Electrical properties of polyetherimide thin films: Non-parametric dielectric response analysis with distribution of relaxation times

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Abstract. High temperature polymeric materials for electrical insulation and energy storage are needed for transformational power applications such as pulsed-power and hybrid electrical vehicles. One of the candidate materials has been polyetherimide, an amorphous thermoplastic with a glass transition over 200 °C. Here, the dielectric studies on the material are reported by taking into account the polarization and conduction processes in the polyetherimide. The dielectric data were analyzed with the distribution of relaxation times approach with Debye expression as the kernel in the inversion algorithm, the results then created the relaxation map for polyetherimide. The method satisfies the Kramers-Kronig relationships, so the ohmic conductivity and permittivity at high frequencies could be estimated from the experimental data even if there exists significant measurement error. The data were compared to the published results on polyetherimide in the literature. The materials is a low loss polymer with negligible ohmic losses below 200 °C. The estimated fragility of the polyetherimide was high about 284 from the resolved relaxation map using the Vogel-Fulcher-Tammann-Hesse expression. Polyetherimide is a good dielectric for advanced energy storage and electrical insulation technologies up to 200 °C.

1 Introduction

General Electric developed polyetherimide in its Corporate Research Center in the early 1970s [1,2]. The material is a high performance amorphous thermoplastic (class H insulation, 180°C operation temperature) dielectric with low losses [3], and with high glass transition temperature ≈ 215 °C [4, 5]. Different grades of polyetherimide have been derived for specific applications [6]. Its low loss behavior and high glass transition temperature property make it a valuable alternative to some of the conventional electrical insulation materials and dielectrics, i.e., polypropylene based thin film electrostatic capacitors [7-9]. Polymeric thin film based electrostatic capacitors require materials with high dielectric permittivity, low dielectric losses (see Sarjeant et al. [7] and Reed and Cichanowski [8] for reviews on capacitor technology). One should not forget the process-ability of the film and working range of the polymer; and high temperature capability. Conventional polymeric film capacitors are mainly designed with polypropylene, polyethylene teraphthalate [9] and polycarbonate films.

Previous studies on polyetherimide have indicated that depending on the grade of the material [6] and its moisture content [10], dielectric behavior as a function of temperature and frequency were different. Molecular mobility, chain dynamics and merging of the segmental local motion (β-relaxation) and cooperative process (α-relaxation) are studied through the dynamic glass transition [11, 12] via dielectric or impedance spectroscopy [13, 14]. We determine the charge transport just before and after the glass transition. The behavior of charge mobility and polarization determines the electrostatic charging of the film and how it can be dissipated during the production of capacitor devices [15, 16]. Special precautions needs to be taken to abate static charge on near-to-perfect insulating dielectrics films.

Here we concentrate on the dielectric relaxation properties of an amorphous polymer, polyetherimide, for dielectric applications. The paper is organized as follows: a brief introduction to the distribution of relaxation times approach is given in sect. 2; the information on polyetherimide and the measurements are described in sect. 3; results on electrical properties and resolved distribution of relaxation times with information on the fragility are presented in sect. 4; finally, a discussion and conclusions are presented in sect. 5.

2 Distribution of relaxation times approach

2.1 Preliminary results

In order to separate the frequency-independent intrinsic properties from those of the frequency-dependent ones,
a method based on the distribution of relaxation times was adopted [17–22]. Different approaches have been suggested to resolve the distribution function from experimental data in the literature [23–31]. The method developed to resolve the relaxation times by the present author is a non-parametric approach and it is universal for any linear dielectric system [32–34], which does not suffer from initial guesses, a priori assumptions, such as selection of empirical expression(s) or model and predictions for the model parameters. The method resolves continuous distributions and has been applied to many different systems previously (see refs. [32–43]). The method has no limitations related to the frequency, even the low frequency dispersion in dielectrics [21] can be analyzed with the distribution of relaxation times approach [43].

2.2 Dielectric representation

The relative complex dielectric permittivity of materials \( \varepsilon^* \) as a function of state variables (angular frequency \( \omega \), temperature \( T \), pressure \( P \), etc.) should be expressed as follows:

\[
\varepsilon^*(\omega, T, P, \ldots) = \chi^*(\omega, T, P, \ldots) + \varepsilon_\infty(T, P, \ldots) + \sigma_0(T, P, \ldots) (i\omega_0 \omega)^{-1}, \tag{1}
\]

here \( \chi^* \) is the complex dielectric susceptibility; \( \varepsilon_\infty \) is the permittivity at optical frequencies or higher frequencies than the probed frequency region; \( \varepsilon_0 \) is the permittivity of vacuum, \( \varepsilon_0 = 8.854 \text{ pFm}^{-1} \); and \( \sigma_0 \) is the ohmic conductivity of the material. For low loss dielectric or good electrical insulators to resolve their low conductivity one needs to perform measurements at low frequencies where the ohmic loss contribution becomes significant compared to the polarization losses.

In the adopted formalism in the data analysis method, the complex permittivity is expressed as

\[
\varepsilon^*(\omega, T) = \chi^*(\omega, T) + \varepsilon_\infty(T) + \sigma_0(T)(i\omega_0 \omega)^{-1}, \tag{2}
\]

with

\[
\chi^*(\omega, T) = \int_0^\infty g(\tau, T)(1 + i\omega\tau(T))d\tau, \tag{3}
\]

where \( g(\tau) \) is the distribution of relaxation times and \( \tau \) is the time constant. The frequency-dependent term on the right-hand side inside the parenthesis in eq. (3) is the kernel, and it is the response of a single dipole derived by Debye [44] (see also Macdonald [45] and Fröhlich [17]). Observe that the two material parameters \( \varepsilon_\infty(T, P) \) and \( \sigma_0(T, P) \) are frequency dependent. The numerical procedure yields the distribution of relaxation times \( g(\tau) \), the high frequency permittivity \( \varepsilon_\infty \) and the ohmic conductivity \( \sigma_0 \). Solution and numerical details to this inverse problem and other problems in dielectrics are discussed elsewhere [33, 34, 46–51].

The most probable relaxation time \( \bar{\tau} \) for a process is estimated with

\[
\bar{\tau} = \left[ \int_a^b \tau \times g(\tau)d\tau \right] \times \left[ \int_a^b g(\tau)d\tau \right]^{-1}. \tag{4}
\]

The integral limits \( (a, b) \) were taken from the corresponding time axis \( (\sim \omega^{-1}) \) for the measurement window frequencies.

3 Materials and methods

The studied film was a 5 \( \mu \text{m} \) polyetherimide film (commercially known as Ultem®1000 from SABIC Innovative PlasticsTM). The studied film was supplied from Shin-Etsu Polymer Co., Ltd Japan.

We have employed a Novocontrol Quatro Broadband Dielectric Spectrometer to characterize the electrical properties of a film metalized with thermally evaporated aluminum. The frequency \( \nu \) window in the measurement was between 0.3 Hz and 1 MHz. Since polyetherimide is prone to absorb water [10], the sample was pre-heated under nitrogen flow to 200 °C to remove water; this temperature was close to the reported glass transition temperature in the product sheet (\( T_g = 215 \) by ASTM D3418). The temperature during the measurements was between −110 °C and 270 °C.

4 Electrical properties of polyetherimide

4.1 Dielectric properties

The complex dielectric susceptibility \( \chi^* \) of a polyetherimide 5 \( \mu \text{m} \) thick film is shown for the data collected at 1 kHz in fig. 1. Because of the sensitivity of capacitance measurements to the film thickness, the measured relative permittivity value of polyetherimide was slightly lower \( (\varepsilon_r \sim 3.18) \) than the supplier reported data \( (\varepsilon_r \sim 3.15) \), which could be explained with the ±10% error in the thickness measurement for the film. The dielectric behavior of the film starts to change abruptly close to the glass transition temperature as expected. Isochronal measurement at 1 kHz applied electric field clearly indicates the different relaxations in polyetherimide: one at low temperatures peaking around 120 °C and one at high temperatures around 230 °C, see fig. 1.

The complex susceptibility \( \chi^* \) at 1 kHz is shown in fig. 1. The susceptibility was calculated with the representation described in eqs. (2) and (3). The inset in the figure shows the high frequency value of the permittivity, which does not change between 0 °C and 200 °C. Since the losses below 0 °C are low the large oscillations in the relative permittivity at high frequencies were related to the numerics.
Fig. 1. Complex dielectric susceptibility of polyetherimide as a function of temperature at 1 kHz. The susceptibility is calculated using eqs. (2) and (3) with the analysis of the distribution of relaxation times approach. The open (◦) and filled (●) symbols represent the real and imaginary parts of the susceptibility. The inset shows the permittivity at high frequencies as a function of temperature calculated with the distribution of relaxation times approach. The solid vertical lines show the position of the glass transition \( T_g \) temperature.

The polyetherimide is a low loss material below 200°. The evolution of the segmental motion is clear, where the losses \( \chi'' \) increase starting from the lowest temperature measured (−110 °C) to 200 °C. The cooperative motion or cold liquid phase start to rearrange the polyetherimide molecules. The observation shows that the polyetherimide is a hot liquid with molecules which easily change their confirmation over 255 °C, where the real part of the susceptibility is decreasing and the imaginary part is becoming low again; few frictional losses; no polarizability over 255 °C.

The low temperature process is the change in the segmental motion with temperature (assigned as the \( \beta \)-relaxation [11]) and is a broad relaxation. The \( \alpha \)-peak related to the cooperative motion of the polarizing units, which occur after the glass temperature, is much sharper in temperature axis and indicates the glass transition, softening of the polymer. The data presented by Belana et al. [4] on the physical properties of polyetherimide had similar findings from the mechanical and dielectric studies for the \( \beta \)- and \( \alpha \)-relaxations but at lower temperatures because they have presented their data at 10 Hz and 3 Hz for the electrical and mechanical measurement, respectively. The data presented by Diaz-Calleja et al. [5], on the other hand, presented dielectric loss data at different frequencies with labeled relaxations. The main difference between the current polyetherimide and the ones presented by Belana et al. [4] and Diaz-Calleja et al. [5] is the \( \gamma \)-relaxation, which is not present in the studied material. This observation indicates that the tested current film did not have any side-groups, which were proposed to contribute to the dielectric relaxation at low temperatures [5].

The space charge relaxation (denoted by \( \rho \)) mentioned by Belana et al. [4] could not be observed at 1 kHz, however, our data show the presence of this relaxation at low frequency isochronal data, which was not shown here. One should mention that the \( \rho \)-relaxation is related to the blocking electrodes and is an indication of free ions; therefore related to the \( \alpha \)-relaxation. Observe that the on-set position of the increase in the dielectric losses (\( \chi'' \)) and the increase in the relative permittivity at high frequencies \( \varepsilon_\infty \) are just below the glass transition reported in the literature [5,52]; the solid line in fig. 1 show the reported glass transition \( T_g = 218–220 \) °C. The complete melting of the polymer is visible over 240 °C, where the real and imaginary parts of the susceptibility are decreased quickly.

4.2 Relaxation map

The Argand plot of complex permittivity is shown in fig. 2. The relaxations just below and over \( T_g \) are shown due to clear change in the dielectric response. The \( \beta \)-relaxation is starting to merge with the \( \alpha \)-relaxation. The resolved relaxation times are shown in semi-circle which is due to the \( \alpha \)-relaxation. The data used to generate the responses...
Fig. 3. Distribution of relaxation times resolved for three temperatures as indicated in the plot. The distributions are shifted to guide the eyes. The data from bottom to top show the temperatures 207°C, 230°C and 244°C, respectively. The estimated expected relaxation times from eq. (4) are shown with filled symbols for the three temperatures.

were calculated using the distributions in fig. 3. The peak positions for the distributions are estimated using eq. (4). Observe that the relaxation at 207°C (labeled with • in the figure) has no clear peak in the considered experimental window. However using the expression we were able to assign a most expected relaxation to it. The other temperatures show relaxation peaks corresponding to α- and β-relaxations.

The dielectric response of polyetherimide at the three different temperatures is shown in fig. 2. The open symbols show the experimental values and the solid lines are the data obtained from the distribution of relaxation times approach. Below the glass transition no semi-arcs are observed, however with the formation of cold liquid after the glass transition the cooperative motion peak was visible. The conductivity was also getting into the picture as the temperature was increased further due to increased mobility of ions and molecules.

The distribution of relaxation times for these temperatures is shown in fig. 3. The distribution at 207°C is nearly flat with some indication of slow relaxation moving into the measurement window. Clear peaks for the cooperative motion are observed at 230°C and 244°C. The filled symbols for each temperature indicate the most expected relaxation time $\bar{\tau}$ from eq. (4), which was used to build the relaxation map.

Keeping track of the most expected relaxation times $\bar{\tau}$ at different temperature, we were able to compile the relaxation map as shown in fig. 4. The filled and unfilled symbols were used to differentiate between $\max \{ g(\tau) \}$ and the results from eq. (4) on the temperature region over $T_g$. Only the α relaxation yields maximum relaxation times at temperatures higher than the glass transition temperature $T_g$. The data from Fontanella et al. [10] is also shown in the figure with solid lines (curve “D”). The literature data was not able to represent the relaxations resolved in our analysis, which might be due to slight differences in the raw materials and temperature range; we were able to perform measurements up to 270°C. In fig. 4 the inset illustrates the cross-over region and the α-relaxation to indicate differences in the expressions used in the data analysis. No care was given here to separate α and β relaxations as in previous studies [10,52–56].

Several different models were adopted to model the relaxation map. The Arrhenius model has been applied to model the β relaxation of many relaxing systems. In our analysis since we do not have a clear peak that moves on a straight line in the inverse temperature logarithm of the relaxation rate representation for this relaxation in the distribution of the relaxation times function $g(\tau)$, the β-relaxation was not considered with the Arrhenius expression

$$\omega_0 = A \exp\left[-W_E(k_b T)^{-1}\right],$$  \hspace{1cm} (5)

where $A$ is the pre-exponential term usually around $10^{14}$ s; $W_E$ is the activation energy in eV units; $k_b$ is the...
Boltzmann constant $k_b = 8.6132 \times 10^{-5}$ eV; $T$ is the temperature in kelvins. However, we have adopted the Vogel-Fulcher-Tammann-Hesse (VFTH) equation [57–59], due to the bending of the curve to upward in the relaxation map:

$$\omega_0 = B \exp[D/(T - T')]$$  \tag{6}

where $B$ is the pre-exponential; $D$ is a fitting parameter in temperature units; $T'$ is the critical temperature $T > T'$. The segmental motion ($\beta$-relaxation) was modeled with $B = 2.24 \times 10^{13}$ and $D = -885.96$ and $T' = 612.35$ with negative temperature difference in the denominator. The VFTH expression can represent the data to temperatures close to the merging region of the two relaxations; it is labeled with “A” in the inset of fig. 4. After $\alpha$ and $\beta$ relaxations merge, it is interesting to see that the continuation of the fitted curve is also able to represent the $\alpha$-relaxation, which is defined by Donth [11] as the relaxation process at high temperatures after the two relaxations merged. Our modeling indicate that the segmental motion becomes faster as we approach to the glass transition. This observation was expected since the thermal energy provided would not only provide kinetic energy to relaxing units, but it would also change the landscape and might create free sites for the relaxing units to hop. It is hard to study thin samples at temperatures higher than the glass transition temperature due to phase change and melting of the polyetherimide, however, it would be valuable to record dielectric data at high temperature to attempt to follow the relaxations on this region ($T > 220^\circ$C) in future studies.

The $\alpha$-relaxation was modeled with four expressions: one Arrhenius, one VFTH and two William-Landel-Ferry [60] (WLF) models. The WLF model was adopted as proposed by the authors [60] and also modified with free parameters. The WLF expression is as follows:

$$\ln \omega_0 = C_1 (T - T_0) [C_2 + (T - T_0)]$$  \tag{7}

where $C_1$ and $C_2$ are fit parameters and $T_0$ is a reference temperature. The parameters $C_1$ and $C_2$ were given as 17.44 and 51.6, respectively [60, 61], if the reference temperature is taken to be the glass transition $T_g$ temperature (which is 215°C). This curve is illustrated in fig. 4 with label “C”. It was not enough to express the relaxation behavior of polyetherimide over the glass transition.

When we took all the parameters free and applied a curve-fitting procedure, the parameters obtained were as follows: $C_1 = 25.99$, $C_2 = 54.87$ and $T_0 = 485.51$, this curve is shown with “E” in fig. 4 inset. The glass transition temperature $T_g$ obtained from the WLF analysis is lower than the $T_g$ estimated with VFTH, which is shown with “E” in the inset of fig. 4. The VFTH parameters obtained from the analysis were $B = 2.156 \times 10^8$, $D = -446.86$, and $T' = 469.32$. Using the interpolation value at $\log_{10} \omega_0 = -2$ for the $T_g$ estimate, the glass transition $T_g$ temperature of the polyetherimide film is 214.57°C, which is close to the values published in the literature [4, 5, 10]. The data from Fontanella et al. [10] for the $\alpha$-relaxation is plotted in fig. 4 and labeled as curve “D”. Their Arrhenius model was not representing our data. Note that the volume recovery measurement performed on polyetherimide has reported a glass transition temperature $T_g$ of around 207.5°C.

The full relaxation map estimated by the distribution of relaxation times and the single point expected relaxation time estimates are shown in fig. 5. The relaxation times for the $\alpha$ process is much sharper than the $\beta$ one, which is nearly flat below the glass transition $T_g$. The relaxations resolved for $\beta$ resembles “fingers” coming out of the $\alpha$-relaxation. Below room temperature ($< 300$ K), there are no peaks in the map. The influence of the experimental error on the numerical error in the adopted inversion procedure has been estimated numerous times and has shown that the inversion method is not sensitive to the experimental data. One explanation to the “fingers” could be that this oscillating sort of behavior is due to the flat distribution as proposed by Fröhlich [17], who employed an arbitrary box distribution. Such a distribution was tested in the literature previously with the current analysis method [40]. The results have shown that one might get oscillations of this sort. However, in the polyetherimide case the oscillations were more significant.

### 4.3 Fragility of polyetherimide

The fragility of glass-forming molecular systems was studied by Angell, Ngai, Kenna, McMillan, and Martin [62]...
The non-Arrhenius temperature dependence of the relaxation is defined as the dynamic fragility. It refers to deviations from the Arrhenius temperature and is expressed with VFTH [57–59] (eq. (6)) or WLF [60] (eq. (7)) behavior. The fragility \( m \) is estimated as

\[
m = d(-\log_{10}\omega_0)d(T_g/T)^{-1}|_{T=T_g} \\
≡ DT_g(T_g - T)^{-2}\log_{10} c|_{T=T_g} \text{ for eq. (6)} \\
≡ C_1T_g(C_2)^{-1}|_{T=T_g} \text{ for eq. (7)}.
\]

The fragility index \( m \) indicates how rapidly the molecular system changes as it approaches to the glass transition temperature from the high temperature side of \( T_g \); similarly just after passing \( T_g \) from the low temperature side, the molecular system would re-arrange (re-configure) itself, which is abruptly in the polyetherimide. For high \( T_g \) thermoplastic polymers this change would yield to melting or even decomposition after the glass transition.

The polyetherimide has a high fragility \( m \) around 284 and 230 that is estimated from eq. (9) and eq. (10). These values are much higher than those reported for other polymers in the literature [11, 64–66]. The difference in our estimations using VFTH and WLF models should be understood in the view of how well these expressions describe the experimental data; as shown previously VFTH was a better approach to the data. Notice that the idea of using different curve fitting models is to better describe the data and avoid any numerical derivation, which is not straightforward. One can use the polynomial approach as well, however, it has not been applied in the dielectric relaxation studies probably due to its non-physical basis.

As mentioned by Sokolov et al. [65] high temperature polymers would have high fragility, which is mainly due to the high glass transition temperature \( T_g \) and their rigidity. Using the proposed expression for the fragility index by Qin and McKenna [66]

\[
m \sim 0.28(±0.067)T_g + 9(±20),
\]

we obtain \( m \sim 200(±32) \), which is lower than the value we have estimated. However, it yields a high fragility index. The fragility index of polyetherimide was reported [67] as 214 for polyetherimide estimated with the WLF model eq. (10) (also listed in the table provided by Qin and McKenna [66]).

### 4.4 Conductivity of polyetherimide

Conduction processes in polyetherimide have been reported in the literature by several different groups [9, 10, 53, 55, 68]. As mentioned previously the distribution of relaxation times approach inherently performs or checks the Kramer-Kronig relations [69–71], therefore the ohmic (direct current) conductivity is estimated directly, and its value does not involve any electrode polarization losses due to low frequency polarization processes. The conductivity as a function of the inverse temperature is shown in fig. 6. Due to the limitation imposed by the selection of the low frequency limit in the measurements, the conductivity values \( \sigma_0 < 1.67 \times 10^{-14} \text{Sm}^{-1} \) and \( \log_{10}\sigma_0 < -13.8 \) would not be possible to estimate with any numerical method using the current instrument; more sensitive measurement techniques are needed to resolve such low conductivities. This is clearly presented in fig. 6 as a flat conductivity estimate below 1000/\( T \) = 2.5 or 130°C. Over 130°C an increase in the conductivity is observed. This increase in the conductivity changes after the glass transition temperature indicating the change in the mobility in charge carriers after \( T_g \). A similar behavior was observed for \( \alpha \)-polyvinylidene fluoride [39].

We have adopted two equations to describe the conduction in polyetherimide: Arrhenius and VFTH expressions in eq. (5) and eq. (6) by converting \( \omega_0 \)'s to \( \sigma_0 \)'s in the expression, respectively. Data from Mudarra et al. [53] was included in the conductivity plot (curve “C”), which was generated with \( A = 3.6 \times 10^8 \text{Sm}^{-1} \) and \( W_E = 1.98 \text{eV} \) in eq. (5); it was only applicable to a limited temperature range. While the other data on polyetherimide conductivity from the literature [9, 55, 68] were also analyzed with our results.
The curve generated to represent the data for temperatures just below the glass transition (curve “D”) yields close conductivity values, when extrapolated to low temperatures, to those given by Zebouchi et al. [68] and Rzad et al. [9]. The curve was generated with $A = 2.31 \times 10^{-5}$ and $W_E = 0.71$, while the data from Suh et al. [55] had one data point matching our results. Observe that the data of Rzad et al. [9] is 3 decades lower than that which could be resolved with the measurements with the lowest frequency chosen (0.3 Hz) and the numerical technique. To observe such low current values, we must have employed several $\mu$Hz frequencies in our measurements.

Since the fragility of polyetherimide is high, at high temperatures the behavior of conductivity could both be represented with VFTH and Arrhenius, due to a steep change. Both curves “A” and “B” were obtained from the optimization with $B = 1.50 \times 10^{-7}$, $D = 315.8$ and $T' = 478.11$, and $A = 8.82 \times 10^{25}\text{Sm}^{-1}$ and $W_E = -3.74\text{eV}$, respectively. The VFTH expression was a better fit due to its ability to model high temperature regions. The estimated VFTH temperatures for the $\alpha$-relaxation and the conductivity ($T$’s) are close to each other indicating that the conduction in polyetherimide is ionic assisted by the cooperative motion, which determines the movement of the charge carriers. This observation was similar to $\alpha$-polyvinylidene fluoride [39].

5 Discussion and conclusions

An attempt to characterize the dielectric response of thin polyetherimide films was presented here. A numerical method based on a numerical inversion algorithm was applied to resolve the distribution of relaxation times in $5\text{\mu m}$ thick polyetherimide film. The employed method, as shown several times previously [32–43], does not suffer from a priori assumptions like in other non-linear fitting procedures [72, 73]. It is therefore more straightforward for the user to analyze large data sets as the one presented here, i.e., the broadband dielectric response of a polymer at different temperatures. However, post-processing of the data needs improvement for representation and storage. In conventional curve fitting methods, although the fitted data needs improvement for representation and storage.

The estimated direct-current conductivity of polyetherimide was compared to the data in the literature [9, 10, 53, 55, 68]. A good agreement was obtained for the data below the glass transition temperature $T_g$. The extrapolation of the curve that described the data matched with those of Rzad et al. [9], Zebouchi et al. [68], which were measured with time domain methods.

The results of the dielectric response have shown that polyetherimide has superb properties for passive electrical component applications. Its high glass transition temperature $T_g \sim 214$ and low loss behavior over a wide range of temperatures (up to $200^\circ\text{C}$) would be useful for high temperature applications, primarily capacitors for transformational power systems. The low conductivity again up to $200^\circ\text{C}$ is important for direct-current applications and energy storage because of nearly negligible leakage through the material in those conditions. However, the conductivity brings in other challenges as charging and dissipation of the induced charge in free-standing films together with high dielectric permittivity compared to other thermoplastics used in energy storage applications.

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