, coaxial airlines filled with the sample, open-ended semi-rigid coaxial lines terminated by the sample, and resonant cavities.

A third approach to the analysis of cure data is to examine changes in the shape parameters of relaxation modelling functions. The shape parameters generally describe the width or slope of the relaxation peak. In the frequency domain, the Havriliak-Negami function, the Jonscher two power law, and in the time domain the Kohlrausch-Williams-Watts function are discussed. In general, after the separation of the α and β relaxation peaks in early cure, the α peak tends to broaden as cross linking develops.

Phenomenological and Molecular Theories of Dielectric and Electric Relaxation of Materials

by G. Williams and D. K. Thomas, Department of Chemistry, University of Wales Swansea, Singleton Park, Swansea SA2 8PP, UK.

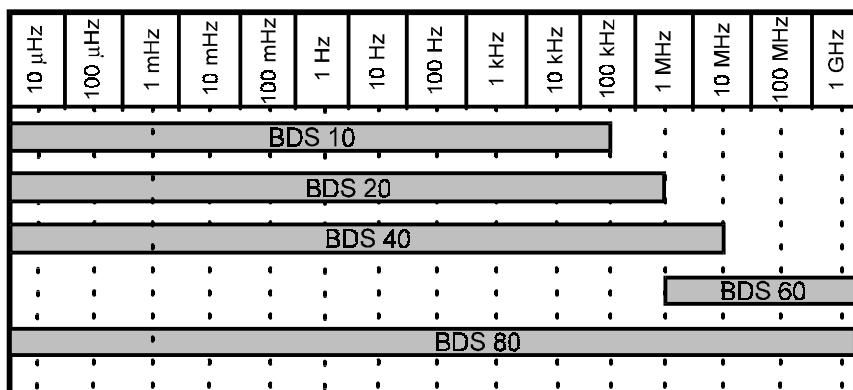
Dielectric Spectroscopy on the Molecular and Collective Dynamics in Liquid Crystals

by F. Kremer, Universität Leipzig, Linnéstr.5, D-04103 Leipzig, Germany

These two articles are in preparation and will be reviewed in the next DNL issue.

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