Scaling of broadband dielectric data of glass-forming liquids and plastic crystals

U. Schneider, R. Brand, P. Lunkenheimer, and A. Loidl
Experimentalphysik V, Universität Augsburg, D-86135 Augsburg, Germany

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Abstract. The Nagel-scaling and the modified scaling procedure proposed recently by Dendzik et al. have been applied to broadband dielectric data on two glass-forming liquids (glycerol and propylene carbonate) and three plastic crystals (ortho-carborane, meta-carborane, and 1-cyano-adamantane). Our data extend the upper limit of the abscissa range to considerably higher values than in previously published analyses. At the highest frequencies investigated, deviations from a single master curve show up which are most pronounced in the Dendzik-scaling plot. The loss curves of the plastic crystals do not scale in the Nagel-plot, but they fall onto a separate master curve in the Dendzik-plot. In addition, we address the question of a possible divergence of the static susceptibility near the Vogel-Fulcher temperature. For this purpose, the low-temperature evolution of the high-frequency wing of the dielectric loss peaks is investigated in detail. No convincing proof for such a divergence can be deduced from the present broadband data.

PACS. 77.22.Gm Dielectric loss and relaxation – 64.70.Pf Glass transitions

1 Introduction

Investigations of the dynamic response of glass-forming materials are of fundamental importance to describe glass transition phenomena 1. Here the α- or structural relaxation is the most commonly investigated dynamic process. In the frequency dependence of the dielectric loss, ε″(ν), of dipolar systems the α-peak is the dominating spectral response, strongly slowing down with decreasing temperature. The microscopic origin of the spectral shape of the α-peak, which significantly deviates from the mono-dispersive Debye behaviour, is still unclear in many respects. Usually phenomenological functions as, e.g., the Cole-Davidson (CD) function 2 or the Fourier transform of the Kohlrausch-Williams-Watts (KWW) function 3 are used to describe the α-peak. However, these empirical functions, which extrapolate to a power law, ε″ ∼ ν⁻β, β < 1, at high frequencies, provide good fits of the experimental data at best up to 3 decades above the peak frequency, only. At higher frequencies an excess wing appears, which can be described by a second power law, ε″ ∼ ν⁻𝑏 with b < β 456. This excess wing was already noted in the early work of Davidson and Cole 7 and is a universal feature of glass-forming liquids 4567, at least for all systems where a β-relaxation is present (see below). Recently, we were able to show that this excess wing does not show up in various plastic crystals 7, a class of materials, whose properties resemble those of glass-forming liquids in many respects. In plastic crystals the centres of mass of the molecules form a regular crystalline lattice but the molecules are dynamically disordered with respect to the orientational degrees of freedom. Due to similarities in the relaxation dynamics, orientationally disordered crystals are often considered as model systems for structural glasses, but are much simpler to be treated in theoretical approaches.

It has to be stated that until now there is no commonly accepted explanation of the microscopic origin of the excess wing in glass-forming liquids, although there are some theoretical approaches that are able to describe the wing at least partly 10. In addition, it is possible to describe α-peak and wing by a superposition of two relaxation processes 111213. A secondary relaxation process, usually termed β-process, was demonstrated to be present in a variety of glass formers by Johari and Goldstein 14. Also recent theoretical developments within the coupling model 15 may point in the direction of a universal β-relaxation, closely connected to the α-process 1215 and it cannot be excluded that the wing is caused by a β-relaxation, whose peak is hidden under the dominating α-peak. Of course, as long as the occurrence of the excess wing and its evolution with temperature remains a mystery, also its absence in plastic crystals cannot be explained.

A very successful description of the wing is given by the so-called Nagel-scaling 15. Nagel and coworkers found that ε″(ν)-curves for different temperatures and even for different materials, including the wing, can be scaled onto one master curve by plotting Yν := 1/w log10[ε″νp/(Δεν)] vs. Xν := 1/w(1 + 1/w) log10(ν/νp). Here w denotes the half-width of the loss peak normalized to that of the Debye-peak, νp is the peak frequency, and Δεν the relaxation...
strength. During the past years some criticism of the Nagel-scaling arose concerning its universality [18,19,20] and accuracy [18,19,21,22,23]. However, it is still commonly believed that the Nagel-scaling is of significance for our understanding of glass-forming liquids and many efforts have been made to check for its validity in a variety of materials [19,20,21,22,23]. Recently a modification of the original scaling procedure was proposed by Dendzik et al. [19] and reported to lead to a better scaling, especially at low frequencies. Here $\Delta T := \log_{10}\Delta \nu/(\Delta \omega)$ is plotted vs. $X_D := (1 + \beta) \log_{10}(\nu/\nu_s)$ with $\nu_s$ and $\nu$ read of at the intersection point of the two power laws $\varepsilon'' \sim \nu$ and $\varepsilon'' \sim \nu^{-\gamma}$ below and above $\nu_p$, respectively.

The universal scaling of $\alpha$-peak and excess wing strongly suggests a correlation of both features. Especially, making certain assumptions, it was shown [24] that the wing exponent $b$ should become zero for a limiting relative half width, $w^* \approx 2.6$. It was argued, that this constant loss behaviour could imply a divergence of the static susceptibility, supporting speculations about an underlying phase transition from the liquid to the glassy state. By a low temperature extrapolation of $w(T)$ curves of various glass-formers, Nagel and coworkers deduced, that this constant-loss limit is reached near the Vogel-Fulcher temperature $T_{VF}$ [21]. $T_{VF}$ is determined from fits of the $\alpha$-relaxation time $\tau$ with the Vogel-Fulcher law, $\tau = \tau_0 \exp[D/(T - T_{VF})]$. In addition, it was shown [6] that, if the scaling is valid and assuming $w = 1/\beta$, the exponents $\beta$ and $b$ are related by

$$\frac{b + 1}{\beta + 1} = \gamma,$$

where $\gamma$ should be equal to the high-frequency limiting slope in the Nagel-plot, $\gamma'$. Indeed, in [6] within the error bars a temperature-independent value $\gamma = 0.72(\pm 0.02)$ was found, in agreement with $\gamma'$ read off from the master curve of the Nagel-scaling. Finally, a "divergent" susceptibility was also deduced directly from an extrapolation of $b$ to low temperatures, which for various glass formers indeed seemed to approach zero near $T_{VF}$ [4].

Our group has made available dielectric loss data on glass-forming liquids and plastic crystals extending over the exceptionally broad dynamic range of up to 19 frequency decades [6,23,24,25,26,27,28,29]. The present work has two objectives: First, to check if scaling still works for these extremely broadband data. Second, to look for evidence for a divergent dielectric susceptibility at low temperatures. We will apply both, the original Nagel-scaling [8] and the modified procedure proposed by Dendzik et al. [19] to broadband dielectric data on the low-molecular weight glass-formers glycerol and propylene-carbonate (PC) and on the plastic crystals ortho-carborane (o-CA), meta-carborane (m-CA), and 1-cyano-adamantane (CNA). In addition, these data will be checked for a cross-over of the high-frequency wing to constant loss behaviour at low temperatures.

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**Fig. 1.** Frequency dependence of the dielectric loss of PC (a) and glycerol (b) for various temperatures. For clarity reasons, only selected temperatures are shown and for frequencies $\nu > 1$ GHz only a part of the recorded data points are plotted (for the complete curves, see [28,29]). The solid lines are fits with the CD function. The arrows indicate the cutoff frequency up to which the data have been used for the scaling plots of Figure 2.

**2 Experimental**

To collect data in a broad frequency range, various experimental setups were used, including a time domain spectrometer (10 kHz $\leq \nu \leq 1$ kHz), frequency response analyzers and LCR-meters (10 mHz $\leq \nu \leq 30$ MHz), impedance analyzers (reflectometric technique, 1 MHz $\leq \nu \leq 1.8$ GHz), a network analyzer (reflection and transmission, 100 MHz $\leq \nu \leq 30$ GHz), a quasi-optic submillimeter wavelength spectrometer (transmission and phase measurement, 40 GHz $\leq \nu \leq 1.2$ THz), and a Fourier-transform infrared spectrometer (transmission, 600 GHz $\leq \nu \leq 3$ THz). For more experimental details the reader is referred to [30].

**3 Results and discussion**

Figure 1 shows the frequency dependence of the dielectric loss of PC and glycerol for various temperatures [28,29]. $\varepsilon''(\nu)$ exhibits the typical asymmetrically shaped $\alpha$-relaxation peaks shifting through the frequency window
with temperature. At high frequencies, in the GHz – THz range, a minimum shows up, followed by another loss peak (the microscopic or boson peak). These high-frequency features are treated in detail in [24-26,27,28,29]. The solid lines in Figure 1 are fits of the α-peak region with the empirical CD function [3], which leads to good fits of the peak region. It was shown before [24,25,26,27,28,29], that the CD function describes the data clearly better than the Fourier transform of the KWW function. At frequencies about 2 – 3 decades above ωp the excess wing shows up as a deviation of ε''(ω) from the CD-fits, forming a second power law, ε'' ∝ ω^-b. The slope of the excess wing increases with increasing temperature as found previously for other glass-forming materials [1]. For high temperatures the excess wing seems to merge with the α-peak. At the lowest temperatures investigated, for both materials in the excess wing region a slight downward curvature of ε''(ω) may be suspected. This could be indicative for a β-relaxation process. The associated β-peak could be either superimposed to the excess wing, becoming important at low temperatures only [10], or it may even be the origin of the whole wing-feature [11,12,13]. However, the experimental errors are too large to allow for a definite conclusion concerning the presence of a β-peak and further experiments are in progress to clarify this question.

The data on glycerol and PC, after application of the Nagel [8] and Dendzik-scaling [19] are shown in Figures 2(a) and (b), respectively. To maintain readability, for each material ε''(ω) curves for three different temperatures are shown only. For both materials the lowest temperature has been omitted, as here, due to the relatively high experimental errors near the α-peak region, an unequivocal determination of the scaling parameters is difficult. We also want to avoid contributions from a possible β-peak, superimposed to the wing at low temperatures as, e.g., found for Salol [22]. The data have not been corrected for conductivity contributions, which only weakly affect ε''(ω) about 1–2 frequency decades below ωp. The effect of the conductivity and the correct method of subtraction are a matter of controversy [22,23] and therefore we decided to show the raw data. The scaling cannot be expected to work in the region of the ε''-minimum in the GHz – THz region. Therefore only data-points up to a cutoff frequency that is indicated by arrows in Figure 1 were used. The maximum value of X used at the different temperatures is indicated by the symbols. For the Dendzik-scaling, the present plots extend to higher values of XD than any previously published scaling plots. For the Nagel-scaling, a plot up to XN = 12 has been published in [24] but for one material only (glycerol). In addition, a Nagel-plot up to XN = 16, comparing different glass formers, was shown in [22]. However, this plot was obtained using data where the α-peak was missing and some crude assumptions for the determination of the scaling parameters had to be made to arrive at the scaling at high frequencies. Figure 2 reveals, that both scaling procedures lead to good scaling of the data at not too high values of X. There are reports of some shortcomings of the Nagel-scaling in the region near X = 0 [18,19,20]. In contrast, the modified procedure proposed by Dendzik et al. was shown to lead to a better scaling in this region [14]. For the present data on PC and glycerol in this region [lower insets in Figures 2(a) and (b)] indeed the quality of the Nagel-scaling seems to be slightly worse, but the differences are very subtle. It was shown that the modified procedure proposed by Dendzik et al. is able to scale different CD curves onto one master curve [19]. At high frequencies this master curve is a straight line with slope -1 [straight dashed line in Figure 2(b)]. Then the excess wing should show up as deviations from the CD master curve at high XD values, as is indeed seen in Figure 2(b). The most interesting result of these scaling plots

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1 Indeed, for PC, even after an optimization of the Nagel-scaling parameters [23] within the experimental errors, the 153 K curve shows pronounced deviations from the curves at higher temperatures in the whole frequency range (not shown).
is, that at high values of $X$, in both scaling approaches deviations from a single master curve show up. These deviations are more pronounced in the Dendzik-scaling. For example, in the Dendzik-scaling the curves at 158 K for PC and at 195 K for glycerol start to significantly deviate from each other above an $X_D$-value corresponding to 50 Hz for PC and 1 kHz for glycerol. In the Nagel-plot this deviation occurs at 43 kHz for PC and at 3 MHz for glycerol. While for the Nagel-scaling the deviations are almost within the experimental errors, this is not the case for the Dendzik-scaling (see error bars in Figure 2), which clearly fails at high frequencies. However, for both procedures the curves obtained at different temperatures for a single material scale (also including data at temperatures not shown in Figure 2).

A general problem of the scaling plots is the fact that the $Y$-value varies considerably, roughly following $Y = -X$, but the information of interest is the presence of small deviations from this variation. Therefore a much better resolution can be reached when correcting for this $Y = -X$ behaviour. This can be achieved by plotting the quantity $Z(X) = Y(X) + X$. In the upper insets of Figures 3(a) and (b) the data at $X > 3$ are plotted in this way. Clearly the resolution of the plots is enhanced and the deviations of the different curves from a universal scaling behaviour become more obvious.

Finally it may be mentioned that for high temperatures ($T \geq 253$ K in glycerol and $T \geq 203$ K in PC), the dielectric loss data can also be scaled onto each other by simply plotting $\varepsilon / \varepsilon_p$ vs. $\nu / \nu_p$ with $\varepsilon_p$ being the peak value of $\varepsilon''(\nu)$. Here the excess wing seems to have merged with the $\alpha$-peak and $\beta$ is nearly temperature independent.

In Figure 3 both scaling plots are shown for the plastic crystals (o-CA, m-CA, and CNA). These plastic crystals have a rigid molecular structure and $\beta$-relaxation contributions from intramolecular degrees of freedom can be excluded. Their dielectric loss was shown to exhibit no or only a very weak wing [1]. In Figure 3 for clarity reasons, curves for two temperatures only are shown for each material. Clearly, the Nagel-scaling [Figure 3(a)] does not work for the plastic crystals investigated [2]. Again, in the upper inset of Figure 3(a) the deviations become more obvious. The deviations occur at much lower $X_N$-values and are much stronger than for the glass-forming liquids [compare Figure 3(a)]. This can be rationalized considering that the $\varepsilon''(\nu)$-curves of the plastic crystals all follow a CD behaviour quite closely up to the highest frequencies [2] and that different CD-curves do not scale in the Nagel-plot [7]. In contrast, in the Dendzik-plot [Figure 3(b)] all curves for the plastic crystals scale onto the CD master curve (dashed line). It should be noted, that these plastic crystals are relatively "strong" glass-formers [24, 32, 33] within the classification scheme proposed by Angell and coworkers [34]. It cannot be excluded that the failing of the Nagel-scaling in these materials is due to their high strength and not due to their plastic crystalline nature. This would also be in accord with Ngai’s ideas [12], concerning an explanation of the excess wing within the coupling model. Unfortunately, to our knowledge the Nagel-scaling was never applied to strong structural glass formers.

Finally, we want to check, if the excess wing approaches constant-loss behaviour at low temperatures in the samples investigated. Figure 4 shows the temperature dependence of $\beta_{CD}$ for PC and glycerol, as resulting from the fits of the $\alpha$-peak with the CD function (Figure 2). For the CD function, the limiting half width, $w^* \approx 2.6$, at which $\varepsilon''(0) = \text{const} = 2$ corresponds to $\beta^* \approx 0.18$. Inspecting Figure 4, it seems unlikely that $\beta_{CD}(T)$ will reach $\beta^*$ at $T \approx T_{VF}$. From equation (1) and assuming $\gamma = \gamma' = 0.72$ a different limiting value for the divergence of the static susceptibility, $\beta^* \approx 0.38$, can be deduced [2]. While a linear extrapolation of $\beta(T)$ leads to a somewhat higher value of $\beta(T_{VF})$, an extrapolation to $\beta(T_{VF}) = 0.38$ at least is consistent with the data. The discrepancy between $\beta^*$ and $\beta^*$ arises due to the poor fulfillment of the assumption $\beta = 1/w$ made for the deduction of equation (1) from the Nagel-scaling. In contrast to this, for the Dendzik-scaling, equation (1) should be valid without such an assumption. However, while for the Nagel-scaling a sample-independent value of $\gamma' = 0.72$ is consistent with our data [Figure 2(a)], for the Dendzik-scaling the value of $\gamma'$, read
In order to take into account the additional contributions superimposed to each other. For this purpose, a phenomenological ansatz was shown to provide good fits over a considerable frequency range. In contrast to the scaling plots, for this analysis the lowest-temperature curves have been included as at least the \( \nu^{-\beta} \) power-law seems to be well defined. In addition, a possible \( \beta \)-peak, superimposed to the wing, may be expected to have only a small influence on the exponent \( b \) determined from the fit. The resulting \( b(T) \) and \( \beta(T) \) are shown in Figure 4. \( \beta(T) \) tends to assume somewhat higher values than \( \beta_{CD} \) due to the contributions of the wing power law \( \nu^{-b} \) at low frequencies. In Figure 4 also the results of Leheny and Nagel are included (crosses). Due to the larger frequency range available to us and the different evaluation procedure we were able to extend the results of these authors to considerably higher temperatures. However, results of \( b(T) \) could only be obtained up to 193 K for PC and up to 234 K for glycerol. Above these temperatures the excess wing has almost merged with the \( \alpha \)-peak and an unequivocal determination of \( b \) is no longer possible.

In the insets of Figure 4 the obtained results for \( b \) and \( \beta \) are used to check the prediction \( \gamma = \gamma' \) arising from the Nagel-scaling. For both materials \( \gamma \) agrees roughly with the value of \( \gamma' = 0.72(\pm 0.02) \), found from the high-frequency limiting slope of the Nagel-scaling master curve, however, exhibiting a tendency to increase with temperature. Again, for comparison the results of Leheny and Nagel are included (stars). It is interesting that the slight temperature variation of \( \gamma \), seen in the data of Leheny and Nagel agrees with that detected in the present results. This indicates a systematic nature of this temperature dependence in contrast to the assumption of a constant \( \gamma(T) \) within the error bars, made in [3]. The significant increase of \( \gamma(T) \), detected for both materials, seems reasonable in light of the merging of excess wing and \( \alpha \)-peak at high temperatures which can be suspected from Figure 4. This implies the approach of \( \gamma = 1 \) for high temperatures.

As demonstrated by the solid lines in Figure 4, \( b(T) \) can be reasonably extrapolated to zero at \( T_{VF} \) for PC and to about \( T_{VF} + 25 \) K for glycerol. The result for PC indeed seems to support the approach of a constant loss at \( T_{VF} \). However, even if \( b \) becomes zero, a "divergent" susceptibility will only be reached, if the pre-factor \( c_b(T) \) does not approach zero for \( T \rightarrow T_{VF} \). In Figure 4 \( c_b(T) \) is shown for both materials. As indicated by the dashed lines the results do not allow for a definite conclusion concerning a finite value at \( T_{VF} \).

For the plastic crystals, the situation is different, as here the wing is missing and a "divergent" susceptibility would only be reached if \( \beta(T) \) approached zero at a finite temperature. Figure 4 shows the temperature dependence of \( \beta_{CD} \) for o-CA and m-CA. Indeed, \( \beta_{CD}(T) \) weakly decreases with decreasing temperature. Due to the fact that these plastic crystals are relatively "strong" glass-formers [23, 33], \( T_{VF} \) is difficult to determine. It can be expected to be located far below \( T_g \) where it is not possible to obtain significant information from an
eral failure of the Dendzik-scaling at the highest frequencies. The extended frequency range available reveals a gen-
to a satisfactory scaling of the loss data at low frequen-

\[ \beta(T) \]

The line in Figure 6 proposes a linear extrapolation of the low temperature data to \( \beta_{CD}(T = 0\text{ K}) = 0 \) but other extrapolations are also possible.

4 Conclusions

In summary, we have investigated the scaling of extremely broadband dielectric data using two different procedures. The spectra on two glass-forming liquids and three plastic crystals have been analysed, extending the range of the abscissa to higher values than in former analyses. For the glass-forming liquids, the approaches proposed by Nagel and coworkers [9] and by Dendzik et al. [10] both lead to a satisfactory scaling of the loss data at low frequencies. The extended frequency range available reveals a general failure of the Dendzik-scaling at the highest frequen-
cies investigated, while this effect is almost negligible for the Nagel-scaling. More broadband data on glass-forming liquids are needed to check for the significance and universality of these deviations. If they were confirmed, this may indicate that the wing is not so intimately related to the \( \alpha \)-relaxation as thought up to now. Then it could be caused by processes, other than the \( \alpha \)-process (e.g. a \( \beta \)-relaxation [11,12]). But also a superposition of a very weak \( \beta \)-relaxation and the wing may be invoked to explain the failing of the scaling at high frequencies [20,23,11].

In addition, the broader frequency range of our data allowed for an extended check of the cross-over of the wing to constant-loss behavior at a temperature near \( T_{V_F} \), claimed by Nagel and coworkers [9,24]. The temperature dependence of the width parameter of the \( \alpha \)-peak gives no evidence for such a behaviour. For PC and to a lesser extent also for glycerol, the temperature dependence of the wing exponent may be consistent with the proposed approach of a divergent static susceptibility. However, the development of the power law prefactor for low temperatures is still an open question. In our opinion the dielectric data reported in literature and even our extended set of data do not provide a convincing experimental proof for a divergent susceptibility at low temperatures.

The data on the plastic crystals clearly cannot be scaled by the procedure proposed by Nagel and coworkers [9]. But using the Dendzik-scaling, all curves from the investigated plastic crystals fall onto one master curve, different from the master curve of the glass-forming liquids. This master curve is identical with the master curve for the Cole-Davidson function. This result corroborates the recent finding that plastic crystals without intramolecular degrees of freedom do not show an excess wing but follow a CD-behaviour up to the highest frequencies [11]. In light of the missing excess wing, for plastic crystals a divergence of the static susceptibility at finite temperatures seems unlikely, but cannot be fully excluded.

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