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"Conduction-Free" Dielectric Loss ∂ε/∂ln*f* – a Powerful Tool for the Analysis of Strong (Ion) Conducting Dielectric Materials

introduction

Although dielectric spectroscopy generally yields *complex* dielectric relaxation spectra $\varepsilon^*(\omega) = \varepsilon'(\omega) - i\varepsilon''(\omega)$, the data analysis and discussion of the dielectric properties is mainly based on the dielectric loss spectra $\varepsilon''(\omega)$ rather than on the real part spectra $\varepsilon'(\omega)$. Two reasons for this practise are:

1) Dielectric dispersion curves show often more details and are therefore more suitable for visual inspection and evaluation of complicated dielectric data (e.g. superimposed relaxation peaks) than $\varepsilon'(\omega)$.

2) Since ε' and ε'' are interrelated by the Kramers-Kronig relations, one of which is Eq. 1, the two spectra $\varepsilon'(\omega)$ and $\varepsilon''(\omega)$ are fully equivalent with respect to their information about relaxation processes. Consequently, each of the two components of ε^* contains the full information of the dielectric behaviour.

Strong ionic conduction, which usually shows up above the glass-transition temperature T_g, often obscures loss peaks of dipolar origin. In view of the increasing influence of the Ohmic loss contribution at low frequencies according to $\varepsilon''_{cond} = \sigma/\varepsilon_0\omega$ (σ= conductivity, ε_0= vacuum permittivity, $\omega = 2\pi f$ = angular frequency), the conduction should be eliminated in order to elucidate low frequency relaxation processes.

An elegant way to eliminate Ohmic (i.e. frequency independent) conduction from the measured loss

$$\varepsilon''(\omega_0) = \frac{\sigma_{dc}}{\varepsilon_0\omega_0} + \frac{2}{\pi}\int_0^\infty \varepsilon'(\omega_0)\frac{\omega_0}{\omega^2 - \omega_0^2}d\omega \quad (1)$$

spectra uses the Kramers-Kronig relation, which transforms the real part $\varepsilon'(\omega)$ into the imaginary part $\varepsilon''_{KK}(\omega)$. This quantity $\varepsilon''_{KK}(f)$ (2nd term in Eq. 1) lacks by definition the Ohmic conduction contribution and can be calculated by numerical techniques developed by van *Turnhout* and *Steeman* in [1, 2].
An even more convenient technique is based on the derivative

$$\varepsilon''_{deriv} = -\frac{\pi}{2}\frac{\partial\varepsilon'(\omega)}{\partial\ln\omega} \approx \varepsilon'' \quad (2)$$

which yields approximately the dielectric loss for the case of broad peaks like those of the α-transition or the secondary relaxations [1]. In particular, for narrow (Debye-like) relaxations, the derivative results in

peaks which are markedly *narrower* than the measured loss peaks according to Eq. 3:

$$-\frac{\partial\varepsilon'(\omega)}{\partial\ln\omega} \propto (\varepsilon'')^2 \quad (3)$$

For the double stretched Havriliak-Negami function (Eq. 4) we have given the analytical expression for $\partial\varepsilon'_{HN}/\partial\ln\omega$ (Eq. 5). Due to this peak sharpening which is demonstrated in Fig. 1, overlapping peaks can be resolved in a significantly better way.

Although there are several numerical procedures for the implementation of Eq. 2 in order to process experimental data, one should realise that derivatives are sensitive to inaccuracies, i.e. noise,
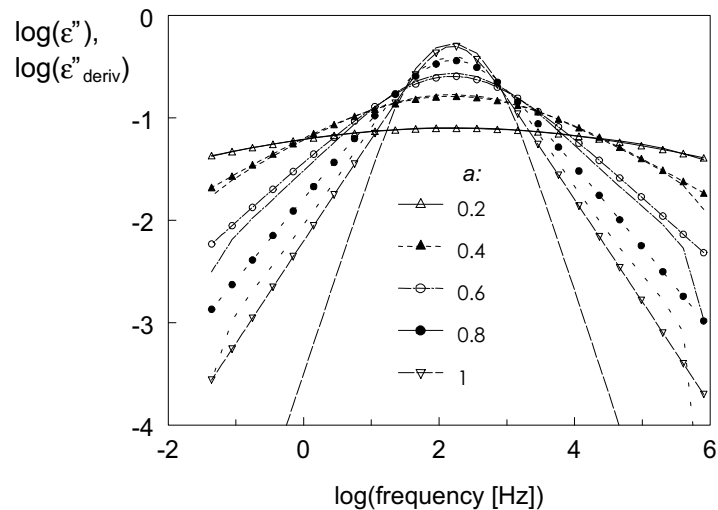

Fig. 1. Comparison of the dielectric loss ε'' (lines with symbols) with ε''_{deriv} (lines) of a symmetric (*b*=1) Havriliak-Negami (HN) function (Eq. 4) for various shape parameters *a* (0.2, 0.4, 0.6, 0.8, 1).

$$\varepsilon'' = \text{Im}\left\{\frac{\Delta\varepsilon}{\left[1 + (i\omega\tau_k)^a\right]^b}\right\} \quad (4)$$

$$\frac{\partial\varepsilon'_{HN}}{\partial\ln\omega} = -\frac{ab\Delta\varepsilon(\omega\tau)^a\cos\left[a\pi/2 - (1+b)\theta_{HN}\right]}{\left[1 + 2(\omega\tau)^a\cos(\pi a/2) + (\omega\tau)^{2a}\right]^{\frac{1+b}{2}}} \quad (5)$$

$$\theta_{HN} = \arctan\left[\sin(\pi a/2)/\left((\omega t)^{-a} + \cos(\pi a/2)\right)\right]$$

which is always present in real dielectric data $\varepsilon'(\omega)$. Therefore, we have used a numerical technique based on the least squares convolution algorithm introduced by *Savitzky* and *Golay* [3], see also *Gorry* [4]. This procedure requires ε' data which are equally spaced on the $\log(\omega)$ scale, usually in a geometric series (ω, 2ω, 4ω...). More details can be found in Refs.[5, 6].

analysis of electrode polarisation

Electrode polarisation (EP) is one of the most undesired effects in DRS which hampers the proper analysis of slow relaxation processes in moderately to highly conducting liquids including polymer melts. The molecular reason for EP is the (partial) blocking of charges at the sample/electrode interface which results in the formation of electrical double layers. These double layers give rise a large capacitance in series to the conducting (bulk) sample material, which manifests itself in high apparent dielectric constants typically in the range of 10^2 to 10^6.

Since electrode polarisation involves charging/discharging of the double layer capacitance, the effective thickness of which is given by the Debye lenght L_D, both its strength and relaxation time scale linearly with the sample thickness roughly to

$$\Delta\varepsilon_{EP} \approx \varepsilon' L / 2 L_D \quad (6)$$

and

$$\tau_{EP} \approx \frac{\varepsilon'\varepsilon_0}{\sigma}\frac{L}{2L_D}. \quad (7)$$

Regarding ε''_{EP}, this scaling behaviour results in a high frequency slope of the EP loss peak of -1 which is independent of the sample thickness L. This is illustrated in Fig. 2 (top).

In contrast, the quantity $\varepsilon''_{deriv}(\omega)$ given in Fig. 2 (below) does not join at the high frequency side due to its $\varepsilon'' \propto \omega^{-2}$ behaviour. This results in an effective shift of the interfering EP peak towards lower frequencies upon increase of L. Consequently, the use of $\varepsilon''_{deriv}(\omega)$ gives us the possibility to separate hidden relaxation processes by optimizing the sample thickness.

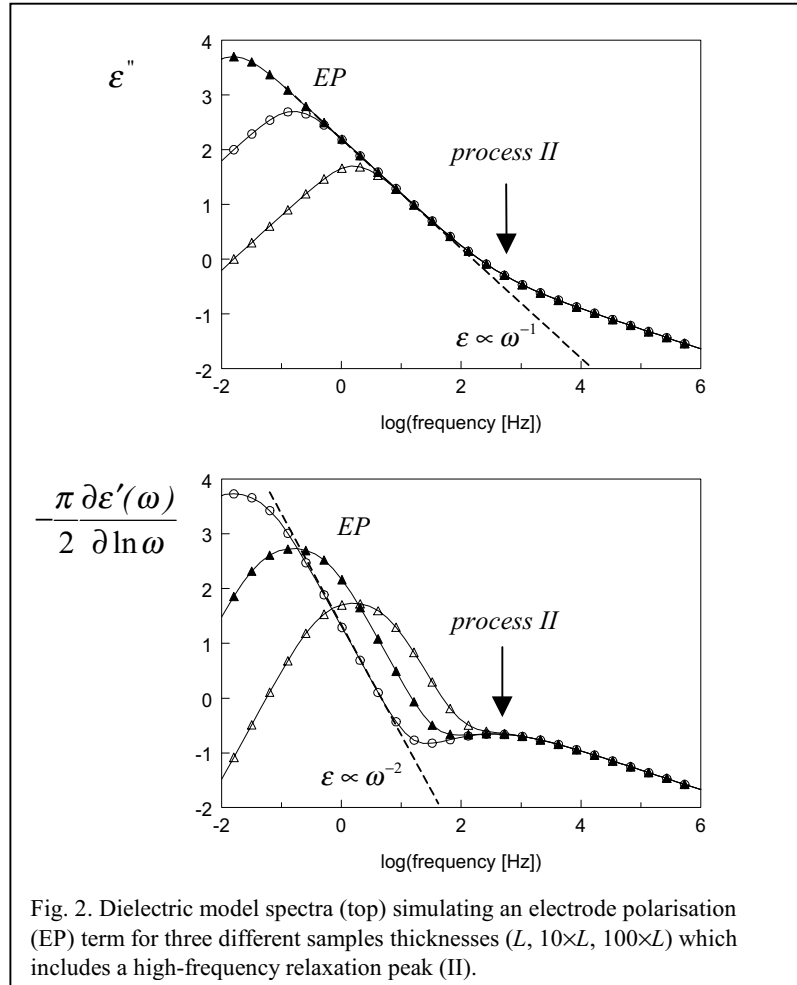## analysis of complex relaxation spectra

Fig. 2. Dielectric model spectra (top) simulating an electrode polarisation (EP) term for three different samples thicknesses (L, $10\times L$, $100\times L$) which includes a high-frequency relaxation peak (II).

Apart from the beneficial use of ε''_{deriv} in order to handle strong electrode polarisation, the derivative technique also turns out to be very useful for polymeric dielectrics which show low-frequency relaxations (compared to the main (α) relaxation) as well as a moderate to high Ohmic conductivity. This is often the case for side-chain liquid crystalline polymers [7], an example is given in Fig. 3.

In the loss spectrum $\varepsilon''(\omega, T)$ shown in this figure, we can identify two relaxation processes above T_g, the α-process related to the dynamic glass transition of the polyurethane backbone and the fastest relaxation mode λ_1 of the nitrostilbene mesogene.

In contrast to the $\varepsilon''(\omega, T)$ curves, the $\varepsilon''_{deriv}(\omega, T)$ spectra clearly reveal the existence of two additional relaxation processes on the low-frequency side of the λ_1 process. A direct comparison between both quantities is made in Fig. 4, which shows a clear peak-sharpening around the λ_1-relaxation, indicating that this relaxation has a Debye-like shape. Figs. 1 and 4 also

show the practical limitations of the derivative approach: 1) slight systematic inaccuracies of the numerical algorithm at the edges of the frequency spectra (in particular visible in Fig. 1), deviations at high frequencies due to experimental phase inaccuracies (Fig. 4) as well as increasing noise in ε''_{deriv} at low frequencies caused by errors in the $\varepsilon'(\omega)$ data. One should note that actually the accuracy in $\varepsilon'(\omega)$ determines the minimum detectable ratio between the desired (*dipolar*) losses and the *total* loss $\varepsilon''(\omega) \sim \varepsilon''_{cond}$, which is typically $10^{-2} - 10^{-3}$.

conclusions

An alternative loss quantity, ε''_{deriv} has been described which combines the advantage of ε' not containing Ohmic conduction, with the vivid display of dispersion curves. For broad relaxations, ε''_{deriv} is a fair approximation for the Kramers-Kronig transformation, while for narrow (Debye-like) relaxations sharper peaks are obtained. The latter feature provides improved separability of superimposed
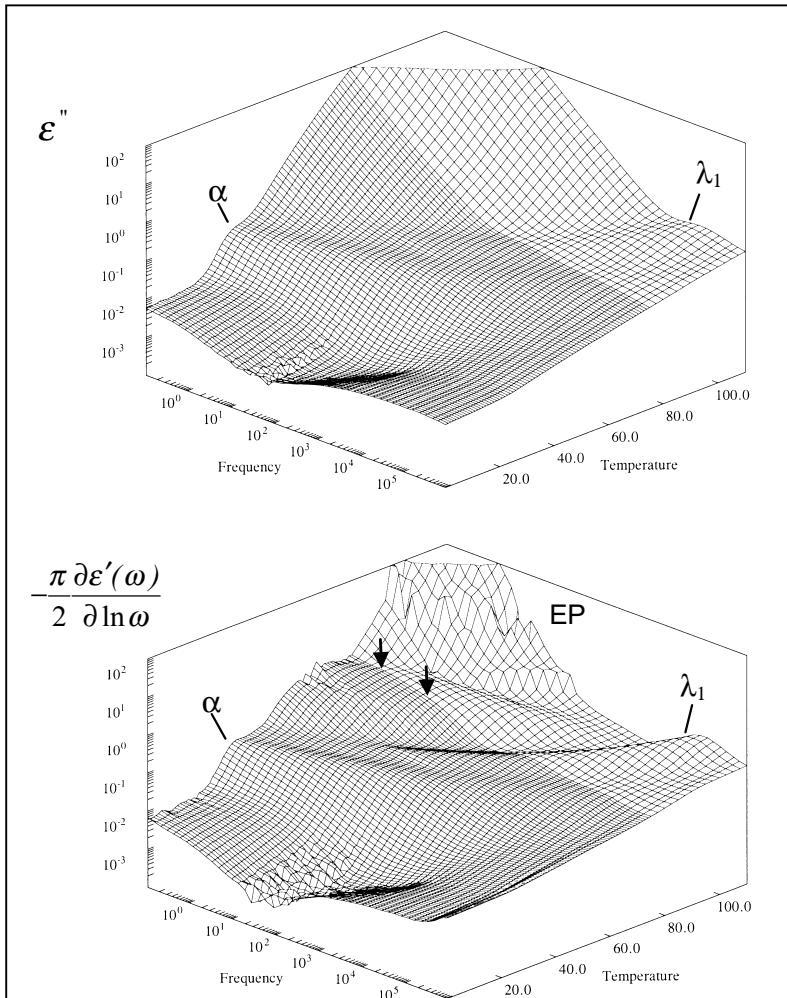
$$\varepsilon''$$

$$-\frac{\pi}{2}\frac{\partial\varepsilon'(\omega)}{\partial\ln\omega}$$

Fig. 3. Dielectric loss ε'' (left) and ε''_{deriv} (right) of a side-chain liquid crystalline polyurethane measured during heating. The derivative technique reveals two additional slow relaxation processes (black arrows) which could be sufficiently separated from the electrode polarisation by using an extra thick sample ($L = 500\mu$m).
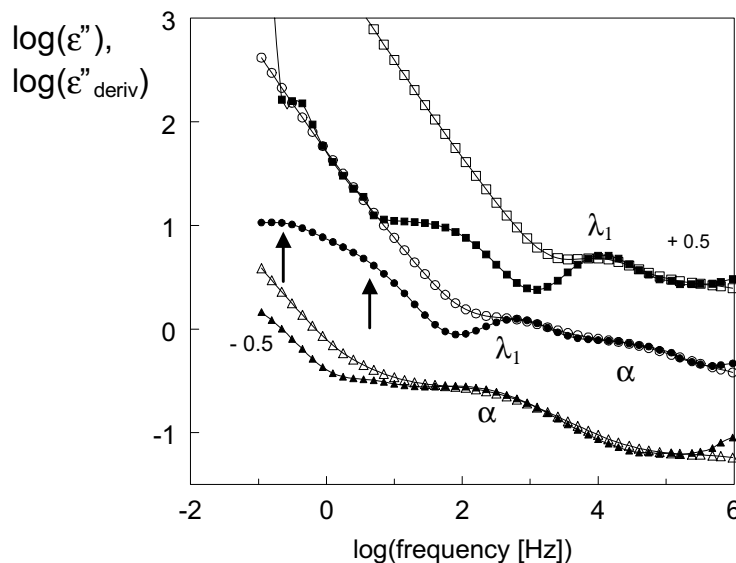

$$\log(\varepsilon''),\ \log(\varepsilon''_{deriv})$$

Fig. 4. Dielectric loss ε'' (open symbols) and ε''_{deriv} (filled symbols) spectra at three different temperatures selected from Fig. 3. For clarity, two curves are vertically shifted by $\log(\varepsilon'') = 0.5$.

relaxation processes and makes the derivative technique a strong tool to tackle electrode polarisation..

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Molecular Dynamics in Grafted Polymers

While the dynamics of polymers in the bulk has been studied extensively by use of different methods, among them dielectric spectroscopy, there are only a few corresponding studies of polymers within a spatial confinement, i.e. at least one dimension of the space containing the polymer sample is drastically reduced compared to the bulk. The goal of such studies is to answer the question of the existence of finite size effects which, for instance, can be expected according to the theory of the glass transition of Adam and Gibbs.

A convenient way to confine polymers is to prepare them as (ultra-) thin films since the preparation (spin coating, the Langmuir-Blodgett- or grafting techniques) is well established and the film thickness as confining size can be easily adjusted. Up to now,

in case of dielectric spectroscopy the problem of the application of electrodes to the polymer layer has been solved by evaporation of metal electrodes on the (ultra-) thin film. Recently, a novel method was described which enables to contact (ultra-) thin (d<50 nm) organic layers without evaporation of metal electrodes [1]. This novel approach was successfully employed to study - for the first time - the molecular dynamics of grafted poly(γ-benzyl-L-glutamate) (PBLG) in the swollen and in the dried state [2] by means of dielectric spectroscopy.

In dielectric measurements on bulk PBLG, two dielectrically active relaxation processes have been found according to its structure (Fig. 1), which are assigned to the (restricted) fluctuation of the helical main chains as a whole (chop stick motion) and to the fluctuation of the polar side groups. While bulk samples have been prepared conventionally as solution cast films, grafted layers have been obtained as result of a surface induced polymerisation (as described in ref. [3]) on aluminium stripes evaporated on glass substrates.

After determination of the thickness of the grafted layers by means of ellipsometry, the upper electrode has been prepared either by evaporation of aluminium on the layer or by application of a solid electrode prepared as described in [1]. The dielectric measurements have been performed using a frequency response analyzer and a Novocontrol broadband dielectric converter with active sample cell (BDC-S). Effects due to a non-linear response of the sample were excluded by setting the generator voltage to 0.4 V. Thus, electrical fields of typically 10^5 V cm^{-1} for sample thicknesses in the range from 20 nm to 60 nm were applied. The temperature has been varied
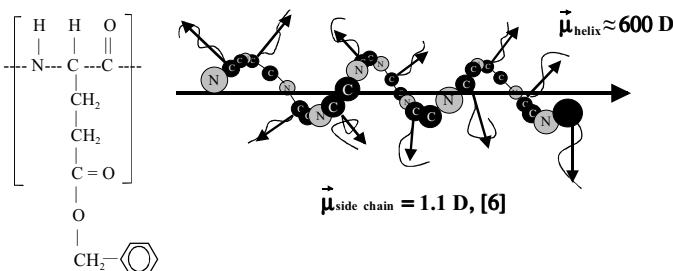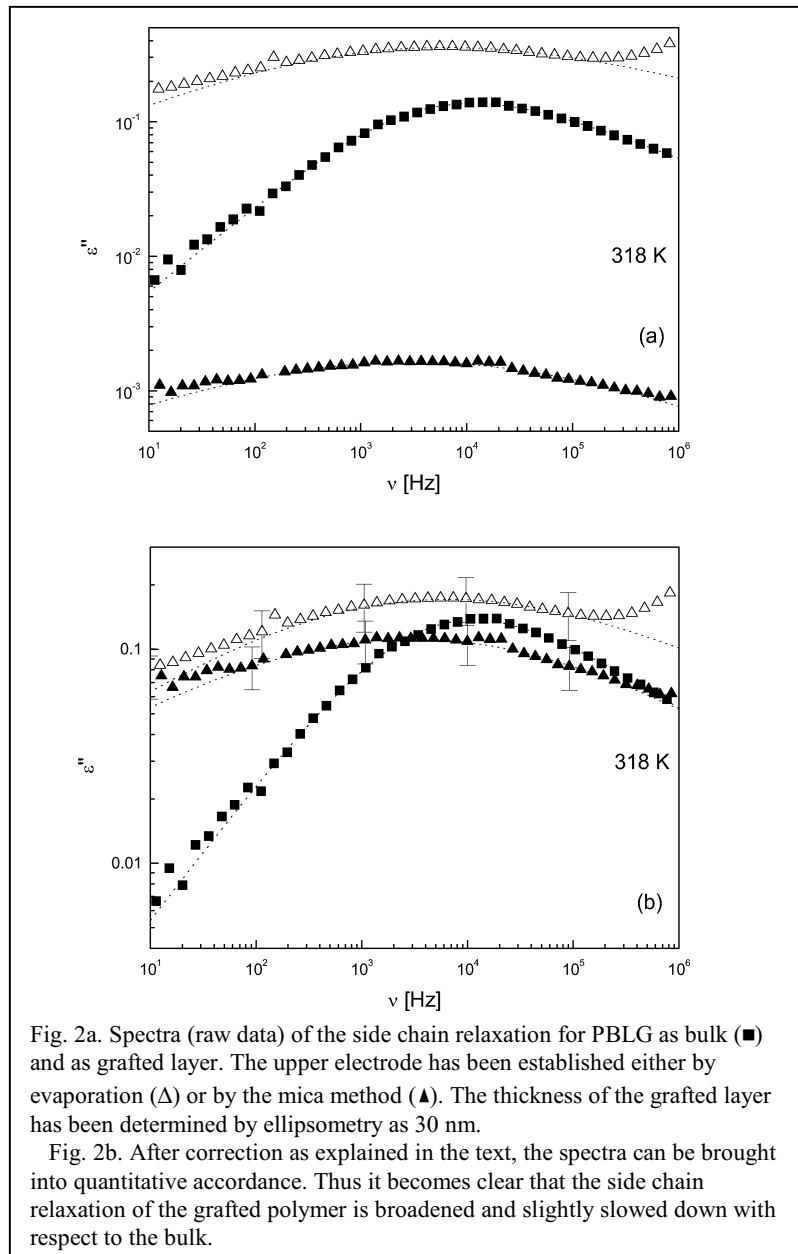

Fig. 2a. Spectra (raw data) of the side chain relaxation for PBLG as bulk (■) and as grafted layer. The upper electrode has been established either by evaporation (Δ) or by the mica method (▲). The thickness of the grafted layer has been determined by ellipsometry as 30 nm.

Fig. 2b. After correction as explained in the text, the spectra can be brought into quantitative accordance. Thus it becomes clear that the side chain relaxation of the grafted polymer is broadened and slightly slowed down with respect to the bulk.

between 450 K and 270 K.

For the analysis of dielectric measurements, the imaginary part ε'' of the dielectric function is fitted using a superposition of a conductivity contribution and a generalized relaxation function according to Havriliak-Negami in order to obtain the relaxation rate $1/\tau_{max}$, which is given by the frequency of the maximum dielectric loss for a certain temperature. The activation behaviour of the side chain fluctuation for the different samples could be described by the Vogel-Fulcher-Tammann law:

$$\tau = \tau_0 e^{-\frac{DT_0}{T-T_0}} \quad (1)$$

Here, D is a measure for the fragility of the glassforming substance, T_0 is the Vogel-temperature and τ_0 denotes the limit of the relaxation time at high temperatures. In all samples (bulk and grafted layers) a rise in the dielectric loss ε'' for frequencies higher than 10^6 Hz is observed. This artefact is due to the electrode resistance R which leads to an

Fig. 1. Primary and secondary structure of poly(γ-benzyl-L-glutamate) (PBLG).

parallel *RC* circuit and therefore to an apparent loss process at high frequencies [4].

It stands to reason that one can not assume to have good contact between the upper (stiff) mica-electrode and the surface of the grafted layer. Additionally, because of its roughness the real surface is larger than that value obtained by multiplying the width of the electrodes. One has therefore to expect distinct differences between the measured capacity and the calculated value according to the geometry of the sample capacitor. This discrepancy is shown by the data of the dielectric loss as well.

However, the data can be corrected by assuming that ε_∞, the high frequency limit of ε', is equal for the bulk and the confined polymer, i.e. it is independent on the preparation of the PBLG and is not affected by any confinement. Then, the measured values $\varepsilon''_{measured}$ have been corrected to $\varepsilon''_{corrected}$ according to Eq. (2):

$$\varepsilon''_{corrected} = \frac{\varepsilon_\infty^{bulk}}{\varepsilon_\infty^{grafted}} \varepsilon''_{measured} \quad (2)$$

In Fig. 2, the raw and the corrected data are shown for the side chain relaxation. However, the preparation technique as well as the treatment of data works likewise for the second expected relaxation process, the chop stick motion. The dielectric loss of the corrected data is of the same order of magnitude for the bulk and the grafted polymer, and it becomes obvious that the side chain relaxation in the grafted layer is clearly broader and slightly shifted to larger relaxation times with respect to the bulk. This effect of broadening of the relaxation time distribution is in accordance with findings of similar measurements on thin films [5] as well as on low molecular weight glass forming liquids confined to a nanoporous matrix [6].

While in the cited publications this result has been discussed as an indication for the existence of cooperative rearranging regions (CRR) which determine the dynamics in vicinity of the glass transition temperature T_g, this explanation does not hold in our case. Since the side chain fluctuation should not be affected by the geometric confinement, we explain our findings by the assumption that the potential governing the dynamics of the side
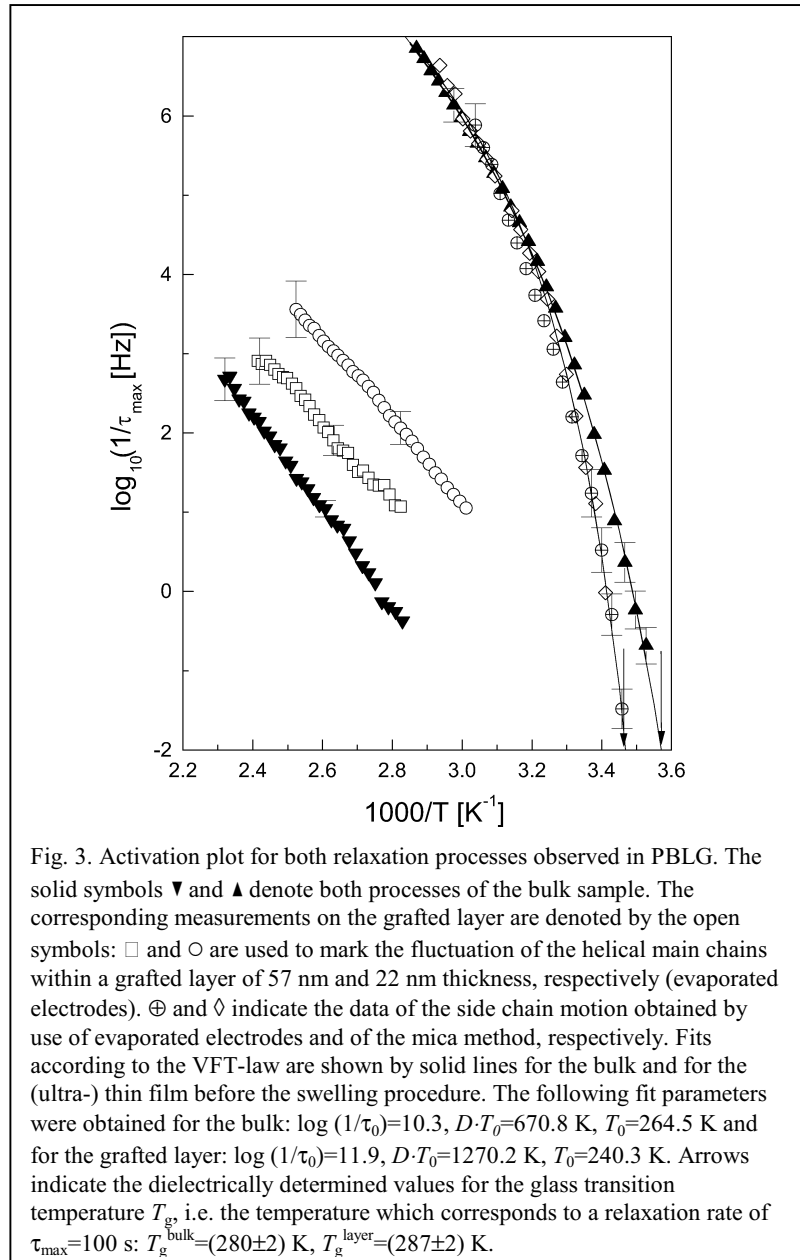

Fig. 3. Activation plot for both relaxation processes observed in PBLG. The solid symbols ▼ and ▲ denote both processes of the bulk sample. The corresponding measurements on the grafted layer are denoted by the open symbols: □ and ○ are used to mark the fluctuation of the helical main chains within a grafted layer of 57 nm and 22 nm thickness, respectively (evaporated electrodes). ⊕ and ◊ indicate the data of the side chain motion obtained by use of evaporated electrodes and of the mica method, respectively. Fits according to the VFT-law are shown by solid lines for the bulk and for the (ultra-) thin film before the swelling procedure. The following fit parameters were obtained for the bulk: log $(1/\tau_0)$=10.3, $D \cdot T_0$=670.8 K, T_0=264.5 K and for the grafted layer: log $(1/\tau_0)$=11.9, $D \cdot T_0$=1270.2 K, T_0=240.3 K. Arrows indicate the dielectrically determined values for the glass transition temperature T_g, i.e. the temperature which corresponds to a relaxation rate of τ_{max}=100 s: T_g^{bulk}=(280±2) K, T_g^{layer}=(287±2) K.

chain dipoles is more heterogeneous in the grafted layer than in the bulk, thus leading to a broadening of this process.

Concerning the thermal activation (Fig. 3) one has to point out that the local fluctuation of the side chains would be expected to have an Arrhenius-like activation behaviour. This was indeed found for glutamates with only methyl side chains [7]. However, for PBLG or similar substances with longer side chains the thermal activation of the corresponding relaxation can be well described by the VFT law [4,8] thus indicating that in these cases the side chains undergo a glass transition.

As a further difference between the bulk and the grafted layer besides the broadening of the

relaxation time distribution it is found that the dynamic glass transition of the side chains in the (ultra-) thin grafted layer is slowed down in comparison to the bulk (Fig. 3). Since these differences between the bulk and the grafted layer were obtained independently from the preparation of the bulk sample and from the way of contacting the (ultra-) thin layer, they have to be attributed to the different structure of both films. To confirm this, further investigations on grafted films with different thicknesses and on spin coated films of PBLG have to be done.

The chop stick motion in the grafted layer is found to be faster depending on the thickness of the grafted layer with respect to the bulk reference (Fig. 3). This could

be explained by the assumption that the molecular weight of the grafted PBLG is smaller than in the bulk. However, by means of gel permeation chromatography (GPC) similar molecular weights for the bulk and for even the thinnest grafted layer have been determined (bulk: M_n=285000, M_w/M_n=1.65; grafted layer (thickness: 22nm): M_n=245000, M_w/M_n=1.95). Thus, it can be ruled out, that the observed effect is due to a decreased molecular weight of the grafted PBLG compared to that of the bulk. We therefore assume that again differences between the structures of the bulk and the grafted layer are responsible for the shift of the chop stick motion to shorter relaxation times. Corresponding investigations on the structure of the grafted PBLG are in progress.

To improve the contact between the layer and the upper electrode, the PBLG has been swollen in a chloroform atmosphere for several hours. As result of this procedure, a higher capacity was achieved, which was in better agreement with calculated values. While the measured capacity was around $3 \cdot 10^{-10}$ F, the calculated value is $8 \cdot 10^{-10}$ F. Furthermore, as a result of the swelling procedure the side chain fluctuation becomes faster in the swollen sample (Fig. 4). With increasing temperature, the content of chloroform decreases and the relaxation rate tends to the values of the unswollen sample. The measurement from high to low values of the temperature shows the same thermal activation like the unswollen sample, thus indicating that the swelling process is fully reversible. The chop stick motion is not affected by the swelling procedure.

In summary, a novel method is introduced which enables to our best knowledge for the first time to study the molecular dynamics in (ultra-) thin layers of grafted PBLG. The results are compared to measurements on the bulk sample and on grafted PBLG layers contacted by evaporating metal electrodes. Two relaxation processes are observed which are assigned to (restricted) fluctuations of the helical main chains as a whole (chop stick motion) and to the dynamic glass transition of the side chains. The latter shows a VFT-temperature dependence which scales well with calorimetric
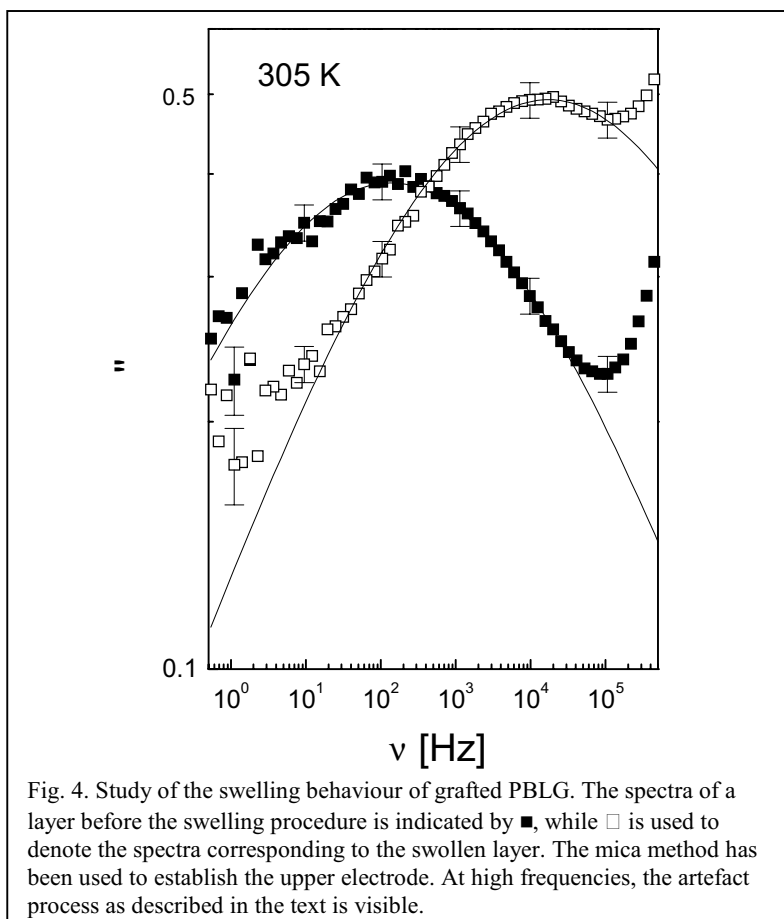

Fig. 4. Study of the swelling behaviour of grafted PBLG. The spectra of a layer before the swelling procedure is indicated by ■, while □ is used to denote the spectra corresponding to the swollen layer. The mica method has been used to establish the upper electrode. At high frequencies, the artefact process as described in the text is visible.

measurements [10]. Its relaxation time distribution is broadened in case of the grafted layers with respect to the bulk and its relaxation rate τ_{max} is slightly shifted to longer times. With decreasing film thickness, the chop stick motion is shifted to shorter relaxation times. These changes in the relaxation processes are explained by a higher order in the grafted layer compared to the bulk. Results obtained for the dielectric strength $\Delta\varepsilon$ are in good agreement with calculations based on free dipole fluctuations (side chain motion) and on the model of Wang and Pecora (chop stick motion). The discussion of $\Delta\varepsilon$ can be found in more detail in ref. [2]. Swelling the polymer with chloroform leads to a faster motion of the side chains. This effect is fully reversible.

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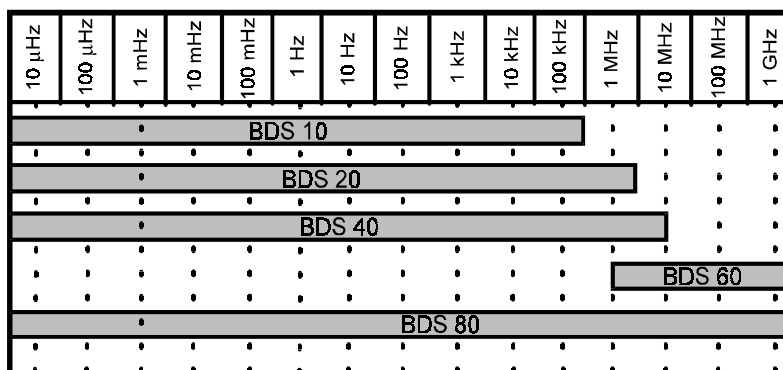[9] H. Block, *Poly(γ-benzyl-L-glutamate) and other glutamic acid containing polymers*, 1st edition, Gordon and Breach Science Publishers Inc. New York, 1983, Chapter 6.

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